



Degradation of diclofenac and 4-chlorobenzoic acid in aqueous solution by cold atmospheric plasma source



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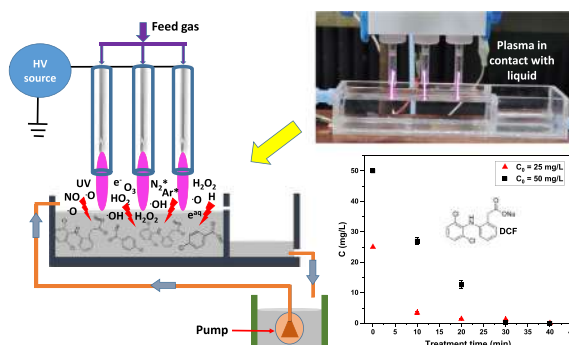
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HIGHLIGHTS

- Multi-pin APPJ utilized to treat DCF and pCBA in aqueous solution.
- Shown efficient removal of contaminants with large plasma/contaminant surface ratio
- Energy yield determined using power deposited to discharge in contact with sample
- A multi-pin APPJ with recirculation system allows for scalability.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, cold atmospheric plasma (CAP) was explored as a novel advanced oxidation process (AOP) for water decontamination. Samples with high concentration aqueous solutions of Diclofenac sodium (DCF) and 4-Chlorobenzoic acid (pCBA) were treated by plasma systems. Atmospheric pressure plasma jets (APPJs) with a 1 pin-electrode and multi-needle electrodes (3 pins) configurations were used. The plasma generated using argon as working gas was touching a stationary liquid surface in the case of pin electrode-APPJ while for multi-needle electrodes-APPJ the liquid sample was flowing during treatment. In both configurations, a commercial RF power supply was used for plasma ignition. Measurement of electrical signals enabled precise determination of power delivered from the plasma to the sample. The optical emission spectroscopy (OES) of plasma confirmed the appearance of excited reactive species in the plasma, such as hydroxyl radicals and atomic oxygen which are considered to be key reactive species in AOPs for the degradation of organic pollutants. Treatments were conducted with two different volumes (5 mL and 250 mL) of contaminated water samples. The data acquired allowed calculation of degradation efficiency and energy yield for both plasma sources. When treated with pin-APPJ, almost complete degradation of 5 mL DCF occurred in 1 min with the initial concentration of 25 mg/L and 50 mg/L, whereas 5 mL pCBA almost degraded in 10 min at the initial concentration of 25 mg/L and 40 mg/L. The treatment results with multi-needle electrodes system confirmed that DCF almost completely degraded in 30 min and pCBA degraded about 24 % in 50 min. The maximum

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calculated energy yield for 50 % removal was 6465 mg/kWh after treatment of 250 mL of DCF aqueous solution utilizing the plasma recirculation technique. The measurements also provided an insight to the kinetics of DCF and pCBA degradation. Degradation products and pathways for DCF were determined using LC-MS measurements.

1. Introduction

Many pharmaceutical and other industrial organic chemical substances have been recognized as emerging persistent organic micropollutants (OMPs) and are frequently found in trace concentrations (ranging from ngL^{-1} and μgL^{-1}) in aquatic bodies (Arslan et al., 2017; Ebele et al., 2017; Patel et al., 2019). It is estimated that several tons of pharmaceuticals, for example, >100,000 tons of pharmaceutical items, are consumed globally each year, inflicting harm to our ecosystem (Kot-Wasik et al., 2007; Wilkinson et al., 2022). Pharmaceutical and other industrial chemicals are discharged to the environment from a wide range of sources, including industry, hospitals and households. Pharmaceuticals are detected in the environment through a variety of channels due to a lack of proper wastewater treatment technologies; they all share the characteristics of providing a considerable threat to public health or the environment, as well as being extremely resistant to traditional wastewater treatment techniques (Arslan et al., 2017; Eggen et al., 2014; Khan et al., 2020). Certain organizations, such as the European Union (EU) and the United States Environmental Protection Agency (USEPA), establishing strict quality criteria for wastewater discharge; yet, various nations and industries are following that (Sathishkumar et al., 2020; Schellenberg et al., 2020). The previous review article by various authors offers thorough information regarding pharmaceutical classification, source and regulations, as well as the impact of pharmaceuticals on the environment and human health. and (Khan et al., 2020; Lonappan et al., 2016; Schwarzenbach et al., 2006).

In this work, DCF sodium and pCBA were chosen as model test pollutants and treated with two plasma sources with different reactor geometry under a variety of experimental conditions. DCF non-steroidal anti-inflammatory drug (NSAID) is used on a global scale to treat inflammatory diseases and reduce pain and fever (Rodrigues et al., 2021; Sathishkumar et al., 2020). According to the authors' statistics, a large amount of DCF, approximately >1000 tons, is used globally each year (Lonappan et al., 2016). As a consequence, DCF is regularly observed in high concentrations in sewage treatment plant effluents that are persistent enough to be found in surface, ground and even drinking water. and (Heberer and Feldmann, 2005; Lonappan et al., 2016). The presence of DCF in water can play a toxic influence on aquatic life, plants and human health (Deng et al., 2021; Sathishkumar et al., 2020). This also led to the unprecedented decline of vultures in India (Taggart et al., 2009). Consumption of DCF has been banned by various countries (e.g. India, Nepal, Bangladesh, etc.) in order to reduce the further decline of the vulture population (Markandya et al., 2008). The pCBA was the second compound that was chosen for the treatment as it is used in various chemical syntheses. The pCBA was found as a by-product in the treatment of various organic chemicals (Bing et al., 2012). The pCBA is an industrial chemical that is commonly used in the pharmaceutical industry. Since it has strong reactivity with $\text{HO}^\bullet$, pCBA was also utilized as a probe compound in several water treatment procedures to determine $\text{HO}^\bullet$ levels (Bing et al., 2012; Pi et al., 2005).

Since conventional wastewater treatment plants (WWTPs) do not completely decompose a large number of OMPs of anthropogenic origin due to unfavorable properties such as poor biodegradability, they are released into natural water bodies (Sathishkumar et al., 2020). For example, conventional WWTPs are reported to remove just 30–70 % of DCF from wastewater streams (Deng et al., 2021; Lonappan et al., 2016). As a result, untreated DCF effluents from WWTPs enters the environment and pollutes ecosystems. Previous research has also stated that DCF can interact with other organic molecules in the environment, leading to the production of other harmful contaminants (Mukherjee et al., 2022).

AOPs have been used successfully as a tertiary treatment method and are regarded as a promising method in the field of wastewater treatment to eliminate OMPs (pharmaceuticals, organic dyes, pesticides, etc.) (Cuerda-Correa et al., 2019; Garrido-Cardenas et al., 2020). The primary feature of AOPs is the generation of $\text{HO}^\bullet$ radicals (high oxidizing potential), which are particularly reactive with a wide variety of organic contaminants and then degrade them, resulting in effective mineralization of organic pollutants into H_2O and CO_2 (An et al., 2010; Wang and Xu, 2012). The goal of generating $\text{HO}^\bullet$ radicals, different AOPs based on chemical admixture ($\text{O}_3/\text{H}_2\text{O}_2$, O_3/UV , $\text{O}_3/\text{UV}/\text{H}_2\text{O}_2$), photocatalysis (UV/TiO_2 , $\text{UV}/\text{TiO}_2/\text{H}_2\text{O}_2$), Fenton ($\text{Fe}_2^+/\text{H}_2\text{O}_2$, $\text{Fe}_2^+/\text{UV}/\text{H}_2\text{O}_2$) and application of electric energy (electrochemical oxidation: Anodic, Electro-Fenton) have been addressed and shown in past study (Amor et al., 2019; Kumar et al., 2021; Macias-Quiroga et al., 2021; Wang and Xu, 2012). The majority of AOPs rely on external chemicals and catalysts to create $\text{HO}^\bullet$ radicals. There are some reviews where authors revealed possible possibilities and limitations related with various AOPs for OMPs degradation (Hijosa-Valsero et al., 2014; Ma et al., 2021).

Cold atmospheric plasmas have been shown to effectively decompose OMPs effectively and are a promising innovative AOP that has been explored for water decontamination (Hijosa-Valsero et al., 2014; Kumar et al., 2022; Li et al., 2014; Magureanu et al., 2015; Malik, 2010; Topolovec et al., 2022). Gaseous plasma can be a very complex mixture of electrons, ions, metastables, UV lights, electromagnetic fields, electric fields, etc. (Bruggeman and Brandenburg, 2013; Chen, 2016; Foster, 2017; Jaiswal et al., 2020). The interaction of cold plasma with an open or controlled environment can produce a plethora of reactive species that can be used for a variety of beneficial chemical reactions in applications such as agriculture, medicine, sterilization, disinfection, material synthesis, electronics and so on (Barjasteh et al., 2021; Domonkos et al., 2021; Fridman, 2008; Tomić et al., 2021).

Plasma-liquid interactions have grown in popularity in recent years due to crucial characteristics such as the simultaneous production of many powerful chemical reactive oxidants at low temperatures, which has the ability to have a large impact on a variety of prospective applications. For example, the APPJ source which was used in this study for plasma formation in the gas phase above the liquid surface can cause a variety of chemical and physical effects, including the generation of reactive oxygen and nitrogen species (RONS: $\text{HO}^\bullet$, $\text{O}^\bullet$, HO_2 , H_2O_2 , O_3 , NO_3^- , NO_2^- , $\text{NO}^\bullet$, ONOO^- , etc.), UV photons, as well as hydrated electrons (free electrons in the solution) without the usage of chemical agents (Bradú et al., 2020; Bruggeman et al., 2016; Foster et al., 2012; Lukes et al., 2014). The APPJ has a high potential for transporting highly concentrated reactive species from the gas phase to the liquid phase via the jet's gas flow (Bruggeman et al., 2016; Du et al., 2018). As a consequence, such a plasma source can be used as a green approach of destroying complex non-biodegradable organic contaminants. Previous studies attempted to use certain CAPs with various reactor configurations, powered by different electrical signals (AC, DC, RF sources), different working gases (argon, helium, oxygen, nitrogen) and several process parameters to decontaminate water (Hijosa-Valsero et al., 2014; Kumar et al., 2021; Malik, 2010). It was also noted that there have been a few earlier experiments that used CAPs to generate large plasma and surface contacts to remove organic contaminants from water.

The purpose of this study was to investigate the degradation of pCBA and DCF in aqueous solutions utilizing CAPs (pin-APPJ and multi-needle electrodes-APPJ with recirculation), as well as to compare the performance of both plasma reactors in terms of pollutants removal and energy yield. The pin-APPJ, which has 1 jet, was utilized to treat 5 mL of polluted water. The multi-needle electrodes-APPJ, which have 3 jets and continuous

In this paper, we will present electrical characterization, Optical Emission Spectroscopy and ICCD (Intensified Charged Coupled Device) imaging of 1-pin APPJ and 3-pin APPJ. The measurements were made during the removal of pollutants from contaminated water samples. These two plasma devices are used for DCF and pCBA removal. The HPLC analytical instrument was used to study the degradation kinetics of DCF and pCBA. The obtained data was used to determine the energy yield and compared to the

The schematic of the experimental setup of pin-APPJ and the picture of the plasma jet contacting the liquid sample is shown in Fig. 1a. The plasma source consists of a concentrically placed glass tube, a ceramic tube and a stainless steel wire as an electrode. The outer and inner diameters of the glass tube are 6 mm and 4 mm, respectively. The stainless steel wire with



a diameter of 1 mm with a sharpened tip is used as a high-voltage electrode and while the rest of the wire was covered with a ceramic tube. The electrode is powered by a high voltage radio frequency (RF) power supply with a frequency of 332 kHz. The other grounded electrode is the solution placed in a vessel with copper tape glued to the outer bottom side of the vessel and connected to the ground. In this configuration, plasma was generated between the electrode tip and stagnant liquid surface using argon as a working gas.

2.1.2. Multi-needle electrodes-APPJ

The schematic diagram of the experimental setup of multi-needle electrodes-APPJ is shown in Fig. 1b. The atmospheric pressure plasma jet over the flowing liquid surface was generated through a multi-syringe needle electrode type configuration. The inner and outer diameter of each needle was about 1 mm and 1.8 mm, respectively. The needles were inserted inside the glass tubes, where the inner diameter, outer diameter and length of each tube were about 3.7, 5 and 45 mm, respectively. The distance between the tip of each needle and the end of each tube was 7 mm. The distance between the two adjacent needle electrodes was 20 mm. The distance between needle tips and solution was set at 15 mm. Similarly as for a single-pin system, the copper tape was wrapped over the bottom of the sample vessel while the electrodes were supplied by a sine signal at a frequency of 351 kHz. Since the same power supply was used for the pin-electrode jet, the difference in the electrical impedance of the circuit causes a change in the signal frequency. Argon gas with a total flow rate of 2 slm (standard liter per minute) was used as a feed gas, distributed evenly between 3 pin electrodes.

In both configurations, the voltage (v) at the powered electrode was determined by a high voltage probe (Tektronix 6015A). The current at the powered electrode was measured with a current probe (Agilent N2783B). The voltage drop (v_r) across the 1 k Ω resistor used to establish the current in the grounded part (i_g) was recorded using a voltage probe (Agilent 10073C). The time variable voltage and current signals were monitored by using a 4-channel, 200 MHz, 2.5 GSps digital oscilloscope (Tektronix MDO3024). Simultaneously, the data from the oscilloscope was transferred to a laptop for further analysis. The power delivered from the power supply to the plasma source and the power in the grounded line, i.e. the power deposited from the plasma passing through the sample, was calculated.

2.2. Treatment of solution

In the configuration of pin-electrode APPJ, 5 mL of solution was exposed to the jet with different treatment times. In each experiment, 2 mL of plasma-treated samples were taken for further analysis. In the assembly of the flow system, a total volume of 250 mL of solution was treated, where around 12 % of the solution was continuously exposed to the treatment zone. The solution was circulated by a pump (kept inside the solution reservoir) at a flow rate of 300 mL/min and a corresponding Reynolds number of 1349. The Reynolds number determines the flow pattern, hence at this value, the flow was in the laminar flow regime (Xiong et al., 2012). The total treatment times were 30 min for DCF and 50 min for pCBA, where 2 mL of samples were taken at specific intervals for analysis. In the event of the treatment of 5 mL using pin-APPJ (1 jet), each sample corresponded to a single experiment. In the case of treatment using a plasma (3 jets) recirculation system (volume 250 mL), there was continuous sampling at various time intervals.

In both experimental systems, there were minor reductions in volume due to heating and gas flow (always <0.4 % for 3 jets and 36 % for 1 jet, longer treatment time). The volume reduction was always recorded and incorporated into the final calculation of degradation removal. It shall also be noted that volatilization of the target contaminants is not expected due to their low volatility.

2.3. Solution characterization

DCF (chemical formula: $C_{14}H_{10}C_{12}NNaO_2$, molecular weight: 318.13 g/mol, purity ≥ 98 %, CAS number: 15307-79-6) and pCBA (chemical

formula: $C_7H_5ClO_2$, molecular weight: 156.57 g/mol, purity ≥ 98 %, CAS number: 74-11-3) were purchased from Sigma Aldrich.

In this investigation, DCF with initial concentrations of 25 mg/L and 50 mg/L and pCBA with initial concentrations of 25 mg/L and 40 mg/L were used in the treatment. First, a stock solution with a high initial concentration was prepared by dissolving each compound in distilled water. The lower concentration was obtained by diluting the concentrated solution with distilled water. Both DCF and pCBA are soluble in water to a considerable extent. For example, DCF and pCBA solubility in distilled water have been reported to be 2425 mg/L (Jankunaite et al., 2017) and 80 mg/L (Phatak and Gaikar, 1996), respectively. The initial concentrations chosen are greater than those found in the water bodies (ng/L to g/L). The higher initial concentrations evaluated allowed for proper examination of the degradation pattern. The authors claimed that prior studies on the degradation of OMPs by AOPs were also conducted in similar, higher initial concentration ranges (Kumar et al., 2021; Lesage et al., 2013; Rong et al., 2014).

Chromatographic analysis of the samples was carried out on an Agilent HPLC instrument, series 1100 (Agilent Technologies, Waldronn, Germany), with a photodiode array detector, using Hypersil BDS-C18 column (125 $\times$ 2 mm) with 5 μ m particle size (Phenomenex, Torrance, USA), thermostated at 30 °C. All samples were filtered through a 0.2 μ m cellulose filters (Agilent technologies, Santa Clara, CA, USA) prior to analysis. The mobile phase consisted of a 1 % (v/v) solution of orthophosphoric acid in ultrapure water (solvent A) and acetonitrile (solvent B). Acetonitrile and orthophosphoric acid were LC-MS grade (Fisher Scientific, Leics, UK) and ultra-pure water was generated by using the Water Purification System (New Human Power I Integrate, Human Corporation, Seoul, Republic of Korea). The flow rate of the mobile phase was 0.5 mL/min and the injection volume was 10 μ L. A gradient program was used as follows: 80–30 % A, 0–7 min; 30–80 % A, 7–12 min (DCF) and 80–60 % A, 0–8 min; 60–80 % A, 8–12 min (pCBA). The detection wavelengths were set at 240 nm and 276 nm for pCBA and DCF, respectively. Quantification was performed using standardized calibration curves. The content of products in the samples was determined by calculation of peak area and expressed as milligrams per liter. Chromatographic data were recorded and processed by using HP ChemStation Chromatographic Software (Palo Alto, CA, USA).

The byproducts derived from the degradation of DCF were analyzed by using Orbitrap Exploris 120 high-resolution LC-MS (Thermo Fisher Scientific). A reverse-phase column (Hypersil GOLD-Selectivity C18, 3 μ m, 2.1 mm $\times$ 50 mm) was used for the separation of the component in the mixture. The column temperature was set at 30 °C. The injection volume and flow rate was 10 μ L and 0.4 mL/min, respectively. The analysis was carried out in a negative ion and positive ion mode with ionization voltages of 2500 V and 3500 V, respectively. Heated electrospray ionization (H-ESI) system was used as an ion source. The mass spectra were acquired in full scan mode in the mass range of 40 to 5000. In the positive ion mode, the mobile phase composition was HPLC water with 0.1 % formic acid and methanol. In the negative mode, the mobile phase consists of HPLC water with 5 mM ammonium acetate (at pH 8) and methanol. Variation in composition, first started with 98:2 % and increased up to 2:98 % in 4.7 min and remained constant for 6 min and decreased up to 98:2 % in 9 min. The extracted data was transferred to Thermo Scientific™ Compound Discoverer software for further processing. The software also provided an excel based report containing details of the compound, isotopes, retention time, mass to charge ratio, fragment ion, intensity, mass error (ppm), etc. The chemical structure and fragmentation pattern for each possible transformation product were drawn by using ChemSketch software (ACD/ChemSketch Freeware).

DCF and pCBA removal efficiency was defined as Eq. (1).

$$\text{Removal (\%)} = \frac{C_o - C \times d}{C_o} \times 100 \quad (1)$$

where C_o (mg/L) is the initial concentration before plasma treatment, C (mg/L) is the final concentration after plasma treatment and d is the evaporation coefficient.

The energy yield of the removal of DCF and pCBA is determined by the following Eq. (2).

$$\text{Energy yield} \left(\frac{\text{mg}}{\text{kWh}} \right) = \frac{C_o \left(\frac{\text{mg}}{\text{L}} \right) \times V_o (\text{L}) \times \frac{1}{100} \times \text{Removal}(\%)}{P_{\text{mean at the sample}} (\text{kW}) \times t (\text{h})} \quad (2)$$

where C_o (mg/L) is initial concentration of the plasma treated compound, V_o (L) indicates the initial volume of plasma treated compound, P_{mean} (kW) is discharge power at the sample and t (h) is the treatment time.

In all plasma treatment processes, the solution pH was determined by a pH meter (HANNA-HI1330).

2.4. Optical characterization

Plasma emission spectra was captured by using Maya2000 Pro-UV-NIR (Ocean Insight-High Sensitive Spectrometer) with an optical resolution of 0.18 nm full width at half maximum. The optical fiber (M114L02) length of 2 m and core diameter of 600 μm was placed perpendicular to the plasma jet in order to capture the emission from the whole volume of the plasma jet. The emission was recorded with an exposure time of 50 ms. The raw OES data was transferred to a laptop and analyzed in OriginPro data analysis software. Plasma emission is measured with and without plasma (background). The background spectra were subtracted from the plasma emission spectra to achieve the exact spectrum. The spectral intensity was corrected for the optical system efficiency.

The plasma emission profiles were characterized by using an Andor iStar DH734i ICCD camera with a Nikon UV-105 mm f/4.5 lens mounted. The lens of the ICCD camera was set perpendicular and in front of the plasma jet. During the analysis, the sample was filled in a quartz-type petri dish (to avoid the blockage of light having a lower wavelength (e.g. UV)) and placed under APPJ. There was no sample flow during ICCD imaging. In order to capture the spatial emission distribution of various reactive species ($\text{HO}^\bullet$, $\text{O}^\bullet$, $\text{H}^\bullet$, N_2 (SPS: second positive system & FNS: First negative system), Ar^*), band pass filters (attached with holder) were used and placed in the front of the lens. The transmittance percentage of each filter was calculated by spectrophotometer (Beckman Coulter DU 720 UV/Visible). A MATLAB script was used to estimate the geometry of the plasma jet and to process ICCD camera images.

3. Results and discussions

3.1. Electrical measurements

The electrical measurements were implemented to investigate the discharge parameters (voltage, current, power deposition). The discharge

voltage and current are the critical parameters that are used to obtain dissipated power in the plasma system. The high voltage was applied to the electrodes (to pin and multi-needles). The sharp edge electrode configuration was selected because it creates a high electric field around the tip. When the applied voltage is strong enough this electric field is enough for the breakdown to occur followed by the production of a conduction channel in the discharge gap.

The voltage-current parameters were investigated and the power deposition to the plasma system for both plasma sources was estimated. Fig. 2 displays the relationship between RMS voltage vs RMS current and power deposition at sample vs RMS voltage for 3-jets.

The mean power deposition at the sample was calculated using eq. 3, by averaging the instantaneous power (product of time-varying voltage $v(t)$ and time-varying current $i(t)$ waveforms) over a time interval of $n = 6$ periods.

$$P_{\text{mean at sample}} = \frac{1}{nT} \int_{T_1}^{T_2} v(t) \times i_g(t) \times dt \quad (3)$$

where, $P_{\text{mean at sample}}$: mean power at the sample; $v_R(t)$: voltage drop at the resistor; resistance $R = 1 \text{ k}\Omega$; $i_g(t)$: current at the ground (sample); $nT = T_2 - T_1$.

$$i_g(t) = v_R(t)/R \quad (4)$$

The plasma system was electrically described in two modes: when the plasma was ignited and when it was not. When there was no plasma ignition, voltage and current changed linearly, but voltage decreased after plasma discharge began (Fig. 2a). The power delivered to the sample after plasma ignition, which is essential in our investigation, was in the range of 5.8 W–16 W (Fig. 2b).

3.2. Optical characterization of plasma

3.2.1. OES measurements

The recorded spectra during the treatment of DCF by a multi-needle electrodes-flow system, shown in Fig. 3. The spectra between 200 and 427 nm, mainly correspond to various emission bands, such as the vibrational transition of $\text{HO}^\bullet$ ($\text{A}^2\Sigma^+ \rightarrow \text{X}^2\Pi$), N_2 SPS ($\text{C}^3\Pi_u \rightarrow \text{B}^3\Pi_g$), N_2^+ FNS ($\text{B}^2\Sigma_u \rightarrow \text{X}^2\Sigma_g$) and nitric oxide NO_γ ($\text{A}^2\Sigma^+ \rightarrow \text{X}^2\Pi$). The spectrum lines between 697 and 965 nm are dominated by the excited argon atoms ($4p \rightarrow 4s$) and $\text{O}^\bullet$ ($3p^5P \rightarrow 3s^5S$ and $3p^3P \rightarrow 3s^3S$). The spectra of N_2 SPS, N_2^+ FNS, $\text{O}^\bullet$ and NO_γ are typical for open-air-argon plasma discharges operating above a water sample (Lamichhane et al., 2022).

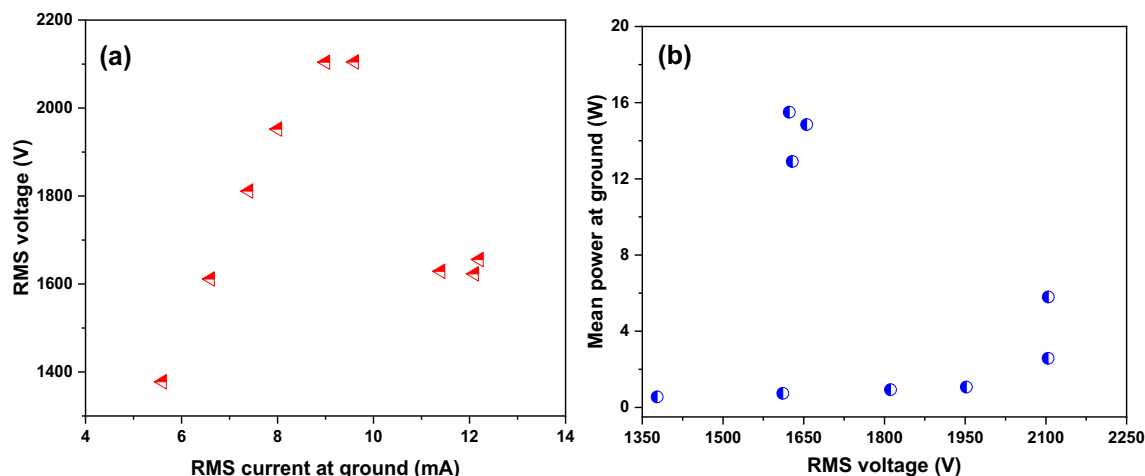


Fig. 2. (a) V-A characteristics (RMS voltage as a function of RMS current at sample/ground), (b) variation of mean power (delivered at the sample/ground) as a function of RMS voltage.

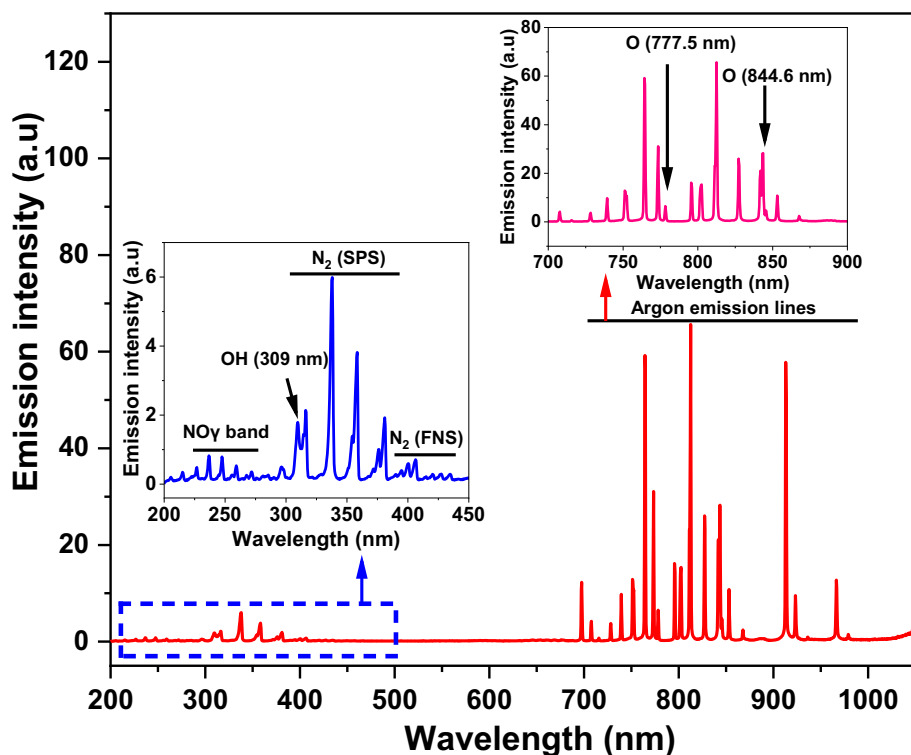


Fig. 3. The emission spectrum of argon – atmospheric pressure plasma jet in contact with ambient air and over the liquid surface, integration time 50 ms. Experimental conditions: $C_0 = 25$ mg/L (DCF), $V_0 = 250$ mL, P_{mean} at the sample 8 W, argon flow of 2 slm. Inset graphs depict the zoomed-in portion.

The collision of energetic species with H_2O molecules may be the primary source of $HO^\bullet$ production (Eqs. (5) & (6)) (Invernizzi et al., 2020; Jaiswal and Aguirre, 2021). Water molecules in the gas phase are formed mainly through evaporation, plasma species bombardment, gas movement and air (Ghimire et al., 2021; Liu et al., 2016). The threshold energy required to dissociate H_2O by electron impact dissociation is approximately 4.4 eV. As a result, the reaction necessitates a lower electron temperature, which is easily attained in argon-APPJ.



In general, argon metastables have energies ranging from 11.5 to 11.8 eV, which is high enough to react with H_2O as well as excite N_2 ($C^3\pi_u \rightarrow B^3\pi_g$, 11.03 eV) (Soler-Arango et al., 2018), as explained by the following reactions (7) and (8).



Excited molecular nitrogen and its metastables have enough energy to react with and dissociate H_2O molecules (Eq. (9)) (Adhikari et al., 2021; Uhm et al., 2018).



The threshold energy for N_2^+ ion generation is substantially greater (18.8 eV) than for excitation, hence the FNS band appeared with much lower intensity (Cullen and Milosavljević, 2015; Gazeli et al., 2015). One possibility for the FNS generation reaction is that argon metastables with high energy can ionize N_2 via impact excitation and ionization (Eq. (10)).



Atomic O can originate after the dissociation of molecular O_2 by energetic electrons and argon metastables via collisional dissociation (Eqs. (11) & (12)). The process reaction requires lower threshold energy (below 11 eV) (Adhikari et al., 2021).



The emission from the NO_y system was observed. NO_y production is caused by admixing air within the plasma (Yousfi et al., 2011), described by the following equations.



The presence of UV emission in the APPJ can also generate strong oxidants, such as $HO^\bullet$ and $O^\bullet$ by photolysis of H_2O and O_2 (Attri et al., 2015; Invernizzi et al., 2020).

From the point of water decontamination, the presence of $HO^\bullet$ and $O^\bullet$ in the plasma is found to be major short-lived oxidants having high oxidative potential that can break down almost most non-biodegradable refractory organic substances by the following reaction mechanism: hydrogen atom abstraction, electrophilic addition and electron transfer reactions. Abundant excited argon and other RONS can also play an important role in water decontamination (García et al., 2017; Yehia et al., 2020). The reactive species produced by the plasma-gas-liquid-interface can transfer/diffuse/penetrate in the liquid phase and form secondary reactive species in the bulk solution, which can further drive degradation and change the pH and conductivity of the solution (Bruggeman et al., 2016; Foster et al., 2012; Kumar et al., 2022; Lukes et al., 2014).

We have also performed OES for pin-APPJ, which can be found in our prior published work (Kumar et al., 2022) and it was found that the emission spectra were almost identical. In case of pin-APPJ, a weak H_α line

was present in the spectra, but the H_{α} emission line was absent in multi-needle electrodes-APPJ, which might be created and promptly quenched.

The production of RONS in liquid phase is directly connected to the plasma chemistry in gas phase and gas/liquid interface (Bruggeman et al., 2016; Lukes et al., 2012; Pawlat et al., 2019). The presence of reactive species in the gaseous phase of plasma discharge responsible for further reactions at plasma gas/liquid interface can be determined by OES. In our previous work, we have used several methods for the quantification of long lived RONS in the plasma treated liquid (Kumar et al., 2022; Tomić et al., 2021). Several long-lived RONS (e.g., H_2O_2 , NO_3^- and NO_2^-) were detected and quantified in the aqueous phase.

3.2.2. ICCD imaging of pin and multi-needle electrodes-APPJ

3.2.2.1. ICCD imaging of pin-APPJ. The ICCD imaging with filters was performed to investigate the spatial resolution emission of reactive species produced by argon-APPJ when it interacts with liquid in the presence of ambient air. Various optical filters (310 nm, 425 nm, 660 nm and 780 nm) were employed to identify certain lines/bands inside the plasma. Each filter's bandwidth (bandpass region) was around 10 nm. Acquisition parameters such as gate pulse width, exposure time and gain remained constant during ICCD imaging with filters. Fig. 4 shows the spatially resolved ICCD images.

The emission of $HO\cdot$ and vibrationally excited N_2 (SPS, 315.8 nm) was monitored using a filter with a center wavelength of 310 nm. The emission from mostly $O\cdot$ was measured at 777.5 nm in combination with excited argon (since excited argon also emits near 780 nm) using a 780 nm-centered filter. The filter with a central wavelength of 660 nm is primarily employed to record H_{α} emission at a wavelength of 656 nm. The emission of excited N_2 (SPS) and ionic nitrogen N_2^+ (FNS) was monitored using a 425 nm filter.

Fig. 4a shows an image captured using a 310 nm filter. Fig. 4b shows the images associated with the remaining filters, such as 780, 425 and 660 nm; the intensity level in these images was kept constant. In the case of the 310 nm filter, the emission from $HO\cdot$ (containing N_2 SPS) was extensively dispersed across the active plasma. It should be noted that the images with the highest emission intensity were obtained using 310 nm filters. The emission profiles recorded at 425 nm, 660 nm and 780 nm were nearly identical. Nevertheless, intense emission occurred in the case of the 780 nm filter (mainly owing to plasma interaction with the ambient air) and dominated at close to the liquid surface. Overall, ICCD time-integrated images demonstrated the presence of several reactive species in the plasma that could be responsible for the degradation of chosen OMPs in this investigation.

3.2.2.2. ICCD imaging of multi-needle electrodes-APPJ. The ICCD time-integrated imaging of multi-needle electrodes-APPJ was also done. The

camera and filters were placed in such a way that they captured emissions from all three jets. Fig. 5 displays images captured with 310 nm and 780 nm filters, with the intensity scale constant for both filters for comparison. The spatially resolved emission intensity in all three jets was observed to fluctuate in both filters. For example, one jet's intensity was higher while the intensity in the other two jets was different at the same time. The variation in emission intensities could be related to a different level of excitation in the jets.

The emission intensity profile of $HO\cdot$ and N_2 (SPS) was brighter all along the jets but more intense in the middle jet, which could be owing to an intense concentrated electric field and excitation. $O\cdot$ and excited argon were observed in the jets, but strong emission was noticed in the middle of all three jets and the intensity was significantly reduced downstream at the jets' top.

3.3. Treatment of pharmaceuticals with pin-APPJ and multi-needle electrodes-APPJ

3.3.1. Pin-APPJ

The treatment studies were carried out by directly exposing the pin-APPJ to an aqueous solution containing DCF and PCBA while taking into account various experimental parameters that influence the degradation of both compounds. The parameters, such as initial pollutant concentration and treatment time, were adjusted and the resulting degradation results were thoroughly explained.

Fig. 6a depicts the degradation of pCBA in solution as a function of plasma treatment time. The degradation profile revealed that more than half of the initial pCBA concentration was eliminated in the first 5 min. The greatest degradation occurred in the first 3 min, with degradation efficiency values of approximately 63 % for 25 mg/L and 56 % for 40 mg/L. The initial concentration has a considerable impact on the behavior of the oxidation curves during the first 3 min of plasma treatment. After 10 min of treatment, both degradation curves merged, resulting in the complete removal of pCBA.

We fitted the data displayed in Fig. 6a to obtain pCBA degradation kinetics. The kinetics of pCBA decay demonstrate that it is well suited to the first order:

$$\ln \frac{C}{C_0} = -kt \quad (15)$$

where k is the kinetic rate constant.

The calculated R^2 (correlation coefficients) was evidence that the degradation of pCBA fitted well with first-order kinetics. Lower initial pCBA concentration promoted the highest degrading first-order rate constant, while larger initial pCBA concentrations reduced the rate constant. Higher concentration of pCBA represents more pCBA molecules in the

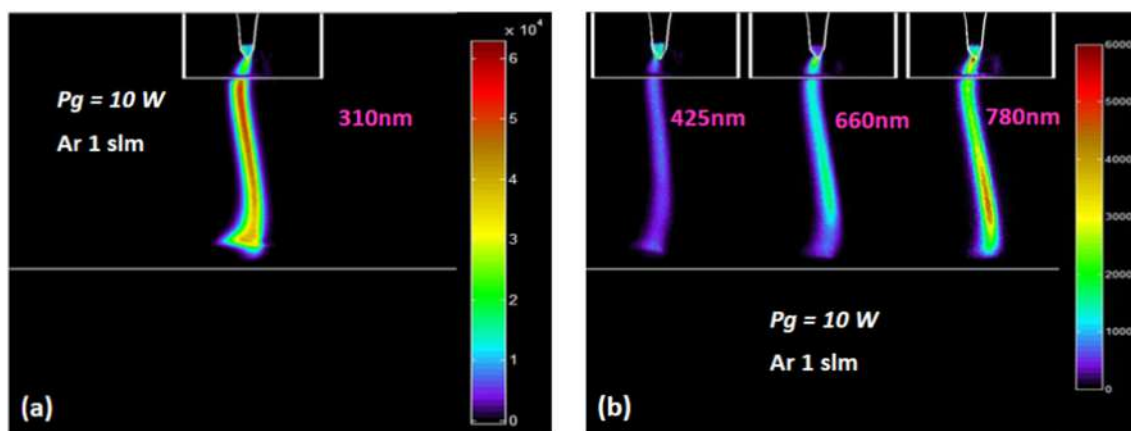


Fig. 4. The ICCD images with filters, (a) 310 nm, (b) 425 nm, 660 nm and 780 nm. Acquisition parameters: exposure time 20 ms, gate pulse width 5 ms and gain 50.

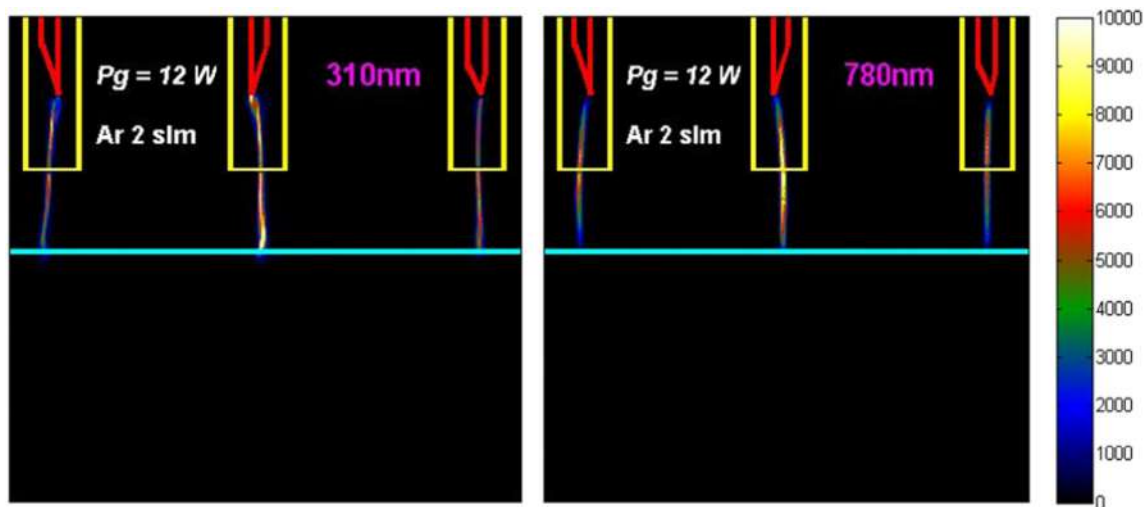


Fig. 5. The ICCD images with filters (310 nm and 780 nm). Acquisition parameters: exposure time 20 ms, gate pulse width 3 ms and gain 80.

solution resulting in more reaction sites for plasma species. Moreover, during the treatment by-products of pCBA are formed so not all introduced plasma species continue reacting with the original pCBA molecules. Taking

into account that the rate of plasma species production remains relatively constant in time this can lead to lower degradation rate at higher concentrations.

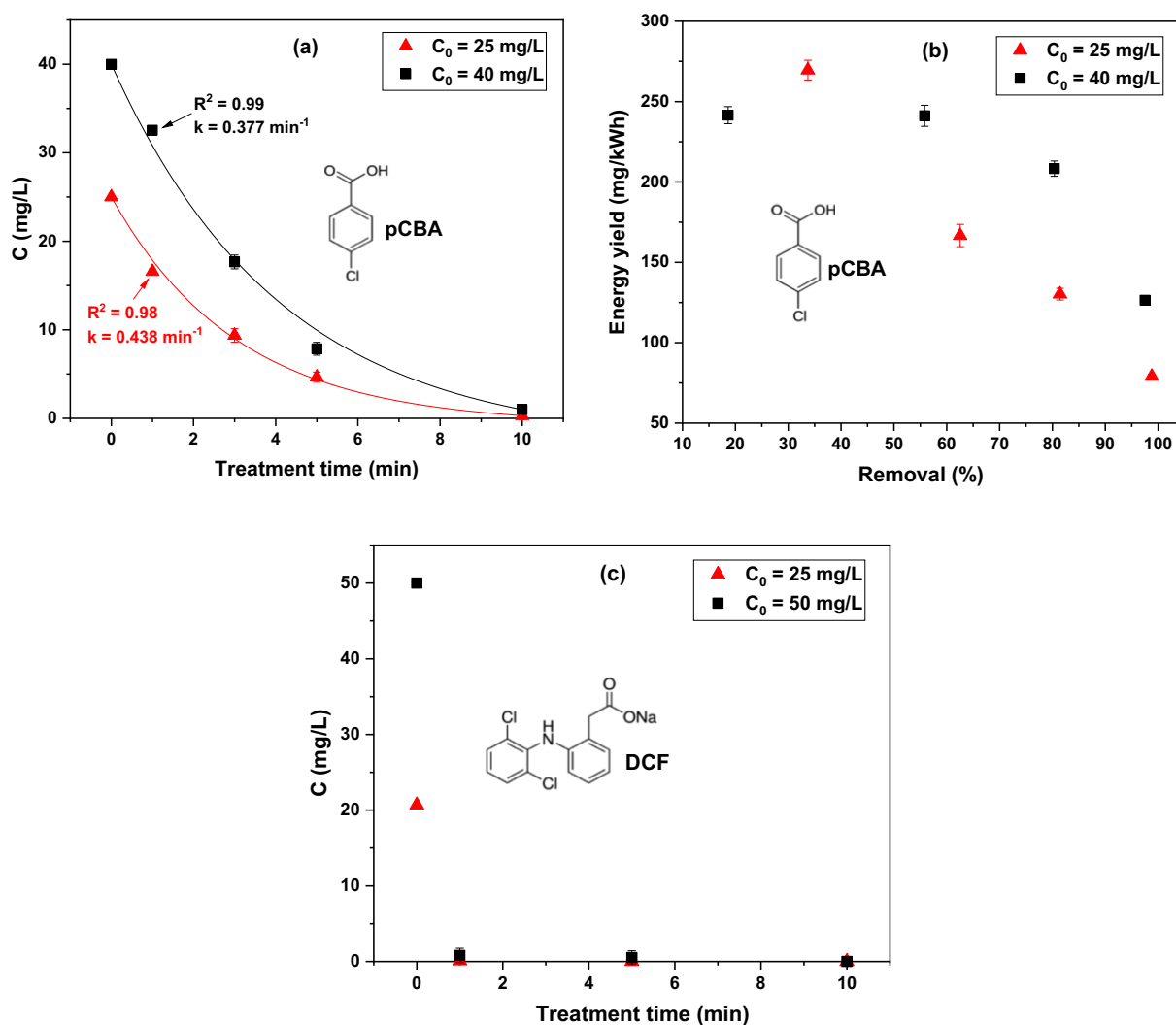


Fig. 6. (a) Change in pCBA concentration with treatment time, (b) energy yield vs removal %, (pCBA, $C_0 = 25$ mg/L and $C_0 = 40$ mg/L) and (c) change in DCF concentration with treatment time (DCF, $C_0 = 25$ mg/L and $C_0 = 50$ mg/L). Experimental conditions: $V_0 = 5$ mL, argon flow of 1 slm, P_{mean} at the sample 10 W.

Consequently, the half-life ($t_{1/2}$) pCBA decomposition time was determined as:

$$t_{1/2} = \frac{0.693}{k} \quad (16)$$

The time required to remove 50 % of the pCBA was 1.58 min for 25 mg/L and 1.83 min for 40 mg/L.

Aside from degradation, energy yield is a significant factor to consider in investigating the energy effectiveness of the plasma source. The energy yield for removing pCBA was calculated. Fig. 6b depicts the energy yield for removing pCBA as a function of removal %. Because of the longer treatment duration, the energy yield was lower at higher removal. For a longer treatment, power deposition may become increasingly concentrated in unproductive processes (e.g., the interaction of byproducts with plasma-induced species), resulting in a drop in energy yield. In the literature on plasma-based treatment, the energy yield is typically reported in terms of 50 % elimination for comparison. As a result, the amount of energy required to decompose 50 % of contaminants was calculated. Energy yield at 50 % pCBA elimination was 253 mg/kWh for 25 mg/L and 354 mg/kWh for 40 mg/L, respectively. More detailed comparison of the data on energy yield collected from the literature for pCBA removal by plasma was given at the end of Section 3.3.2.

DCF was treated with plasma while the same experimental conditions were considered as pCBA. As the solution was subjected to plasma, DCF decayed rapidly, as shown in Fig. 6c. The rapid degradation in the early stages of the process suggests that the majority of plasma-induced reactive species interacted and oxidized DCF. The chemical nature of organic substances (e.g. structural features/chemical reactivity) influences degradation. It has been found, for example, that DCF is strongly reactive not only with HO•, but also with other oxidants. As a result, HO• and other reactive species may play a significant role in the rapid degradation of DCF.

The degradation kinetics of DCF was not established because DCF practically vanished in 1 min and there were just a few points available to analyze the kinetic behavior. So, in our paper, we just reported the DCF degradation pattern.

The energy yield for DCF removal could not be determined precisely from the obtained measurements since DCF degraded quickly during the shortest sampling interval of 1 min for both initial concentrations. From these measurements, we can only conclude that the energy yield for DCF was higher than pCBA.

The pH of plasma-treated pCBA and DCF solutions was found to be lower after treatment. After 10 min of plasma treatment, the pH of pCBA decreased from 4 to 2.57 at 25 mg/L and to 2.65 at 40 mg/L. In the instance of DCF, the initial pH of the DCF solution was 6, but after 10 min of plasma treatment, the pH dropped to 2.66 at 25 mg/L and 2.69 at 50 mg/L. In both cases, the final pH was reduced to similar values after 10 min of plasma treatment. Additional experiments were conducted to treat distilled water without contamination with the same system of pin-APPJ. After 10 min of treatment, the pH of distilled water was reduced from 6.5 to 2.62. So, the reduction of pH is not related to contaminants but probably to plasma-induced nitrogen-based species. Changes in pH demonstrated the existence of acidogenic species (inorganic acids) in the liquid after plasma liquid interaction with ambient air (Judée et al., 2018; Rezaei et al., 2019). The authors found that the principal acidic species formed in the liquid are HNO₃ and HNO₂ (Thirumdas et al., 2018; Wenjuan and Xiangli, 2007). In water, these strong acids dissociate to their respective anion and a hydronium ion. Researchers determined that the pH value influences the degradation kinetics of OMPs in water -lower pH results in faster degradation. Rong et al., 2014, for example, investigated DCF degradation using an air DBD plasma source and reported that DCF removal was greater at pH 2.9 and 6.15 as compared to an alkaline environment at pH 10.1. Similar effect of low pH on pCBA degradation was reported in a study conducted by He et al., 2011. According to these results, we presume that lowering the pH value in the plasma treatment in our experiments contributed to the degradation of DCF and pCBA.

3.3.2. Multi-needle electrodes-APPJ

Another approach for treating significant amounts of polluted water, at least a quarter liter, was tested. Treatments were carried out using a continuous flow system with varying treatment times. The plasma source with multi-needle electrode geometry was employed to create plasma over the recirculated DCF and pCBA solutions. The goal of generating multi plasma jets over the recirculation solutions was to increase the plasma-liquid contact area and introduce more reactive species into the liquid, which can enhance the degradation.

The DCF degradation curve as a function of treatment time is depicted in Fig. 7a. DCF decayed significantly in the first 10 min at both initial DCF concentrations, although the rate of degradation was faster at the lower initial concentration. The complete removal of DCF was seen with both initial concentrations in 30 min. The quicker degradation could be explained by plasma-generated reactive components participating in DCF removal.

To undertake the kinetic analysis and determine the reaction order and rate constant, the experimental results were evaluated. When the treatment was performed at an initial concentration of 25 mg/L, the decay of DCF was well-matched with a first-order equation with a rate constant of 0.145 min⁻¹. The first order revealed that plasma-generated reactive species not only interacted with DCF, but also with intermediate products. DCF degradation is governed by a zero-order equation for rich initial concentrations, such as 50 mg/L and the rate of degradation was 1.636 mgL⁻¹ min⁻¹, as indicated by provided Eq. (17). Zero-order decay (Eq. (17)) demonstrates that the reaction rate is steady and independent of DCF concentration.

$$C = C_0 - kt \quad (17)$$

The time required to degrade 50 % of DCF was assessed and the half-life ($t_{1/2}$) for zeroth-order decay was calculated using the given Eq. (18). It took roughly 5 min for lower initial concentrations of DCF to degrade 50 % and three times longer for higher concentrations.

$$t_{1/2} = \frac{C_0}{2k} \quad (18)$$

Fig. 7b depicts the calculated energy yield as a function of DCF removal for two initial concentrations. DCF concentration has a substantial impact on energy yield. The high energy yield for DCF elimination is most likely owing to a greater contribution of plasma-induced reactive species to the oxidation process. At 25 mg/L and 50 mg/L, the energy yield values were approximately 1542 mg/kWh and 3084 mg/kWh for complete degradation of DCF, respectively.

Multi-needle electrodes-APPJ was also explored for the treatment of pCBA, the degradation result can be seen in Fig. 7c. During the treatment of pCBA, slow degradation was observed, about 24 % removal of pCBA after 50 min of plasma treatment. A kinetic study was conducted to explore the kinetic behavior of pCBA. The investigation was carried out with only three treatment times in consideration. The degradation of pCBA corresponded closely to a first-order equation. The kinetic constant for pCBA was 0.00497 min⁻¹, which was lower than the kinetic constant for DCF.

The slow decomposition of pCBA in a case of 3-pin APPJ can be attributed to the different experimental setup compared to 1-pin APPJ. In case of 3-pin jet, although the effective plasma-liquid contact is larger than in case of 1-pin APPJ here we had a better mixing of the contaminated sample. This could lead to a higher competition for the reactive oxygen species (HO• radicals for example), which plays important role in interactions with pCBA byproducts and with the original compound. Also, increment of treated volume is reducing the efficiency of the treatment as with DCF compound.

The obtained energy yields at 50 % removal of DCF and pCBA by both plasma sources are estimated and compared to previous plasma-based DCF and pCBA removal. As previously stated, DCF was the most efficiently degraded pollutant in both plasma sources in our investigation. However, when the plasma sources were compared, the resulting energy yields were

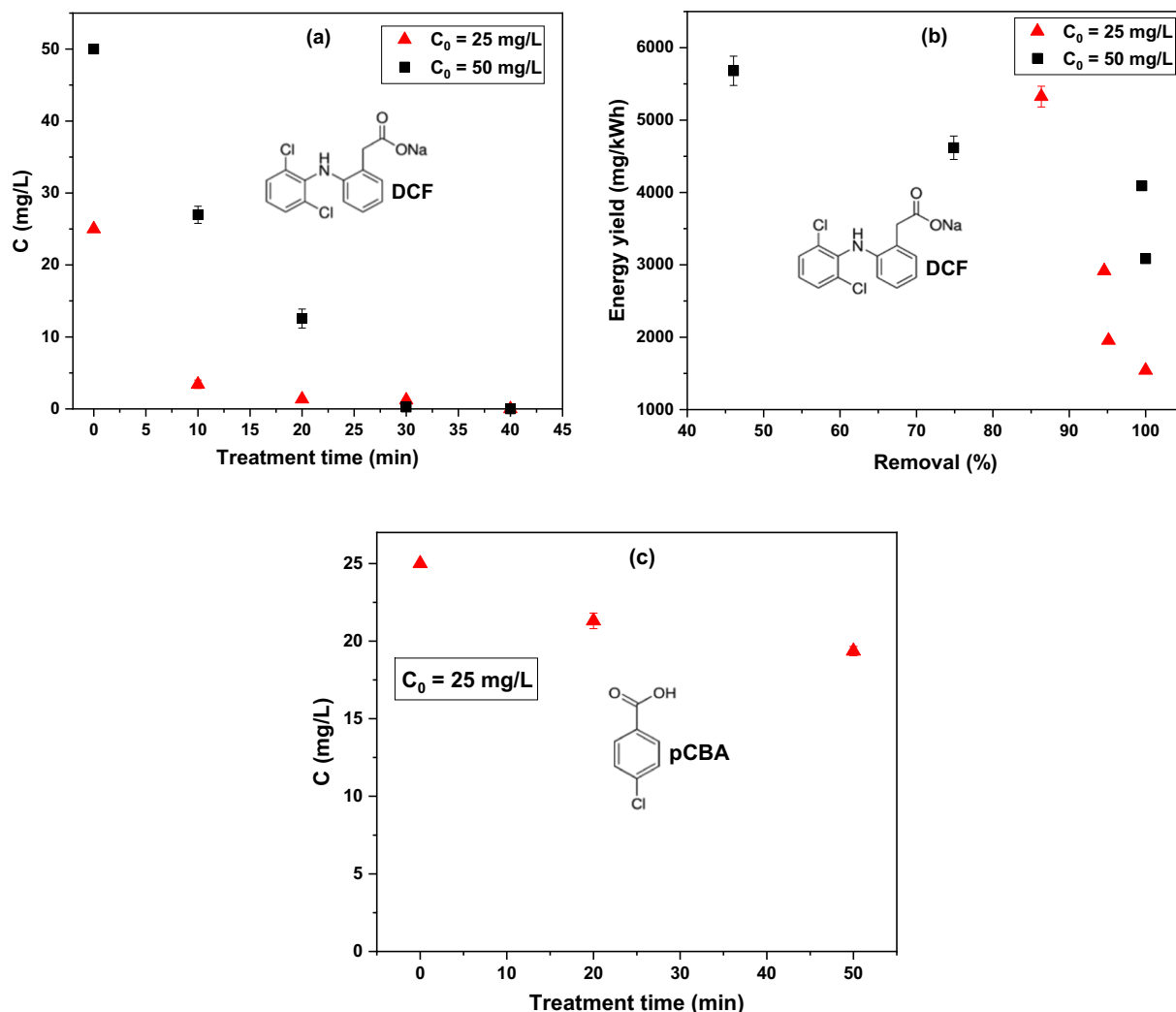


Fig. 7. (a) Change in DCF concentration with treatment time, (b) energy yield vs removal %, (DCF, $C_0 = 25$ mg/L and $C_0 = 50$ mg/L) and (c) change in pCBA concentration with treatment time (pCBA, $C_0 = 25$ mg/L). Experimental conditions: $V_0 = 250$ mL, argon flow of 2 slm, P_{mean} at the sample 8 W.

approximately one order of magnitude higher in multi-needle electrodes-APPJ than in pin-APPJ. This can be attributed to the increased plasma-liquid contact caused by three jets and a large initial volume, which results in a larger energy yield. In case of pCBA removal, the difference in energy output obtained with both plasma sources was not statistically significant.

Authors, for example, Dobrin et al., 2013 reported 1000 mg/kWh for 50 % and 760 mg/kWh for energy yield 90 % removal during the degradation of DCF by using pulsed corona discharge, where the plasma was formed over a liquid surface. Deng et al., 2021 used dielectric barrier discharge (DBD) plasma and obtained 2458 mg/kWh for 50 % and 1332 mg/kWh for 90 % DCF removal. According to Back et al., 2018, after the treatment of DCF with DBD, energy yield values varied from 20 to 715 mg/kWh for removal up to 90 %. When compared to our work, where DCF treatment was performed with the three jets-recirculation system, published data show relatively low DCF removal energy yield. In case of pCBA treatment, Lesage et al., 2014 explored the DBD-recirculation system and plasma was generated over the thin falling film, they found 624 mg/kWh of energy yield for 50 % removal. In the study of Schönekerl et al., 2020, degradation of pCBA was carried out with a plasma-multi electrodes system, as a result, 320 mg/kWh energy yield was obtained for 50 % removal. The reported energy yield associated with pCBA removal in the literature was not significantly different from our results.

If we look at the results, we can see that efficient removal of pollutants with a high energy yield suggests that treatment of some pollutants by

plasma with recirculation could be considered a potential method for wastewater treatment and the technology can also be scaled up for large polluted water treatment.

3.3.3. Degradation products and mechanism of DCF

The use of full-scan ultra-high performance liquid chromatography coupled to Orbitrap MS (UHPLC-Orbitrap-MS) allows the identification of seven TPs (see supplementary, Table S1) during the plasma treatment (by plasma recirculation system): DCF-154 ($C_8H_{10}O_3$), DCF-166 ($C_8H_6O_4$), DCF-202 ($C_8H_{10}O_6$), DCF-241 ($C_{14}H_{11}NO_3$), DCF-259 ($C_{14}H_{10}ClNO_2$), DCF-277 ($C_{14}H_9Cl_2NO$) and DCF-308 ($C_{14}H_9Cl_2NO_3$). The TP analysis data was processed with Compound Discoverer software (Thermo Scientific). Elemental composition and double-bond equivalent (RDB) were selected. In all cases, possible elemental compositions for ions with a deviation of ± 5 ppm of error were assigned (see supplementary, Fig. S1-S7). On the basis of the TPs identified, tentatively DCF degradation pathways were proposed (Fig. 8).

Due to APPJ-liquid-interactions, abundant ROS can be produced such as including hydroxyl radicals ($HO\cdot$), hydrogen peroxide (H_2O_2), singlet oxygen ($^1O_2$) and ozone (O_3) (Cheng et al., 2020; Foster, 2017). Thereby, $HO\cdot$ radicals can cleave DCF in the aqueous solution, leading to the formation of TPs, which could be further degraded and mineralized (Deng et al., 2021; Dobrin et al., 2013; Liu et al., 2019). Fig. 8 shows different reactions mechanisms $HO\cdot$ -mediated such as hydroxylation, dechlorination,

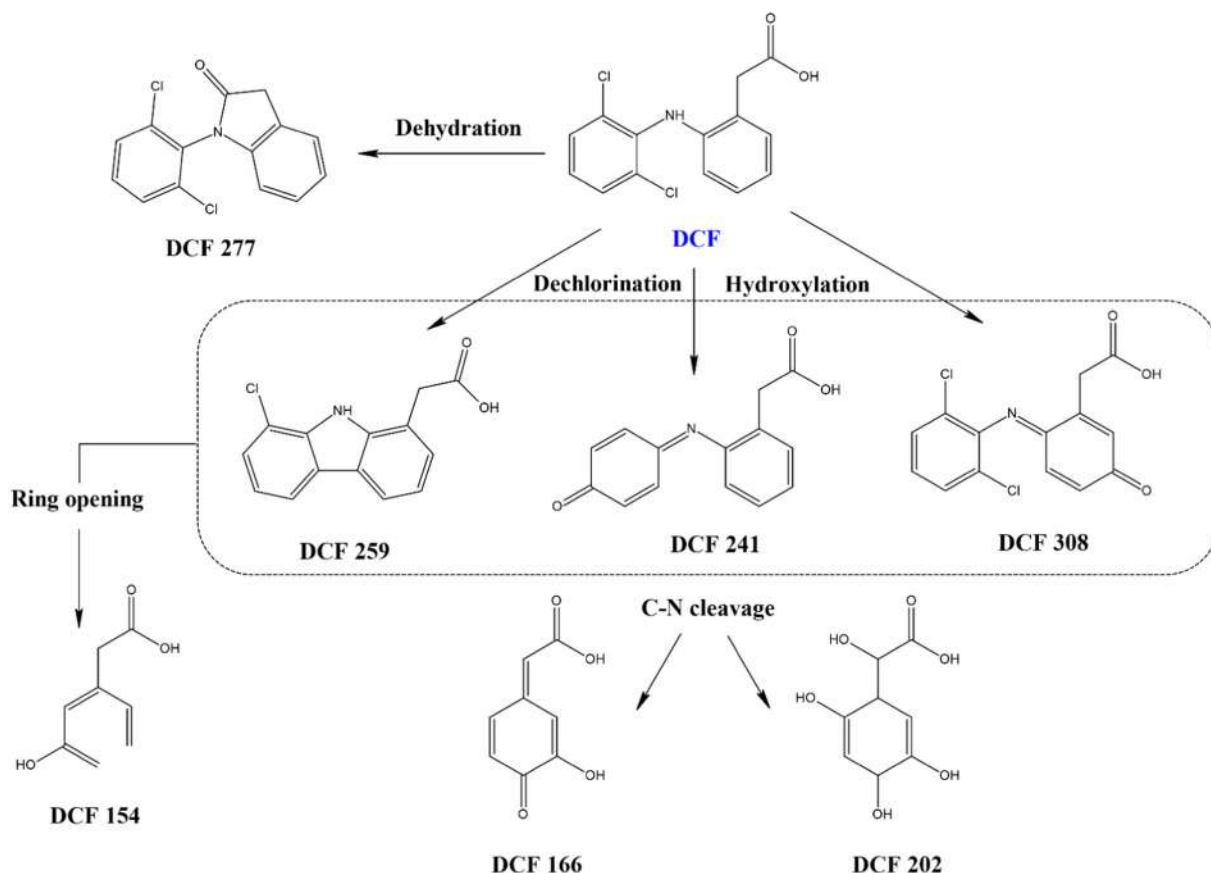


Fig. 8. Proposed possible reaction pathways for degradation of DCF.

cyclization and C—N cleavage, these tentative mechanisms are in agreement with the literature (Banaschik et al., 2018; Peng et al., 2021; Zhang et al., 2020).

The initial reaction of DCF with $\text{HO}\cdot$ to lead to either the abstraction of a hydrogen or the addition of the hydroxyl radical in the electron-donating position of the aromatic ring, DCF could undergo hydroxylation and lead to the production of $\text{C}_{14}\text{H}_{11}\text{NO}_3$ (DCF-241) and $\text{C}_{14}\text{H}_9\text{Cl}_2\text{NO}_3$ (DCF-308), it is known that $\text{HO}\cdot$ addition to benzene ring is kinetically more favored than H abstraction from the ring carbons (Agopcan Cinar et al., 2017). The presence of a Cl-benzyl bond in the aromatic ring of DCF resulted in fast dechlorination under plasma conditions and led to the release of chloride ions with reaction Gibbs free energies around 84–88 kcal/mol (Agopcan Cinar et al., 2017). Two dechlorination TPs were identified, $\text{C}_{14}\text{H}_{11}\text{NO}_3$ (DCF-241) and $\text{C}_{14}\text{H}_{10}\text{ClNO}_2$ (DCF-259). DCF-259 showed a transformation route involved the cyclisation into a carbazole derivative and loss of Cl (Salgado et al., 2013).

The mechanism suggested for the formation of DCF-277 ($\text{C}_{14}\text{H}_9\text{Cl}_2\text{NO}$) starts with cyclization and is followed by water elimination. According to Agopcan Cinar et al., 2017, nitrogen attack on the carbonyl carbon results in formation of the cyclic intermediate. DCF-277 was also identified in Sono-activated persulfate oxidation (Monteagudo et al., 2018).

Subsequently, the cleavage of C—N bonds could occur and form low molecular weight byproducts including $\text{C}_8\text{H}_{10}\text{O}_3$ (DCF-154), $\text{C}_8\text{H}_6\text{O}_4$ (DCF-166) and $\text{C}_8\text{H}_{10}\text{O}_6$ (DCF-202). The cleavage of the C—N bond can be facilitated by the attack of reactive species at the amino group in DCF and breaking the bridge of aromatic rings. Degradation of DCF could continue by cleavage of the ring and the formation of small molecules.

4. Conclusion

In this study pin-APPJ and multi-needle electrodes-APPJ sources were utilized as chemical free AOPs approaches for degradation of DCF and

pCBA in water. We have constructed the 3-pin APPJ to be able to treat larger volumes and in flow regime. Both plasma sources operated in argon as a working gas and plasma was in contact with contaminated samples during the treatments. We have performed electrical characterization and obtained power deposited in the plasma in contact with liquid. This was necessary because power given by the power supply is not the same as power actually deposited in the discharges due to the losses in the system. The power deposited in the part of the discharge in contact with sample was needed for more precise calculation of energy yield. OES and ICCD imaging showed the presence and spatial distribution of excited species (for example $\text{HO}\cdot$, $\text{O}\cdot$, N_2 (SPS), argon excited species etc.) that are major initiators of the reactions in the liquid phase.

Pollutant degradation was efficient by using both plasma sources with DCF degrading much faster than pCBA. For both contaminants, the degradation was more efficient with 1-pin APPJ due to the much smaller volume of the contaminated sample. The results showed that the multi-needle electrodes-APPJ treatment was more energy-efficient and the energy yield varied by the magnitude difference. The maximum energy yield values at 50 % elimination were 6465 mg/kWh and 4036 mg/kWh for DCF concentrations of 25 mg/L and 50 mg/L, respectively. The energy yield for pCBA removal was much lower. With the introduction of recirculation of contaminant, plasma created RONS had more time to react. As a result, the plasma-recirculation system has the most substantial impact to the degradation process.

The elemental composition of transformation products of DCF was identified by high-resolution Orbitrap-LC-MS. The tentative molecular structure of transformation products was analyzed and compared to the literature. The DCF oxidation routes are described and products that formed after various reaction processes including hydroxylation, dechlorination and cyclization are reported based on the chemical formula and molecular weight.

Overall, the findings of this study indicate that cold plasma technology can be efficient option for removing organic pollutants from water that are

extremely difficult to degrade in conventional WWTPs. A multi-needle electrodes-APPJ recirculating system implemented in the research has the ability to enhance plasma and liquid interaction.

CRedit authorship contribution statement

Conceptualization A.K. N.P., W.G., N.S.; writing – original draft preparation A.K., review and editing A.K., N.S., N.P., W.G., O.J., A.P., S.Z., E.C.L., M.J.F.; supervision N.P., W.G.; project administration N.P., W.G. All authors have read and agreed to publish the manuscript.

Data availability

Data will be made available on request.

Declaration of competing interest

The authors state that they have no known competing financial interests or personal ties that could have seemed to affect the work reported in this study.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2022.161194>.

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Plasma water treatment for PFAS: Study of degradation of perfluorinated substances and their byproducts by using cold atmospheric pressure plasma jet

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ABSTRACT

This study evaluates the effectiveness of non-thermal plasma at atmospheric pressure (NTP APPJ) for treating PFAS - contaminated water in different matrices. Successful removal of several perfluoroalkyl carboxylic acids (PFCAs) (C6 to C4), perfluoroalkane sulfonic acids (PFSAs) (C8 to C4) and perfluoropolyethers (PFPEs) (GenX and ADONA) PFAS compounds was achieved in laboratory scale experiments. In the deionized water (DW), high removal efficiencies (> 90%) were observed for longer-chain PFAS, PFOS (99.89%), PFHxA (94.61%) and ADONA (94.83%), while shorter-chain compounds had lower removal rates. Real water samples (tap water and synthetic effluent) showed lower overall degradation percentages (8–50%) depending on compound and matrix. Short-chain PFAS displayed around 10% removal in tap water, while PFOS and GenX achieved 50% and 32% removal, respectively. Complex matrix effects influence degradation rates. Byproducts from the plasma treatment were investigated, revealing distinct degradation mechanisms for various PFAS compounds. For PFSAs and PFCAs, degradation involved electron transfer, bond breaking and subsequent reactions. Conversely, ADONA and GenX degradation initiated with ether-group cleavage, followed by additional transformation processes. Plasma-based technology shows potential for degradation of PFAS, especially for newer substitute compounds like ADONA and GenX. However, further research is needed to optimize plasma performance for complete mineralization of PFAS. This study also proposes a degradation mechanism for ADONA, marking a novel investigation into ether-group PFAS degradation with potential implications for further research and understanding toxicological implications.

1. Introduction

The dispersive occurrence of per – and polyfluorinated substances (PFAS) in the environment and the impact on human and ecological health are raising concern and social awareness in the last few decades [2,52]. These organic molecules, so called “forever chemicals” are part of a man-made production since the 1950 s and are used on everyday basis for commercial and consumer purposes. PFAS, as a complex group of substances with over 4700 identified chemicals, have varying properties, but one thing in common – incredibly strong and stable carbon-fluorine bond which is very difficult to break down. Fluorinated

carbon chain or “tail” can vary in length chain; therefore, substances are sometimes divided into short-chain and long-chain PFAS. On the one end (or both) polar or ionizable “head” group is attached which also can be variable, like carboxylic and sulfonic acid [8]. This structure provides them with unique physicochemical properties, high thermal and chemical stability, repellent to water, oils, and grease. Because of such properties and variability in structure, they can be used for diverse application, such as electronics manufacturing, aqueous film-forming foams (AFFF), dust suppressions, hydraulic fluids, textile stain repellents, food containers, cleaning products, polishes, waxes, paints, cosmetics, etc. Barzen-Hanson et al., [3,37,49].

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The reports on PFAS presence in drinking water near chemical plants and blood serum samples of humans and animals from the areas that are close to fluoropolymer production facilities are known for a few decades, but not until studies about findings of PFAS in remote areas, certain global concern increased and PFAS have been started to be monitored worldwide [4,34,38]. Those were one of the first proves that these substances are very persistent and even though the most important input of PFAS to the environment comes from direct release from industrial facilities, and indirect source is also affective, from different precursors as degradation products, with long-range transport potential. Other studies had shown that their bioaccumulation in drinking water sources and potential to accumulate in the dietary products and humans are related to serious health effects, such as immune suppression, thyroid disease, cancer, significant (chronic) toxicity of some [20,25,33]. Since 2009, one of the most known compounds to be used and present in the environment, perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) were recognized as potentially dangerous compounds for human health and the environment, and have been regulated throughout United States and Europe [12,45]. In 2022, The European Commission adopted the proposal to revise the list of priority substances in surface water and groundwater where it stays that sum of 24 PFAS the groundwater quality standards are 0.004 $\mu\text{g/L}$ [13]. Therefore, global production of PFOA and PFOS started to be restricted or banned while new fluorinated substances, as replacement, were developed [47]. These new substances are mostly part of short-chain PFAS group, such as perfluorobutane sulfonic acid (PFBS) with C4 chain-length. Another interesting group are perfluoropolyethers (PFPEs), or shortly ether-PFAS, which include (among others) substances named hexafluoropropylene oxide dimer acid (HFPO-DA; GenX), dodecafluoro-3 H-4,8-dioxananoate (ADONA) and 6:2 chlorinated polyfluoroalkyl ether sulfonate (6:2 Cl-PFESA; F-53B). More detailed discussion can be found in [47] and [30]. While, because of the alkyl-ether (C-O-C) addition in their chemical structure, higher mobility properties and less bioaccumulation can be expected, environmental impact and toxicity is still not certain, yet concerning. Recent studies shows that even though they do not have a high potency to accumulate in aquatic species, their presence in water (drinking and groundwater) has more impact on animals and people that are consuming it and are exposed to them [51]. [31] has reported GenX presence in human serum and fish muscle while ADONA has been detected in blood donor samples in Germany [14]. Levels of ADONA in surface water and its toxicity have been investigated by [17], while GenX has been found in rivers of The Netherlands and Germany [15,21]. Global distribution of these substances has also been reviewed by [32]. Toxicological data can be found in [7] as well.

In terms of wastewater treatment processes, many studies and practice had shown that most PFAS are highly recalcitrant to conventional wastewater treatment, but also to many advanced oxidation processes (AOP). Adsorption on activated carbon and ion exchange methods are the most common water treatment technologies that have been used for PFAS removal from water media [9,10,50]. However, the search for more efficient technologies that do not require high energy input, waste management and long treatment time is needed. Few AOPs have proven to be effective in their removal from water, such as electrochemical oxidation [11], sonolysis [16], photocatalysis [26]) and among them, plasma treatment technologies with somewhat different configurations of plasma sources than one presented here in this paper [35,36,41]. Non-equilibrium or non-thermal plasma (NTP) is based on the production of highly reactive oxygen and nitrogen species which are generally needed for an effective water treatment, namely atomic hydrogen and oxygen, hydroxyl and hydroperoxyl radical, nitrogen-species, aqueous electrons etc. [1,5,6]. The main advantage of this technology is that only the energy applied to the working gas (air, argon, helium, mixtures etc.) is needed for plasma generation. Since plasma contains such a rich and reactive chemistry, it became of great interest in terms of use for removal of very recalcitrant compounds [22,

28,48]. In our recent review paper, we have summarized the current knowledge on the chemistry and degradation pathways of OMPs by using different NTP types, including PFAS [44]. [42] was investigating degradation of PFOA and PFOS substances in groundwater samples. [39] was using landfill leachate in experiments for the investigation of several different PFAS substances with plasma jet above liquid with formation of bubbles. While current studies (and more that have been mentioned in review paper) shown high removal efficiency and promising results in reactors of around 1 L, it should be noted that most of the studies have been investigating PFOS and PFOA substances and up to this date according to the authors' knowledge, a similar investigation has not been done in terms of plasma treatment of fewer known compounds, ADONA and GenX. A few of them mentioned more complex matrices but with still limited information on PFAS byproducts after plasma treatment. [40] had done interesting work in breakdown products of PFAS in plasma-based water treatment processes, PFOA and PFOS compounds.

In this work, NTP in gas-liquid environment generated with pin-type electrode setup at a laboratory scale has been used for the removal of six substances which were selected as a representative of groups of perfluorinated compounds, carboxylates, sulfonates, and ethers, in order to study plasma application on different groups of PFAS. The removal efficiency of each substance, individually, was assessed in 3 different matrices: distilled water (DW), tap water (TW), wastewater or secondary effluent (SE), for the same experimental conditions. The influence of NTP treatment on different working parameters was also observed. In addition to that, in all cases, liquid phase byproducts were studied using HRMS, with more attention on novel substances such as ADONA. The first attempt of proposed structures of studied byproducts of GenX and ADONA as well as degradation mechanism of ADONA have been given.

2. Materials and methods

2.1. Chemicals and standards

Standards for substances that were used in experiments, perfluorooctanesulfonic (PFOS), nonfluorobutane-1-sulfonic acid (PFBS), perfluorobutyric acid (PFBA), undecafluorohexanoic acid (PFHxA) were purchased from Sigma Aldrich. Hexafluoropropylene acid (HFPO - DA or GenX) and dodecafluoro-3 H-4,8,-dioxanoate (ADONA) were purchased from Wellington laboratories. All solutions were prepared in Milli-Q water at room temperature ($\pm 25^\circ\text{C}$). High purity methanol and Milli-Q water used for mass spectrometry analysis were purchased from Thermo Fischer Scientific, while ammonium acetate used for mobile phases was obtained from VWR. The chemicals used were of either analytical grade or high purity (>98%). Details of the compounds such as structures, molar mass etc. can be found in Table S1 of Supporting Information (SI).

2.2. Analytical methods

The analytical method used for target PFAS analysis was based on the method reported from [27] with a few alterations. A 5500 QTRAP hybrid triple quadrupole-linear ion trap mass spectrometer (QqLIT-MS) with a turbo Ion Spray source (Applied Biosystems, Foster City, CA, USA) coupled to a Waters Acquity Ultra-Performance™ liquid chromatograph (UPLC) (Milford, MA, USA) was used. For the chromatographic separation, an Acquity UPLC C18 column (50 mm $\times$ 2.1 mm, 1.7 μm) was used coupled with Phenomenex Luna 5 μm precolumn C8 (50 mm $\times$ 3 mm, 100 Å) to minimize contamination from mobile phases. The chromatographic separation used aqueous solutions of ammonium acetate (5 mM) (A) and methanol (B). The established flow rate was 0.4 mL min⁻¹, and the elution gradient condition was finalized at 90% of B and 10% of A. The sample injection volume was 10 μL , and the limit of detection (LOD) for this method was determined to be 0.1 $\mu\text{g L}^{-1}$. Table S2 (SI) summarizes the optimized compound-dependent

parameters of the MS.

Liquid chromatography – High resolution Mass Spectrometry (LC-HRMS) methodology using Orbitrap Exploris 120 (OE120) was performed to identify possible degradation byproducts of all compounds. EPA Method 57 was adapted from the EPA Thermo Scientific source with some minor modifications. As a mobile phases, 5 mM ammonium acetate (A) in water and methanol (B) were used. Final conditions consisted of 90% (A) and 10% (B) with a flow rate of 0.4 mL min^{-1} and pressure of 195 bar. The injection volume was $5 \mu\text{L}$. The data was processed using the Compound Discoverer (CD) software (Text S2(SI)) from Thermo Scientific, which is designed for non-target screening of all types of organic compounds. General instrument parameters can be found in Table S3 (SI).

2.3. Plasma treatment of PFAS setup

2.3.1. Plasma source configuration

The treatments of PFAS-polluted water samples were performed using a non-thermal plasma (NTP) treatment with an atmospheric pressure plasma jet (APPJ) source. Electrical characterization of this device is presented in [23] and [24]. Laboratory-scale APPJ source configuration is shown schematically in Scheme 1 Figure S1. The APPJ consisted of a stainless-steel high-voltage (HV) electrode (1 mm in diameter) inserted inside a ceramic tube, which was then inserted in a glass tube with outer and inner diameters of 6 and 4 mm, respectively. The electrode was connected to a commercial HV RF power source (T&C Power Conversion AG0201HV) with a frequency of 332 kHz. In the experiments, argon was used as a working gas, with a flow rate of 1 standard liter per minute (slm). The argon flow rate was controlled by mass flow meter (OMEGA, FMA5800/5500). Time-variable current and voltage signals were monitored with a Tektronix oscilloscope (MDO3024 model), and a HV probe (Tektronix 6015 A) was used to determine the voltage at the HV electrode. The glass sample vessel ($\varphi = 55^\circ \text{mm}$) had copper tape at the bottom and it was grounded via a resistor of $R = 1 \text{ k}\Omega$ for current monitoring. The streamer type discharge was generated above the liquid in a 10 mm gap between the tip of the powered electrode and the surface of the liquid sample.

2.3.2. Water samples treatment experiments

Individual solutions of PFOS, PFBS, PFBA, PFHxA, GenX, ADONA were prepared in different types of water matrices: distilled water (DW), tap water (TW) and secondary effluent (SE) with initial concentration of $100 \mu\text{g L}^{-1}$. In all cases, 10 mL of solution was subjected to the plasma

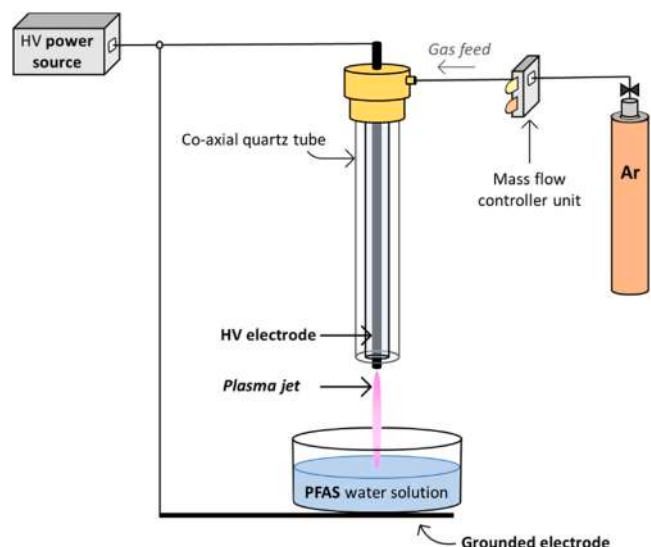
treatment for different time intervals of 3, 5, 7 and 10 min. After the plasma treatment, samples were taken to investigate the degradation profile through target analysis and for the study of byproducts through non-target analysis. Measurements of pH, conductivity, and temperature for both control samples and each plasma – treated sample were conducted. Control samples consisted of untreated solutions in DW, TW and SE. DW served as a model to explore optimal removal conditions without matrix interference. TW and SW allowed us to assess PFAS behavior and degradation efficiency in more complex environments. Additionally, the impact of plasma treatment on various parameters under the same initial concentration and treatment duration as in DW were examined. Furthermore, the volume of all samples was measured after each treatment.

3. Results and discussion

3.1. Degradation of PFAS in distilled water

The removal efficiency of each investigated PFAS in DW during 10 min of plasma treatment is shown in Fig. 1.

As it can be seen, after 10 min of treatment time, removal efficiency of PFOS, PFHxA and ADONA reached over 90%. The removal efficiency trend for all compounds is as follows: PFOS (C8) > PFHxA (C6) > ADONA (C7) > PFBA (C4) > GenX (C6) > PFBS (C4). It can be observed that the longer the chain-length of the compound, the higher the efficiency for the same treatment time. As a result of the structure of PFAS compounds, surface activity increases with chain length, which means that long-chain PFAS will have better contact with the plasma at the plasma-liquid interface and faster degradation. Short-chain PFAS tend to accumulate less at the surface therefore longer treatment time is needed. The fastest degradation occurred at the beginning (between 3 and 6 min) for all studied PFAS, followed by a slow increase. It is possible that slow degradation is due to the presence of byproducts and depending on the compound, those byproducts are produced in different treatment times. The trend of rapid degradation is more pronounced for three compounds with longer chain length (PFOS, ADONA, PFHxA)



Scheme 1. Configuration of plasma system.

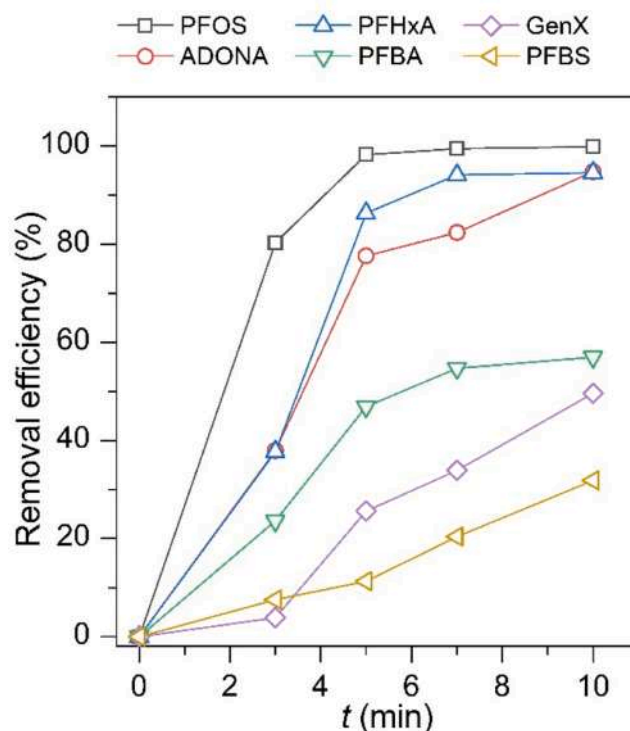


Fig. 1. Removal efficiency of PFAS in DW.

when compared to GenX, PFBA and PFBS. The lowest degradation was observed for PFBS with only around 30% removal efficiency in 10 min. For the same treatment time, it took around 57% for PFBA, which has the same chain length as PFBS. Compared to sulfonates, carboxylates are harder to degrade which has been shown in previous studies [39,41]. Most of the results in this paper shown the same consistency, only not in the case of PFBS and PFBA. However, it seems that the hydrophobicity of PFBA is slightly higher than PFBS when we take their values of $\log K_{ow}$ into account which can explain higher removal efficiency of PFBA. According to the results, high removal efficiency occurred in the case of ADONA (around 95% in 10 min) while for GenX was 50% for the same treatment time. From the compound properties (Table S1, SI), it can be seen that ADONA has CF_3O extra group which can give higher surface activity to the molecule.

A few parameters were measured during the treatment of the solutions. Fig. 2a and b show the pH, conductivity, and temperature measurement results (average values). Additionally, the treated water volume was also measured at the end of each treatment. The same change in pH was observed for each compound in all experiments. The initial pH was between 6 and 7.5, while after 10 min of treatment time, the pH value decreased to around 3. The pH decreased drastically at the beginning of the treatment (in the first two min), and then varied around 3 for the rest of the treatment. The reduction in the pH value could be attributed to a generation of some reactive species. Although argon was used as a working gas, some nitrogen species could occur in the solution due to plasma contact with ambient air [24,29]. Additionally, byproducts resulting from the degradation such as sulfuric acids and the production of few other plasma species, H^+ and hydrogen peroxide, could contribute to an acidic environment and lead to decrease in pH [29].

The measured initial conductivity of solutions was around $4 \mu S cm^{-1}$ and it increased linearly in all experiments. After 10 min of treatment time, it was around $400 \mu S cm^{-1}$. The trend suggests that with longer treatment time, conductivity is expected to increase. What was indicated in some previous work ([39,41,46]) in plasma-liquid systems, conductivity of water tends to increase due to interaction with electrons formed in the discharge which can break water molecules into ions and ionic species produced in plasma are transferred into liquid, hence, more ions are present in water. While some authors indicated negative correlation between the degradation rate of PFAS and extremely high conductivity (above $20 mS/cm$) assuming high concentration of ions reduced the contact area with contaminated water [41], other indicated that conductivity did not affect the degradation in significant way or even have a positive impact [46]. Therefore, for precise assessment it should be considered which type of discharge was involved and what the level of conductivity was. In our experiments, the degradation rate for all compounds decreased over time and this was probably caused by the competition between byproducts and parent compound to react with

plasma species and not due to the changes in conductivity which reached $400 \mu S/cm$ and could be considered as low.

Volume of the samples was measured after the treatment to observe possible loss due to evaporation. The volume loss after 10 min treatment time was 10% and this was taken into account by normalizing the data for all samples. To investigate the evaporation we measured temperature in the bulk of the solutions. The temperature of the solution gradually increased under plasma exposure, with a maximum of $45^\circ C$ in some cases. The increase in temperature over treatment time indicates that surface processes were dominantly governing the evaporation and not the heating of the sample volume. One of the possible mechanisms was the local heating at the point of contact of the plasma streamer and water due to the higher local temperature (still below $100^\circ C$). Another mechanism was the change in the partial pressure in the interface gas/liquid region due to argon flow and the plasma. This localized change in the partial pressure leads to evaporation of the treated liquid sample. The rise in the temperature, as shown in Fig. 2, is not linear and the heating slows down never going above $50^\circ C$, which indicates that degradation of contaminants is purely due to non-thermal processes.

3.2. Degradation of PFAS in complex matrices

In terms of PFAS degradation in NTP treatment – gas-liquid interface, studies so far have shown promising results, but most of them were conducted in pure water matrices, with PFOS and PFOA as representatives. However, in some recent study [18] PFAS removal from groundwater (already contaminated with PFAS) using plasma has been investigated. Singh et al., [39] has investigated PFAS removal by using landfill leachate sampled and showed the potential of plasma technology for treatment of PFAS contaminated water. In this work, the removal efficiency in TW and SE has been assessed for all 6 substances as in DW, as a function of the treatment time. Results have been given in Fig. 3a and b.

In both type of matrices, lower removal efficiency was observed in comparison to experiments in pure water. Short-chain PFAS (PFBS, PFBA and PFHxA) showed almost linear degradation and very low percentage of removal efficiency (less than 10%) with respect to PFOS, GenX and ADONA. A major difference was in the case of PFHxA where efficiency was around 94% in DW while in TW and SE it was around 6% and 9%, respectively. With the complexity of the matrix, the removal efficiency is changing, especially for compounds that do not easily migrate to the liquid surface.

Fig. 4a. shows trend of the pH (average values) of the TW and SE samples during NTP treatment. The initial pH for both TW and SE matrices were around 8.3 and this value did not significantly change with plasma treatment even for the longest treatment time of 10 min. It should be noted that pH is not necessarily dependent on the main

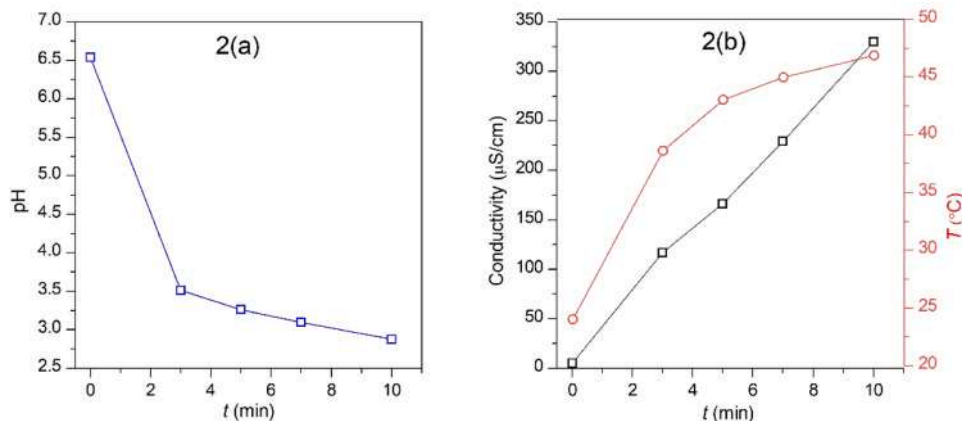


Fig. 2. (a) and (b). Dependency of pH, conductivity and temperature during treatment time of PFAS (total average value).

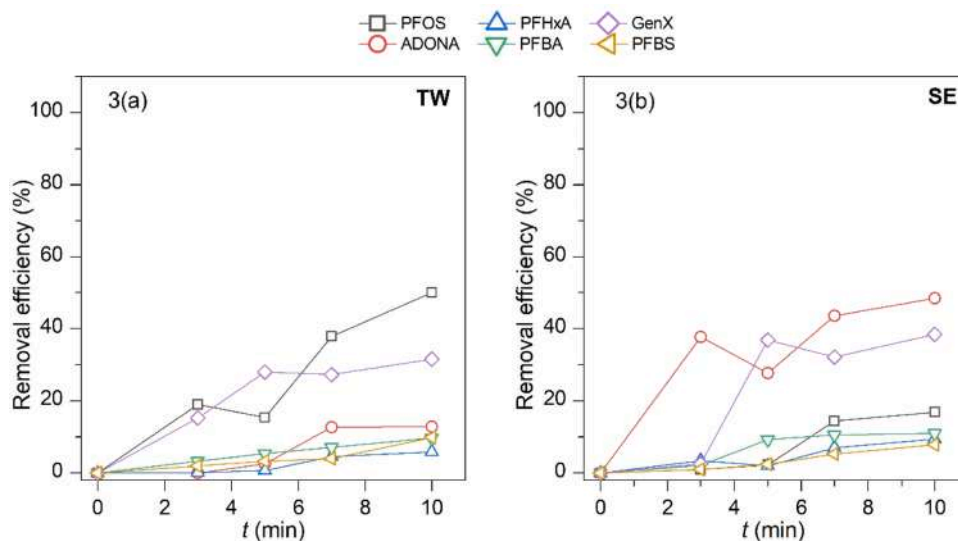


Fig. 3. (a) and (b). Removal efficiency of PFAS in TW and SE.

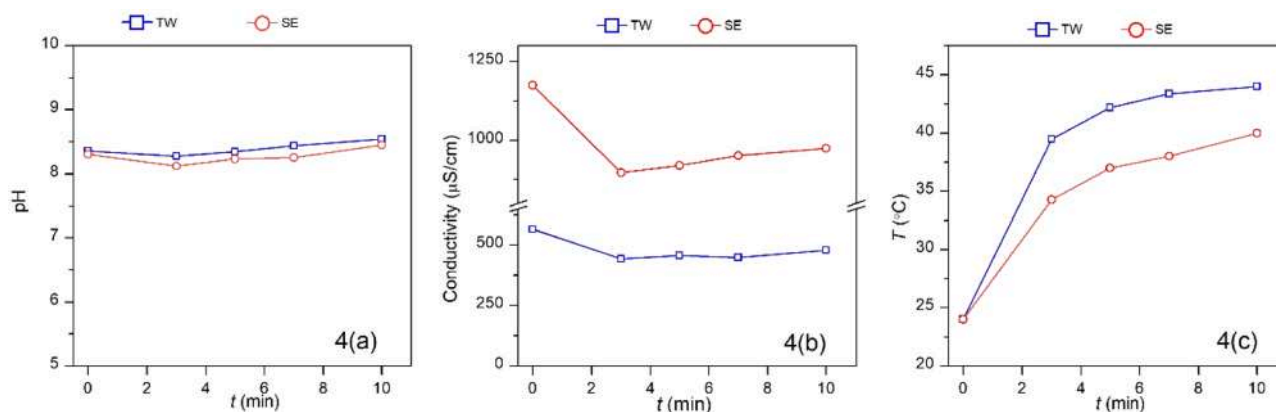


Fig. 4. (a), (b) and (c). Dependency of pH, conductivity and temperature during treatment time of PFAS in TW and SE (averaged values obtained from all samples).

compounds that were investigated. In fact, the pH did not drastically change when PFAS compounds were added to all solutions. Considering the chemistry of water samples, it is possible that in real water samples the concentration of basic products such as carbonate anions calcium and potassium (which were not present in DW) is much higher compared to the expected acid products of PFAS, hence the neutral to base value of pH.

The changes of the solutions conductivity (Fig. 4b) are also diverse when compared to DW. Generally, the conductivity was higher in TW (initial around 500 $\mu\text{S}/\text{cm}$) and SE (initial around 1200 $\mu\text{S}/\text{cm}$) than in DW (5 $\mu\text{S}/\text{cm}$). In the case of all compounds, the trend shows a small decrease at the beginning of the treatment followed with a slight increase in the end of the treatment, in both TW and SE. However, in the case of SE, the decrease is more significant than in TW. Since the solutions are much more complex from the chemical point of view, the matrix definitely impacts their conductivity. Also, it can be concluded that plasma treatment did not significantly affect the water conductivity in real water samples. As for the temperature, it can be seen (Fig. 4c) that the trend was very similar to the one in DW. At the end of the treatment, the maximum volume loss was around 7%, which indicates that the same conclusion related to evaporation mechanisms could be drawn as in the case of DW samples.

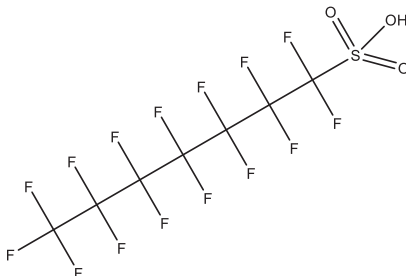
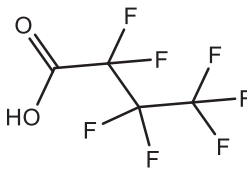
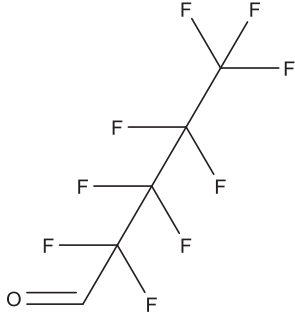
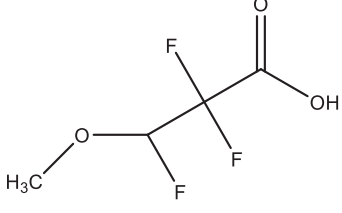
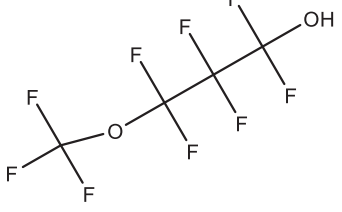
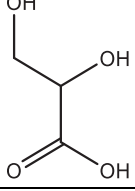
3.3. PFAS byproducts

Few previous studies discussed possible byproducts of PFOA and PFOS by utilizing different plasma configurations, as well as proposed degradation pathways [19,40,42,43]. In this work, the analysis of the transformation products (TP) was performed by using the LC-Orbitrap HRMS for all compounds in all 3 matrices. In total, 6 different byproducts have been found. Table 1. shows proposed structures and observed mass of byproducts as well as their parent compounds and type of matrices where they are detected. TP₂₁₂ has been also reported in [40], while others are reported for the first time in this paper.

3.3.1. Carboxylates and sulfonates byproducts

The cold atmospheric pressure plasma jet (CAP) generates highly reactive species, including e_{aq}^- , $\bullet\text{OH}$, and $\bullet\text{H}$. In water with a neutral pH, PFOS exists as its anion species, capable of swiftly reacting with e_{aq}^- to create the corresponding radical anions. Subsequently, the reactive species could induce the cleavage of C-C, resulting in the formation of TP₄₄₈ (m/z 449.9333). Also, the subsequent reactions with e_{aq}^- and/or $\bullet\text{OH}$ can lead to the formation of perfluorinated acid intermediates, such as TP₂₁₂ (m/z 212.9793) or PFBA. Both have been detected after 10 min of treatment time. According to the previous literature, degradation of long-chain PFAS during plasma treatment involves interaction of argon ions, aqueous and plasma electrons and hydroxyl radicals which lead to the formation of short-chain PFAS. PFBA is known as a common

Table 1Summary of the byproducts with proposed structures, retention time (RT), molecular formula, m/z value.

Parent compound (matrix type)	Byproduct TP mark	Name	Molecular formula	RT	Observed mass m/z	Structure
PFOS (TW)	TP_448	1,1,2,2,3,3,4,4,5,5,6,6,7,7,7-pentadecafluoroheptane-1-sulfonic acid (PFHpS)	$C_7HF_{15}O_3S$	9.87	449.9333	
PFOS (TW)	TP_212	2,2,3,3,4,4,4-heptafluorobutanoic acid (PFBA)	$C_4HF_7O_2$	4.89	212.9793	
PFHxA (DW)	TP_246	2,2,3,3,4,4,5,5,5-nonafluoropentanal	C_5HF_9O	4.80	246.9815	
PFBA (TW) ADONA (TW)	TP_157	2,2,3-trifluoro-3-methoxypropanoic acid	$C_4H_5F_3O_3$	0.54	157.0117	
ADONA (DW, TW)	TP_250	1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propan-1-ol	$C_4HF_9O_2$	6.35	250.9758	
ADONA (TW)	TP_105	2,3-dihydroxypropanoic acid	$C_3H_6O_4$	0.86	105.0192	

byproduct of PFOS, and based on this hypothesis, it should degrade further by losing another $(-CF_2)$ group. Then, the interaction with reactive species degrades into short chain organic acids with the possibility to reach mineralization. In this case, it can be concluded that indeed electrons and argon ions play an important role in initiation reaction by attacking the C-S bond, followed by propagation reaction that

result in the formation of short – chain PFBA. Longer treatment time was needed to detect whether PFBA is present after 10 min and/or it was degraded to some level. In DW samples, no byproducts were detected, which means either PFOS was completely degraded or the level of byproducts was very low. Regarding PFHxA, the $-COOH$ group was targeted by electrons, giving rise to the creation of unstable

perfluoroalkyl radicals through an electron-mediated chain initiation process.

After 10 min of reaction in TW, a byproduct identified as TP_157 (m/z 157.0117) originating from PFBA was detected. This byproduct arises from H/F exchange process. Moreover, the C-C breaks within the molecule can interact with $\cdot\text{OH}$ through intramolecular reaction, ultimately resulting in the formation of an ether compound. The same TP was identified in ADONA samples in TW, which correspond to C-C breaks and defluorination. Since both TW and SE are complex matrices, electrons and ions needed for degradation of PFAS have less ability to react with target compound due to the presence of scavengers such as NO_3^- . This could be a possible explanation for why the same byproducts are not found in different solutions at the same treatment time. Rest of the byproducts that were found in other solutions were TP_246 DW in case of PFHxA and was defined as radical form which was present in 3–10 min treatment time, with slowly decreasing trend after 3 min. Attack of the electrons or argon ions and the OH radical resulted with PFHxA molecule losing ($-\text{CF}_2\text{O}$) group.

3.3.2. Ether byproducts

The identification of byproducts, namely TP_250 (m/z 250.9758) and TP_105 (m/z 105.0192), originating from ADONA, has been established. TP_250 was detected in DW within the 3–7-min treatment, though its presence was no longer detectable after a 7-min treatment. Conversely, in TW, TP_250 persisted from 3 min onward, with a slight decrease noted until the conclusion of the treatment. Additionally, the presence of TP_105 was noted in TW, with its concentration progressively increasing between 3 and 10 min of treatment time.

These results point to several possibilities: *i*) same types of byproducts can be found in different cases, but the higher the complexity, it takes longer times for the detection/formation of byproducts; *ii*) more than one reaction is possible during treatment time; *iii*) level of byproduct concentration is too low in the certain treatment time and does not exclude the presence of the byproduct; *iv*) in DW, byproduct was also degraded during treatment since there was no complex interferences with reactive species. Fig. 5a and b compare ion intensity trend of ADONA as parent compound with detected byproducts in DW and TW.

It was found that a possible byproduct in GenX sample was trifluoroacetic acid TFA ($\text{C}_2\text{F}_3\text{O}_2\text{H}$). But since the same byproduct has been detected in some blank samples, it was impractical to conclude whether TFA is a product or was trapped in the system in MS. Therefore, in this paper the proposal of degradation has been given only for ADONA (Fig. 6) with the verity that the same is possible for GenX based on similar structure of those compounds.

While the carboxylic or sulfonic group is favoring site for direct electron transfer, causing a cleavage of C-C or C-S bond in other PFAS, here, oxidative degradation occurs which causes split of ether-bond (C-O cleavage). With further reactions of decarboxylation, defluorination, HF exchange, TP-250 is converted into TFA. Based on the structure of parent compound and byproducts, it can be seen that C-O cleavage is on the different carbon for TP_157. As for TP_105, it has been defined as a third possible byproduct.

Based on the elucidation of byproducts resulting from PFAS treatment using CAP, can be outlined that the degradation of PFAS in water occurs through electron transfer, giving rise to the formation of corresponding radical anions which means that reactive species trigger chain reactions leading to the cleavage of C-F, C-C, or C-S bonds within PFAS molecules.

4. Conclusion

The results obtained in this work demonstrate the success of application of non-thermal plasma at atmospheric pressure (NTP-APPJ) for PFAS- contaminated water treatment on laboratory scale, for three different water matrices. In DW, high removal efficiency (>90%) was achieved for longer chain PFAS; PFOS, PFHxA and ADONA in 10 min of treatment time while other compounds showed less removal efficiency, but still high for short treatment time. In TW and SE which represent real water samples, overall, percentage of degradation was lower, between 8% and 50%, depending on the compound and matrices. However, while the short chain PFAS in SE showed almost the same percentage as in TW, PFOS was around 16% and GenX and ADONA 39% and 49%, respectively. It can be concluded that degradation is compound-depending and influenced by different matrix effects. What is more important is that the novel compounds, PFAS substances ADONA and GenX have shown promising results and successful removal in plasma system that was used in this work. Overall, results showed that plasma can be successfully used for the treatment of PFAS contaminated water, especially for the novel compounds, ADONA and GenX which are in production and used up to this date.

The focus of this study was also investigation of byproducts produced utilizing plasma-based technology, especially ADONA and GenX. In total, 7 different byproducts were detected. The results proved one of previous hypothesis, that, in case of PFSAs and PFCAs compounds, electron transfer was important initiation step of PFAS degradation, breaking of the C-C bond or C-S bond on the head group. Degradation was then followed by decarboxylation, defluorination, hydrolysis and reaction with OH radical. As for ADONA and GenX, initial break of the bond is on the ether group, possibly followed by process of

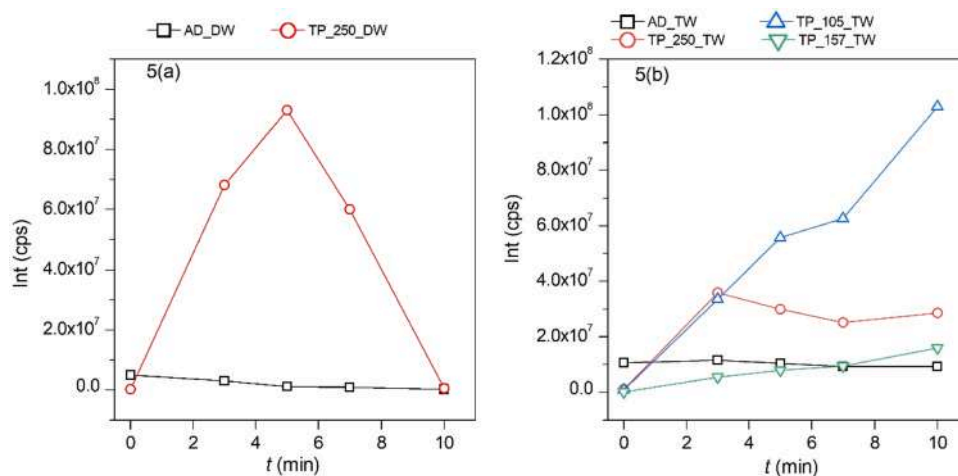


Fig. 5. (a) and (b). Ion intensity (counts per second - cps) of MS signals of ADONA and byproducts: TP_250 (red line/circle) in DW (5a) and TP_250 (red line/circle), TP_105 (blue line/upper triangle) and TP_157 (green line/down triangle) in TW (5b).

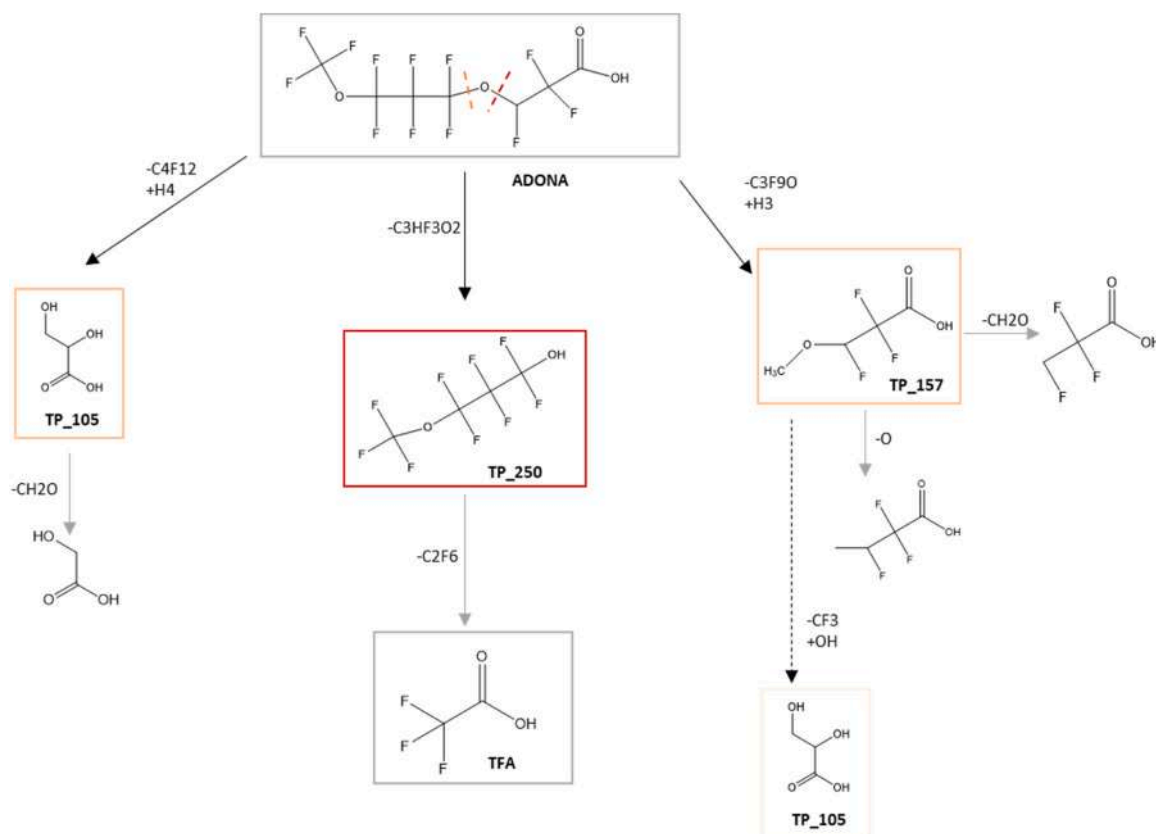


Fig. 6. Identified products of ADONA and possible degradation pathway.

defluorination and decarboxylation. It can be concluded that both reduction and oxidation processes are involved in PFAS degradation, and the mechanism can vary in different compound types. Therefore, application of plasma seems to be a good approach, but more investigation is needed for attaining better performance with the aim to achieve complete mineralization which is needed for PFAS case.

A degradation mechanism of ADONA is proposed in this paper. According to authors knowledge, this was the first attempt to investigate and propose the degradation of ether-group PFAS, ADONA and GenX in plasma-based processes. Therefore, further investigation is needed to demonstrate detailed pathway. Authors also suggest further investigation in terms of toxicity of newly investigated intermediates.

CRedit authorship contribution statement

Elisabeth Lumbaque: Writing – review & editing, Formal analysis. **Nikola Skoro:** Writing – review & editing, Validation, Resources. **Nevena Puac:** Writing – review & editing, Validation, Resources. **Olivera Jovanovic:** Investigation, Formal analysis. **Barbara Topolovec:** Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Formal analysis, Conceptualization. **Mira Petrovic:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jece.2024.112979](https://doi.org/10.1016/j.jece.2024.112979).

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PAPER

A comparison of power measurement techniques and electrical characterization of an atmospheric pressure plasma jet

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A comparison of power measurement techniques and electrical characterization of an atmospheric pressure plasma jet

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Abstract

In the last two decades a growing interest has been shown in the investigation of atmospheric pressure plasma jets (APPJs) that operate in contact with liquid samples. In order to form a complete picture about such experimental systems, it is necessary to perform detailed diagnostics of plasma jets, as one step that will enable the adjustment of system properties for applications in different areas. In this work, we conducted a detailed electrical characterisation of a plasma system configuration used for water treatment. A helium plasma jet, with a pin electrode powered by a continuous sine wave at a frequency of 330 kHz, formed a streamer that was in contact with a distilled water sample. An electrical circuit allowed the monitoring of electrical signals supplied to the jet and also to the plasma itself. An electrical characterisation together with power consumption measurements was obtained by using two different methods. The first method was based on the direct measurements of voltage and current signals, while in the second method we used ‘Lissajous figures’. We compared these two methods when used for discharge power estimation and addressed their advantages and limitations. The results showed that both of these methods could be used to successfully determine power consumed by a discharge in contact with water, but only when taking into account power dissipation without plasma.

Keywords: APPJ, electrical characterization, discharge power, Lissajous figure

(Some figures may appear in colour only in the online journal)

1. Introduction

Over the past decades, non-equilibrium atmospheric pressure plasmas have been extensively investigated, because they represent a powerful tool in various applications. In the treatment of thermally sensitive and unstable materials, especially in biomedicine, the key asset is plasma operating at room temperature [1–4]. Besides biomedical applications, these plasmas are also used in agriculture and water treatment technologies [5–10]. Nevertheless, in all cases, the potential of cold plasma has been used to create specific particles and form a chemically active medium without additional chemical reagents.

APPJs are sources of non-equilibrium plasmas that can be made in various geometries and electrode arrangements.

They usually consist of a dielectric tube, a powered electrode, that can be additionally isolated, and a grounded electrode. Depending on the geometry of the electrodes, operation frequency, power source and working gas, different types and modes of discharge can be generated. In all configurations, the discharge is created when the gas at atmospheric pressure is exposed to a strong electric field. These plasmas are called non-equilibrium, owing to the different temperatures, i.e. energies, of the particles [11].

In the case of APPJs, the realized discharge can be a classical streamer or a more diffuse discharge. Discharges generated by dielectric barrier discharge (DBD) devices can be spatially homogeneous and diffuse [11, 12] while those discharges generated by plasma jets are usually streamers or streamer-like discharges. The streamer discharge occurs after the streamer breakdown. The formed electrical field

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accelerates the charged particles providing energy to the plasma. The presence of an electric field causes the ionization in the gas gap and the formation of space charges at the head of the ionization wave. The field amplification owing to the space charge in the head leads to self-extending streamer discharge. Thus, after inception, the streamer propagation and properties depend on the system configuration, gaseous surrounding and the target. The energy of the fast-progressing streamer is mostly dissipated into the excitation of the working gas [13, 14]. In case of APPJs this is demonstrated through the propagation of PAPS (Pulsed Atmospheric pressure Plasma Streamer) or, so called, 'plasma bullets' [15–17]. Streamers are also present in other types of plasma sources. When the discharge current is not limited they precede other types of discharge, such as sparks and lightning [18].

In several publications it has been investigated how the configuration of a plasma jet system influences the properties of plasma. It has been shown that the efficiency of the power transfer process from the power supply to the plasma system determines the mode of the discharge [14, 19, 20]. For example, a comparison of two discharge regimes produced by different excitation sources (direct current (DC)-pulsed and sine-wave alternating current (AC)) in the same single electrode plasma jet was reported. It was found that the excitation source determines discharge current, the length of plasma plume and plasma temperature [21]. The grounded electrode, due to its capacitive nature, can additionally lead to the energy loss influencing behavior of the plasma bullet. But at the same time it can increase the local electric field that will result in lower voltages needed to sustain plasma [14]. Control of these plasma properties is essential when comes to plasma treatments.

Many studies have shown that an interaction coupling between plasma and the target exists in atmospheric pressure plasma systems. Target properties influence the discharge morphology and affect the creation of turbulence in the gas flow [22, 23]. In the presence of the liquid target, plasma heating leads to evaporation and changes in the composition of the surrounding atmosphere, modifying the physico-chemical properties of both the gas and liquid phases [24]. Studies of the influence of treatment conditions on water properties have shown that small changes in the distance and volume of liquid lead to changes in the concentrations of deposited reactive species [25, 26]. Therefore, an additional complexity is introduced in the plasma system with a liquid target. Exposure of aqueous samples to non-thermal plasma leads to changes in water properties, creating an acidic environment under certain treatment conditions, changing conductivity and redox potential, and creating reactive oxygen and nitrogen species (RONS) [27, 28]. Creating plasma-activated liquid with specific, predetermined properties and a certain content of reactive species provides huge potential for application, but at the same time, is an extremely demanding task. Depending on the plasma jet configuration, the type of discharge and the energy released in the plasma, different reactions will occur in the gas phase (plasma) and the liquid [29]. One step towards a better understanding of the entangled relationship between the characteristics of the plasma source, the discharge properties, and treated sample characteristics, is

to carry out detailed diagnostics of the plasma jet. An important part of that includes the investigation of the electrical parameters, such as the efficiency of the input power transfer to the source, power dissipation, and impedance of the whole system.

In this study, we assessed in detail the electrical characteristics of the He plasma jet operating against a water target. The jet was supplied by a continuous sine high-voltage signal at the frequency of 330 kHz. The electrical discharge is generated in the gas phase above the liquid by applying the voltage at the pin electrode of the jet with distilled water serving as grounded electrode. The main idea of the research was to perform a proper determination of the dissipated powers and evaluate the typically used methods in plasma systems applied for water treatments. To estimate the powers dissipated in the system, we employed two standard methods. In the first method, the power is calculated directly from the product of the measured time-varying current and voltage signals ($V-I$). The second method is based on measuring the voltage across a capacitor connected in a series with the grounded sample ($Q-V$).

These two methods, $V-I$ and $Q-V$, as we named them here, are some of the most widely used power determination procedures. They have been extensively implemented in numerous plane geometry DBD configurations and several plasma jet systems, including conventional plasma jet configurations supplied with both AC [30] and DC voltages [9, 31], driven by either a pulsed high-voltage signal [32] or continuous sinusoidal signal [33]. Furthermore, the input power and the discharge power have been estimated for diverse plasma jet arrangements such as hollow electrode APPJ [14, 34], kINPen [33], single-electrode jet [35] and DBD jet [36], etc.

In recent years, a number of researchers have investigated cold plasma sources that interact with the targets of certain characteristics for a particular application. Plasma jet treatments are most commonly performed in secondary electrode configurations with a grounded or floating electrode. The two methods investigated in this work were used for power dissipation measurement in various plasma jet configurations for the treatment of biological materials and the inactivation of bacteria [37, 38], biomedical applications that include *in vitro* and *in vivo* treatments [39] and producing of plasma activated (tap) water [40].

The research to date has tended to compare the differences between several measurement techniques for power determination only in the field of DBD plasma reactors [41–44]. However, as the $Q-V$ method is mostly used with different DBD source configurations, there are few comprehensive analyses and comparisons of the two methods for pin-to-plate plasma jets [33, 35] operating with pulsed excitation. Although previous studies have investigated pin-to-plate configurations of a plasma jet, there has been no study that we are aware of, which has dealt with plasma jets interacting with liquid targets and driven by a continuous sine wave. Therefore, the aim of this work was to assess the power consumption (at the target) of a high-frequency pin type plasma jet by using both $V-I$ and $Q-V$ methods and to compare the obtained results.

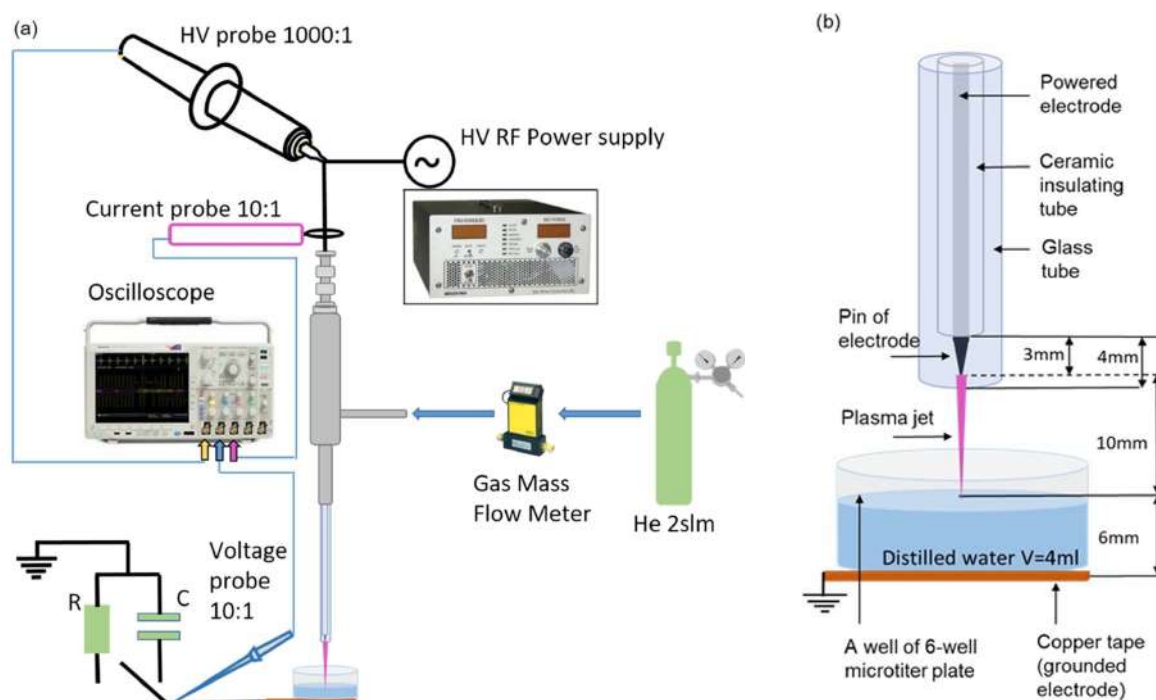


Figure 1. Schematic representation of the experimental setup (a) and enlarged view of the powered electrode and target placed below the tip of the electrode (b).

2. Experimental setup and methods

2.1. Plasma jet system

Figure 1 presents a schematic diagram of an experimental setup. The plasma source used in this experiment was a plasma jet designed to operate as a pin-electrode configuration at atmospheric pressure. It consisted of a cylindrical metal body (diameter 22 mm) and glass tube, whose inner and outer diameters are 4 mm and 6 mm, respectively. A powered electrode was made of stainless steel wire with a diameter of 1 mm. It was covered with a ceramic insulating tube and placed along the glass tube axis. The sharpened tip of the wire protruded 3 mm from the ceramic insulation into the glass tube (see figure 1(b)). The powered electrode was connected to the high-voltage (HV) signal by a BNC connector positioned at the top of the metallic jet body. The second electrode was the target—a distilled water sample placed in a well of 6-well microtiter plate. The initial conductivity of water was $1.5 \mu\text{S cm}^{-1}$ (Hanna Instruments, HI76312 electrode, HI5521 controller). The plate was positioned below the tip of the powered electrode (see figure 1(b)). The volume of water sample in the well was 4 ml. The distance between the liquid surface and the wire tip was 10 mm in all measurements. The copper tape was attached at the outer bottom side of the well and grounded. The experimental setup, together with the positioning of the grounding tape, is a typical system which is used for the plasma treatment of liquid samples. The setup on one side allows for the measurement of electrical signals while, on the other side, treated liquid is in contact only with the clean vessel, so the chemical reactions are governed solely by the plasma-liquid interaction. Treatment of a liquid in

contact with a copper tape showed that it produced additional chemical reactions and specific species in the liquid after the treatment [25]. Comparison between a setup where copper tape is inside the liquid sample or outside has been investigated for a similar system [29] showing rather small differences in the signals recorded in the grounded line. Also, working with the sine wave high voltage signal at high frequencies allows the use of different materials as a vessel for the samples.

The plasma in the experiments was generated and controlled by using a commercial HV high-frequency power supply (T&C Power Conversion AG 0201HV-OS 140 W–500 kHz with continuous sinusoidal signal with a frequency of 330 kHz). The power supply allows control of an output power (not output voltage or current) and measures the power forwarded from the device to the plasma system as well as the reflected power. The reflected power in all experiments was below 2 W. Due to several elements in the electrical circuit, there is a difference between the power measured at the source and the power delivered to the plasma. Since the power delivered to a discharge is a crucial parameter in the electrical diagnostics of a plasma jet, this indicates a need to measure current and voltage waveforms at the plasma jet itself. To monitor the instantaneously applied voltage waveforms at the power electrode we have used an HV probe (P6015A TEKTRONIX). The current waveforms were measured by using a current probe (Agilent N2783B) connected to the main line of the plasma jet (see figure 1(a)). To avoid an offset in the signal, the current probe was regularly demagnetized.

In the part of the electrical circuit containing the water sample we used two different approaches to measure the

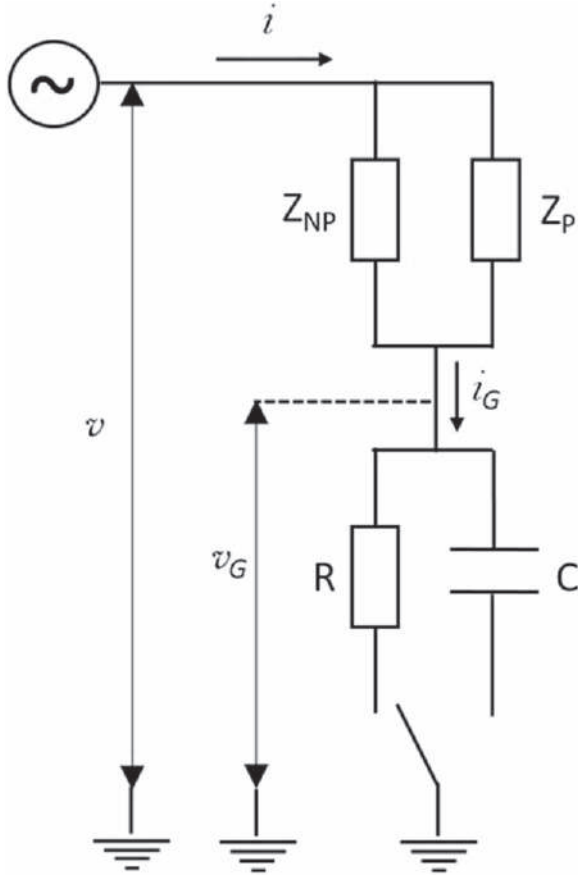


Figure 2. The electrical circuit of the plasma jet system: v —applied alternating voltage at the powered electrode, v_G —voltage in the ground line, i —current in the plasma jet main line, i_G —current through the grounded line, $R = 1 \text{ k}\Omega$, $C = 0.8 \text{ nF}$ and 10.3 nF , Z_{NP} —impedance of the plasma jet system configuration without plasma, Z_P —impedance of plasma.

voltage and current waveforms. When utilizing the V - I method, the resistor of $1 \text{ k}\Omega$ resistance was connected in a series to the ground line of the electrical circuit (see schematic in figure 2). For the Q - V method (Lissajous figures) a capacitor was connected in series to the ground line of the electrical circuit (see schematic in figure 2). For these purposes, the capacitors had capacities of 0.8 nF and 10.3 nF . In both cases, the voltage drop across the elements placed in the ground line (v_G) was measured by using a voltage probe (Agilent N2863B). The resistivity and capacitance of the voltage probe are appropriate so it minimally perturbs the electrical circuit and influences the measurements. We used the Tektronix MDO3024 oscilloscope to record all waveforms.

In all experiments He was used as a working gas with a fixed flow rate of 2 slm adjusted by a mass flow controller (Omega FMA5400/5500). The open-end geometry of the plasma jet enables the mixing of the He with the surrounding air. When the plasma is ignited, the filamentary type discharge operates in the gas mixture around the tip of the powered electrode. With the increase of the input power, the generated streamer propagates through the gap and, for most operating parameters, it was touching the liquid sample.

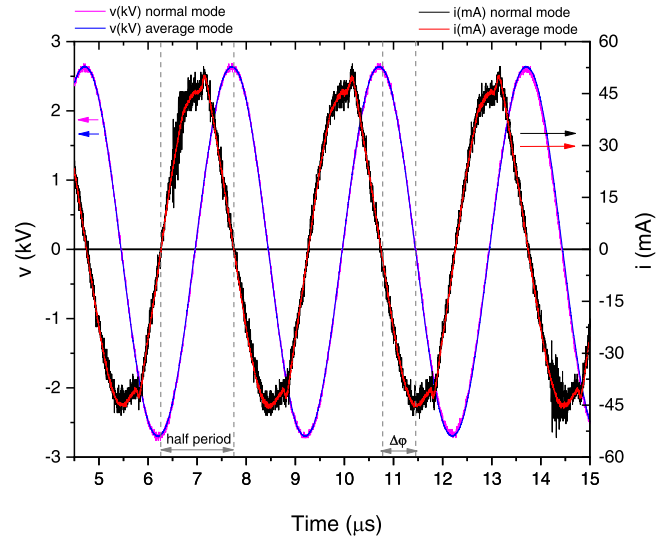


Figure 3. Voltage and current waveforms obtained at the electrode in normal mode and average mode (16 cycles averaged). The waveforms were measured for applied peak-to-peak voltage of 5.5 kV , $f = 330 \text{ kHz}$, and He flow of 2 slm . The electrical circuit configuration was with the resistor $R = 1 \text{ k}\Omega$ in the grounded line. The half period of applied voltage ($T/2 = \pi/f$) and phase difference ($\Delta\varphi$) are noted in the plot.

2.2. The root mean square (RMS) values and V - I method

The electrical circuit (schematic in figure 2) allows the monitoring of voltage and current instantaneous signals through the plasma jet main line and in the ground line. The waveforms of applied voltage v and current i through the plasma jet main line are plotted in figure 3.

These signals were captured by the oscilloscope in normal acquisition mode and also by averaging 16 acquired waveforms. The signals obtained in average mode closely follow the shape of the signals recorded in normal mode but without the noise which is visible mostly at the current waveform.

The acquired time-dependent values of voltage and current through the plasma jet main line, $v(t)$ and $i(t)$, and voltage drop across a resistor in the ground line $v_G(t)$ were used for electrical characterisation and power calculations. The root mean square values of the voltage and current were calculated in the following way:

$$V_{\text{RMS}} = \sqrt{\frac{1}{nT} \int_0^{nT} v^2(t) dt} \quad (1)$$

$$I_{\text{RMS}} = \sqrt{\frac{1}{nT} \int_0^{nT} i^2(t) dt} \quad (2a)$$

$$I_{G\_RMS} = \sqrt{\frac{1}{nT} \int_0^{nT} i_G^2 dt}, \quad (2b)$$

where T is a period of the given signal and n is the number of periods, $i_G(t) = v_G(t)/R$ and R is the electrical resistance of the resistor in the grounded line.

In the V - I method, first the time-dependent powers delivered to the plasma jet and consumed by plasma were calculated as the product of voltage $v(t)$ multiplied by the

appropriate current, $i(t)$ or $i_G(t)$. The average powers are then obtained as:

$$P_{\text{mean}} = \frac{1}{nT} \int_0^{nT} v(t) \cdot i(t) dt \quad (3a)$$

$$P_{\text{mean}_G} = \frac{1}{nT} \int_0^{nT} v(t) \cdot i_G(t) dt. \quad (3b)$$

In all calculations, as input data we used the waveform values obtained in averaging mode. Comparison between two sets of data (obtained in normal and averaging mode) showed differences in calculated values of less than 1%, which is below the experimental error. The phase differences ($\Delta\varphi$) (see figure 3) between high voltage and other obtained signals were calculated in the following way: $\Delta\varphi = (\Delta t/T) \times 360^\circ$, where Δt is the time period between points where both signals had zero values.

2.3. Q-V method

Another method commonly used for the electrical characterization and measurement of power consumed by plasma is based on a charge-voltage plot or Lissajous figure. For these measurements, the capacitor was inserted in series with the plasma jet and grounded line, so that the same electrical charges formed in the discharge flow through the grounded target and the capacitor. By measuring the capacitor voltage, knowing the capacitance and assuming purely capacitive properties, the charge flow through the capacitor can be obtained. In the Lissajous figure, the area enclosed by the curve is equal to the energy dissipated per one period of applied voltage:

$$E = C \oint v(t) dv_G = \oint_T v(t) \frac{dQ}{dt} dt = \frac{P_{\text{mean}_G}}{f}, \quad (4)$$

where C and $v_G(t)$ are capacity and voltage across the additional capacitor (figure 2), $v(t)$ is the applied high-voltage signal, $f = 1/T$ is the corresponding frequency, and P_{mean_G} is the average power consumed by the plasma [43, 45, 46]. In this work, the closed area of a Lissajous figure was obtained by the Polygon area function in the Origin software (Origin Lab Corporation, Northampton, MA, USA). A different approach to calculate the area inside the Lissajous figure can be found in [47].

To ensure that the influence of the capacitor on the whole system is minimal, the capacitance was chosen to be large compared to the equivalent capacitance of the plasma jet, gas gap, and target. This difference is at least one order of magnitude. The second important property is the type of capacitor selected. It has been suggested that ceramic capacitors are preferred, owing to their relatively low parasitic losses and accuracy in measurement of charge [48]. An additional advantage is that they are not polarized and may be safely connected to an AC source.

3. Results and discussion

3.1. Instantaneous voltage and current

Before the onset of the plasma jet electrical measurements, the power supply has been set to operate at the frequency of

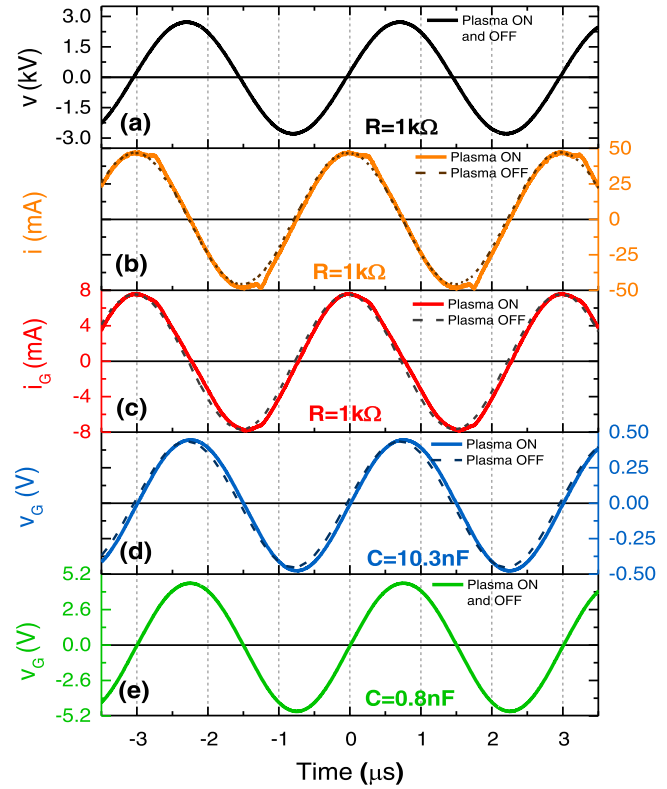


Figure 4. Voltage and current waveforms were measured for an applied peak-to-peak voltage of 5.5 kV and frequency of 330 kHz. The dashed lines represent cases when the plasma was OFF, while full lines represent the Plasma ON regime. In plots (a) and (e) Plasma ON and Plasma OFF signals overlap. (a) v —applied voltage at the electrode. (b) and (c) electrical circuit configuration with the resistor $R = 1 \text{ k}\Omega$, i —current in the plasma jet main line, i_G —current through the grounded line. (d) and (e) electrical circuit configurations with capacitors: v_G —voltage across the 10.3 nF (blue line) and 0.8 nF (green line) capacitor.

330 kHz with the lowest reflected power. The voltage and current signals were monitored for three different configurations of the ground line. In figure 4 we show the waveforms for particular applied voltages. The measurement was performed without feed gas (Plasma OFF) and with the He flow of 2 slm (Plasma ON).

The waveforms when the plasma was on are plotted by a full line, while the dashed line shows the case without plasma (achieved without He flow). All presented signals were recorded with the applied peak-to-peak voltage of 5.5 kV. The waveforms in figures 4(a)–(c) are acquired in electrical circuit configuration with the resistor and represent the averaged value of 16 acquired waveforms. The signals in figures 4(d) and (e) were obtained in electrical circuit configurations with capacitors by averaging 512 waveforms. Identical waveforms were obtained for the applied voltages $v(t)$ (figure 4(a)) and currents $i(t)$ (figure 4(b)) in configurations when two capacitors were inserted in the grounded branch of the circuit. Therefore, for simplicity, only one set of $v(t)$ and $i(t)$ signals are presented.

The high voltage signal applied to the plasma jet in the configuration with resistance R in the grounded branch of the circuit is shown in figure 4(a). It has a regular sinusoidal

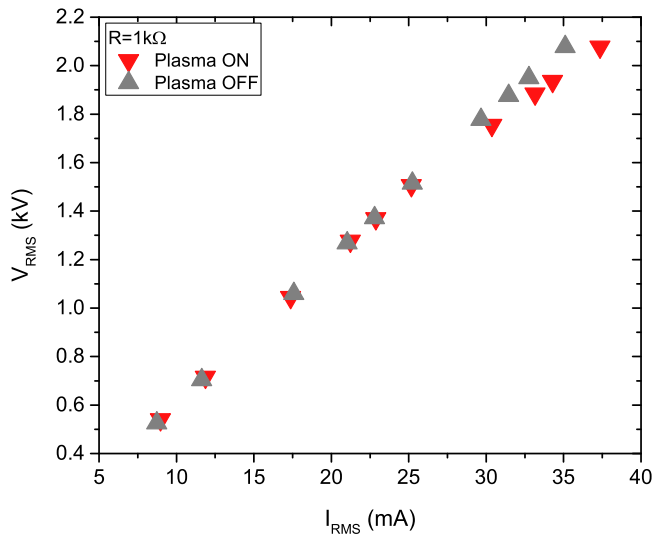


Figure 5. V-A characteristics obtained at the plasma jet in the presence (Plasma ON) and absence (Plasma OFF) of plasma for the configuration with $R = 1 \text{ k}\Omega$ in the grounded line. The V-A characteristics obtained when $C = 0.8 \text{ nF}$ and $C = 10.3 \text{ nF}$ were used in the electrical circuit are the same and not presented here to retain the clarity of the figure.

shape, regardless of whether the plasma is ignited or not. Figure 4(b) shows the current $i(t)$ obtained by the current probe and flowing through the main line to the plasma jet. When there is no helium flow, the obtained current signal is sinusoidal, since the applied voltage is lower than the breakdown voltage in air. When the plasma is ignited, the current waveform is deformed from the sine wave and it has two peaks, one per each positive and negative half period of the imposed voltage. The appearance of the peak in the current waveform is connected to the streamer type of the discharge. The observed deformation in the current waveform when discharge occurs has been found previously in other atmospheric plasma jet systems [15, 49–51]. It was the result of discrete propagation of the discharge outside the gas tube in a form of PAPS. In general, changes in the shape of the current signal occur because the current in the plasma jet main line is superimposed on the displacement current. The displacement (capacitive) current can be measured directly when the tube is not filled with feed gas and there is no plasma.

The waveform of the current through the ground line $i_G(t)$ is presented in figure 4(c). This is the discharge current that passes through the liquid target and a resistor connected in series to the ground. The difference between the waveform shape in the absence and presence of the plasma exists, but it is less noticeable than that in the case of current $i(t)$. Related to the phase difference, the plasma off current waveforms $i(t)$ and $i_G(t)$ in figures 4(b) and (c) lead to the high voltage waveform by 85° and 88° , respectively. This points to a system that is mainly capacitive (for pure capacitance the phase difference is 90°) with small deviation from 90° due to the inherent resistivity of the components of the electrical circuit and the plasma source. Deflection from a 90° phase difference means that we observe dissipation of the power even when the plasma is not ignited. After the ignition of the

plasma these phase differences remain almost the same, indicating that deposited power in plasma is small, in the order of a few watts. Voltages across two different capacitors are presented in figure 4(d) for $C = 10.3 \text{ nF}$ and in figure 4(e) for $C = 0.8 \text{ nF}$. The voltages across the 0.8 nF capacitor are in phase with the applied voltage independently of the plasma being ignited or not, while in the case of 10.3 nF capacitor a small difference in voltage signals is visible.

3.2. Volt-ampere characteristics of the plasma jet

The volt-ampere V-A characteristics of the plasma jet with distilled water as the liquid target are presented in figures 5 and 6. By using the recorded waveforms for each selected operation point, the V-A characteristics were plotted as a function of the RMS current through the plasma jet main line (figure 5) and the RMS current through the target and ground line (figure 6).

Both characteristics were recorded for the applied peak-to-peak voltages from 1.5 to 6 kV. The points were obtained by increasing the output power of the power supply. All measurements were performed in triplicate and observed differences in the obtained values were not more than 7%. For all measurements as a target we used distilled water of the same initial conductivity, because it was noticed that the conductivity of the liquid sample affects the power consumption [52, 53].

The V-A characteristic obtained at the plasma jet (figure 5) was recorded when the resistor was inserted in ground line of the electrical circuit. The characteristics that were the same within the error margins were obtained in the other two configurations of the electrical circuit (with capacitors $C = 0.8 \text{ nF}$ and $C = 10.3 \text{ nF}$) and are not shown to keep the graph in figure 5 clear.

Increasing the power delivered to the plasma jet without plasma (triangles) results in a linear increase of both voltage RMS (V_{RMS}) and current RMS (I_{RMS}). With the gas flow, the discharge becomes visible at a V_{RMS} of 0.5 kV and it first appears as a small bright point at the tip of the powered electrode. At a V_{RMS} of 1.3 kV, a plasma filament forms and extends 2 mm from the electrode tip. The filament elongates with an increase of the input power. The discharge crosses the entire gap between the powered electrode and target, and streamer touches the water surface at a V_{RMS} of 1.75 kV. As can be seen in figure 5, for currents above 30 mA and voltage above 1.75 kV the V-A characteristic measured with plasma on (inverted triangles) changes slope and it differs from the V-A characteristics obtained without plasma (triangles). A further increase in the output power of the HV source (applied voltage $V_{RMS} > 1.75 \text{ kV}$) leads to a more intense light emission from the plasma and to a change of colour. The streamer also begins to move on the liquid surface. For the highest applied voltages, it is observable that several short and thin filaments are adjoined in a streamer.

The V-A characteristics of the discharge plotted against the current in the grounded line I_{G-RMS} , shown in figure 6, were measured in the configurations with the resistor $R = 1 \text{ k}\Omega$ (figure 6(a)) and capacitor $C = 10.3 \text{ nF}$ (figure 6(b)). The

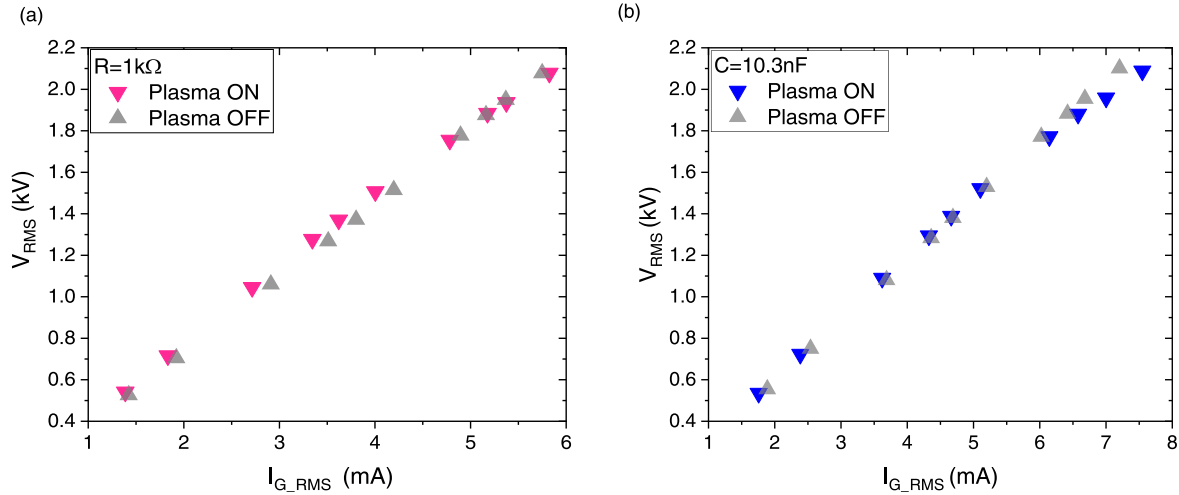


Figure 6. V-A characteristics obtained for the current through the ground line I_{G_RMS} in the presence (Plasma ON) and absence (Plasma OFF) of plasma for the configuration with (a) $R = 1 \text{ k}\Omega$ and (b) $C = 10.3 \text{ nF}$ in the grounded line. The V-A characteristic obtained when $C = 0.8 \text{ nF}$ was used in the electrical circuit is identical to the characteristic in (a). For better clarity in the figure it is not presented here.

pink and blue triangles show the results for measurement when the plasma is ignited, while the grey inverted triangles show results without plasma (without working gas flow).

The V-A characteristics presented in figure 6(a) shows linear dependence and there is no significant difference between the characteristics when plasma is ignited or not. When the capacitor $C = 0.8 \text{ nF}$ was inserted into the electrical circuit, the obtained V-A characteristic is similar to the characteristics presented in figure 6(a). However, the V-A characteristic obtained with capacitor $C = 10.3 \text{ nF}$ (figure 6(b)) has a lower slope and departs from linear dependence at higher currents. The impedances of the capacitors used can explain the observed difference. It can be assumed that the capacitor impedance is equal to the capacitive reactance $X_c = \frac{1}{2\pi fC}$, i.e. that resistance losses are negligible. Thus, the values of $597 \text{ }\Omega$ and $46 \text{ }\Omega$ are obtained for $C = 0.8 \text{ nF}$ and $C = 10.3 \text{ nF}$, respectively. Comparing these results with the resistance of $1 \text{ k}\Omega$, it is clear that the impedance of the 0.8 nF capacitor is closer to $1 \text{ k}\Omega$ while the impedance of the 10.3 nF capacitor differs by one and a half orders of magnitude from that value.

3.3. Impedance of the plasma jet

In order to determine the impedance of the system, we have used a linear fitting of the plotted V-A characteristics obtained at the plasma jet. The total impedance of the plasma jet system $\hat{Z}$ was obtained as the line slope of the calculated linear fit. It was calculated for both regimes, in the absence (Plasma OFF) and presence (Plasma ON) of plasma, and for three different components in the ground line of the electrical circuit. The equivalent circuit presented in figure 2 is a very simple one and serves only for the estimation of the impedance of the used geometry (Plasma OFF) and of the discharge. These rough estimations of the jet system and plasma impedances are important when developing and/or choosing the appropriate power supply system. They present a load that will influence the work of the power supply, its output and

optimal operating frequency. The plasma, which is a highly non-linear system, will in most cases reduce the optimal operation frequency ('frequency pulling') of the power supply [54]. This means that the output voltage can be reduced due to the exiting resonant state with the power supply transformer.

During the Plasma OFF regime, the complex impedance $\hat{Z}$ can be considered as a series connection of the impedance $\hat{Z}_{NP}$ and the impedance of the measuring element in the grounded line of the circuit, the resistor or capacitor (see figure 2):

$$\hat{Z} = \hat{Z}_{NP} + \hat{Z}_{R,C}, \quad (5)$$

where all impedances are in the complex form, $|\hat{Z}_R| = R$, and $|\hat{Z}_C| = X_c = \frac{1}{2\pi fC}$, assuming that the contribution of the equivalent series resistance of the capacitor can be neglected. $\hat{Z}_{NP}$ represents the equivalent impedance of the plasma jet system configuration without plasma, including the capacitance of the plasma jet configuration, target impedance, stray capacitance and parasitic resistance of the cables and connections.

When the plasma is ignited (Plasma ON regime), the plasma impedance is represented by $\hat{Z}_p$ in parallel with the impedance $\hat{Z}_{NP}$ (figure 2). Plasma impedance is a complex system and it is beyond the scope of this work to estimate each of the components. After the plasma inception, the impedance $\hat{Z}$ is given by:

$$\hat{Z} = \frac{1}{\hat{Z}_{NP}} + \frac{1}{\hat{Z}_p} + \hat{Z}_{R,C}. \quad (6)$$

Table 1 shows the total impedances of the plasma jet system $|\hat{Z}|$ obtained in the presence and absence of plasma. The impedances $|\hat{Z}|$ obtained with three different configurations are in agreement with each other, with the difference smaller than 3%. This is expected since the differences in the configurations were in the grounded line part. After the plasma is ignited, the total impedance is reduced by 10%.

Table 1. The total impedance of the plasma jet $|\hat{Z}|$ in Plasma ON and Plasma OFF states for three different measuring elements in the ground line.

Element in the ground line	$ \hat{Z} $ (k Ω)	
	Plasma ON	Plasma OFF
$R = 1.0$ k Ω	54.2	59.0
$C = 0.8$ nF	54.3	59.8
$C = 10.3$ nF	55.6	58.4

3.4. Mean powers dissipated into the system

In this part, we present and discuss two measured powers: the mean power delivered to the plasma jet - P_{mean} and the mean power consumed by the plasma $P_{\text{mean,G}}$, i.e., the power measured in the grounded line originating from the plasma and through the target. Additionally, a comparison of the results obtained using two standard methods, by integrating the current and voltage product (V - I) and by the Lissajous figure method (Q - V) is presented.

The mean power delivered to the plasma jet P_{mean} was determined in all experiments, regardless of which measurement element was inserted in the grounded line. The powers P_{mean} consumed with ignited plasma (red squares in plots) and without plasma i.e. feed gas (empty squares) were calculated by using equation (3) and is presented in figures 7 and 8.

Determination of the power P_{mean} without plasma has significance for determining the mean power delivered only to the plasma, since parasitic resistance in the system is included in the power dissipation measured when the plasma is ignited. Therefore, to obtain the values delivered to the plasma itself ($P_{\text{mean(NET)}}$), the input power measured without He flow (Plasma OFF) was subtracted from the input power when the plasma was on (Plasma ON) for the same applied voltage value:

$$P_{\text{mean(NET)}} = P_{\text{mean}}(\text{Plasma ON}) - P_{\text{mean}}(\text{Plasma OFF}). \quad (7)$$

The minimal uncertainty of both methods in our experiments was estimated to be 0.2 W for voltages below 1.5 kV given by power supply. In this range $P_{\text{mean(NET)}}$ is within this margin. It is found that the power $P_{\text{mean}}(\text{Plasma OFF})$ increases nearly linearly with the applied voltage. Fitting a linear function to the obtained data points permits the determination of power losses for any applied voltage or current in the measured range. Comparing the dependences of P_{mean} on applied voltage V_{RMS} and on the current through the plasma jet I_{RMS} (see figure 8), it is clear that using either voltage or current as an independent parameter leads to the same power dependence.

The obtained $P_{\text{mean(NET)}}$ as function of V_{RMS} is presented in the plot in figures 7 and 8 (star symbol). Figure 7 shows the power P_{mean} obtained in the configuration with capacitor $C = 0.8$ nF, while figure 8 shows results measured in the arrangement with a resistor R . This power was found to be up to 6 W for the V_{RMS} range between 0.5 and 2 kV. In both figures 7 and 8, there is a jump in values $P_{\text{mean(NET)}}$ with the applied voltage above 1.5 kV (or I_{RMS} above 25 mA). This is the situation when the streamer reached and

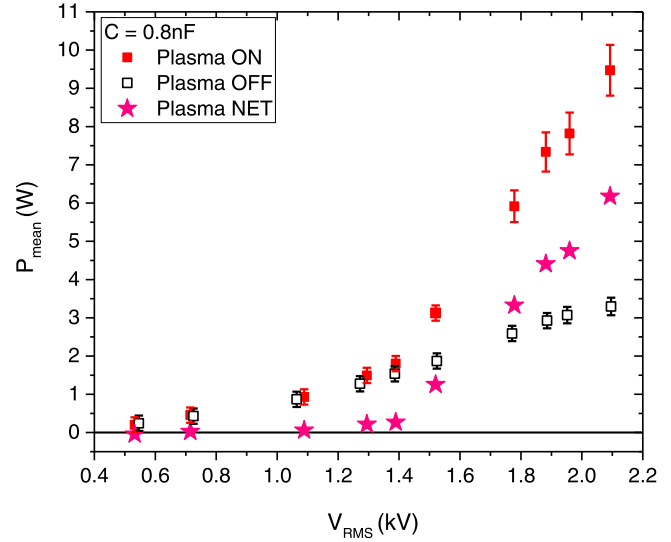


Figure 7. Mean power delivered to plasma jet P_{mean} in the presence (ON) and absence (OFF) of plasma as a function of applied voltage V_{RMS} for a configuration with $C = 0.8$ nF in the grounded line. The power delivered only to plasma ($P_{\text{mean(NET)}}$ points) is acquired by subtracting the values.

connected to the liquid target surface. By switching to that regime, the power P_{mean} jumps to higher values and continues to increase with increasing applied voltage.

In the case of the measurement of power consumption by plasma, $P_{\text{mean,G}}$, we used two different methods (V - I and Q - V). With the serial resistor in the ground line (V - I method), the power $P_{\text{mean,G}}$ was calculated by using equations (3) and (7). When a capacitor was connected in series with the grounded target, the Q - V method was employed. Figure 9 shows the dependence of the charge on the measurement capacitor on the applied peak-to-peak voltage for capacitor $C = 0.8$ nF. Lissajous curves and the calculated deposited powers for three different input voltages of 5 kV, 5.5 kV, and 5.9 kV are presented.

The powers were calculated by using equation (4). Since both the applied voltage and the charge oscillate with the same frequency, a Q - V plot will form an ellipsoid Lissajous figure. The elliptical shape of the Q - V diagrams is obtained when a continuous plasma current flows during a full period of the high voltage signal [21, 48].

From the data in figure 9, it was apparent that the Lissajous loop retained the same shape and changed the slope (the line connecting points of minimum and maximum voltage) as the voltage changed. The centre of the ellipse coincided with the coordinate system point of origin. Comparing the results for two different capacitors, it was found that the power measured using the 10 nF capacitor was slightly higher than the power estimated with 0.8 nF capacitor for the same applied voltage. The difference was around 23%, and it can be due to capacitor properties or plasma adjustment to different circuit elements [33].

Charge-voltage plots for states with and without plasma at the same applied voltage for 0.8 nF and 10.3 nF monitor capacitors are presented in figures 10(a) and (b), respectively. Our measurements show that the obtained Lissajous figure in

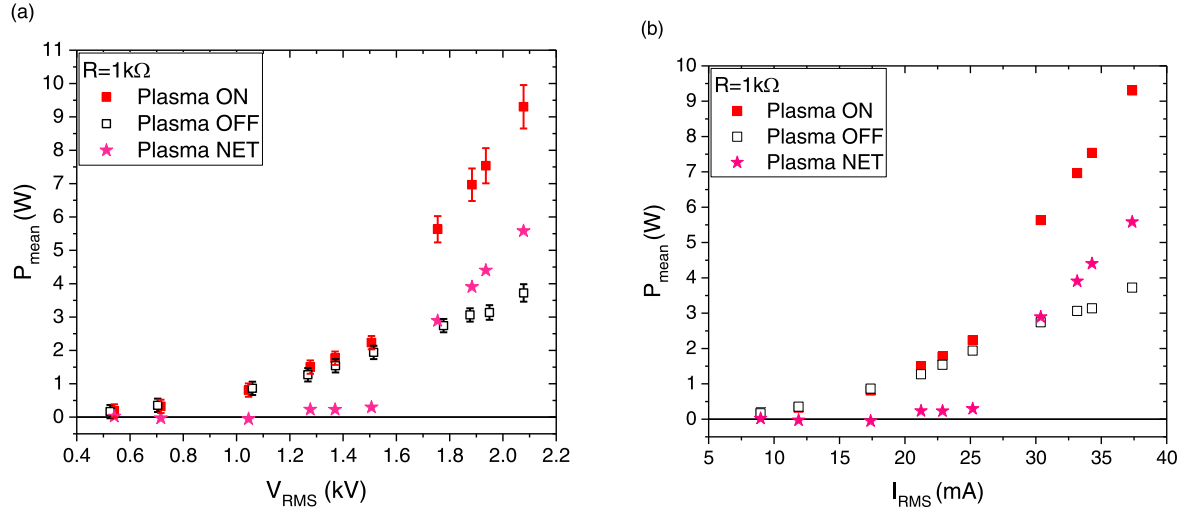


Figure 8. Mean power delivered to plasma jet P_{mean} in the presence (ON) and absence (OFF) of plasma for a configuration with $R = 1 \text{ k}\Omega$ in the grounded line. $P_{\text{mean}}(\text{NET})$ points are acquired by subtracting the values. (a) P_{mean} dependence on the applied voltage V_{RMS} , (b) P_{mean} dependence on the current through plasma jet main line I_{RMS} .

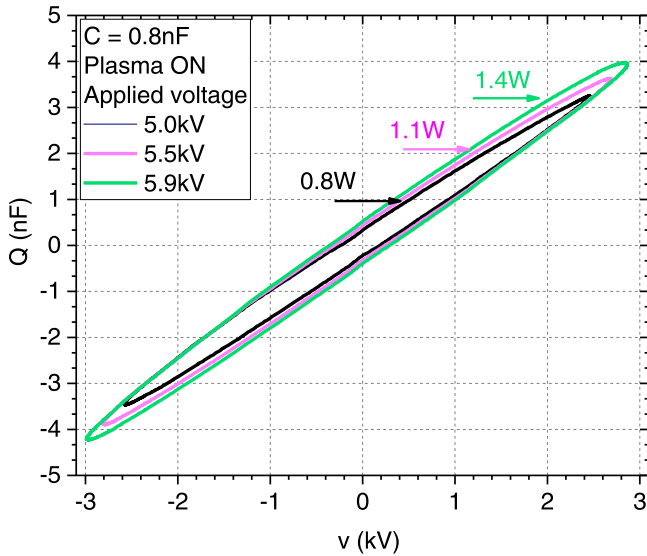


Figure 9. Charge-voltage plot (Lissajous figure) and consumed power P_{mean_G} in the presence of plasma for three different applied peak-to-peak voltages (5 kV, 5.5 kV, 5.9 kV) and a signal frequency of 330 kHz. A capacitor of 0.8 nF was used for measurement.

the absence of plasma had a nonzero area, i.e. it was a very elongated ellipse. Usually, the curves obtained without plasma ignition in the Q - V diagram are considered to be a straight line, as it was assumed in the other studies with different plasma sources employed [42, 44]. The existence of the elongated ellipse when plasma is not ignited may be the result of technical issues such as defective wire connections or uncompensated probes [48]. In our case, these contingencies were checked and excluded. So, this power dissipation in the Plasma OFF regime manifested the resistive losses present in the system, i.e. that a certain power was consumed in this part of the circuit even without plasma. This was observed also with Plasma OFF power values measured

at the plasma jet (figures 7 and 8). Comparing the plots in figure 10, it can be seen that the power losses measured with 10.3 nF capacitor are one order of magnitude less than those in the case of 0.8 nF capacitor. In the case of high excitation signal frequency, the parasitic properties of the capacitor can have a significant value, thus influencing the total impedance of the capacitor in the circuit [33, 41, 55]. This, in turn, influences the determination of power, particularly in the Plasma OFF case.

The Q - V plot in the absence of plasma has a shape of an elongated ellipse, with the sharp turning point at maximum input voltage, meaning that the charge and supplied voltage are in phase reaching the peak value at the same time. When the plasma is turned on, a Lissajous curve gains rounded endpoints indicating additional resistive losses in the system. However, the waveforms of voltage across the capacitor $v_G(t)$ and high voltage $v(t)$ remain in the phase for Plasma ON. Therefore, it can be assumed that the ignition of plasma does not lead to an increase in the impedance of an element of the plasma jet system, which can change the phase between voltage signals. This analysis suggests that the power obtained with Plasma ON contains the total power consumed in the system. Thus, the determination of the mean power consumed only by plasma, $P_{\text{mean}_G}(\text{NET})$, has to follow a similar procedure used for the V - I method in equation (7): the power calculated from the Plasma OFF Lissajous figure should be subtracted from the power determined from the Plasma ON Lissajous figure for the same applied peak-to-peak voltage.

In order to evaluate the two methods for the determination of the dissipated power, the dependence of the mean power consumed by plasma $P_{\text{mean}_G}(\text{NET})$ on V_{RMS} was plotted in figure 11 with values obtained with three different electrical elements in the grounded line.

The power $P_{\text{mean}_G}(\text{NET})$ ranges from 0 to 1.7 W for V_{RMS} between 0.5 and 2 kV. Compared to the results calculated for the power P_{mean} , we observed that less than 30% of P_{mean} is consumed by the plasma and forwarded through the

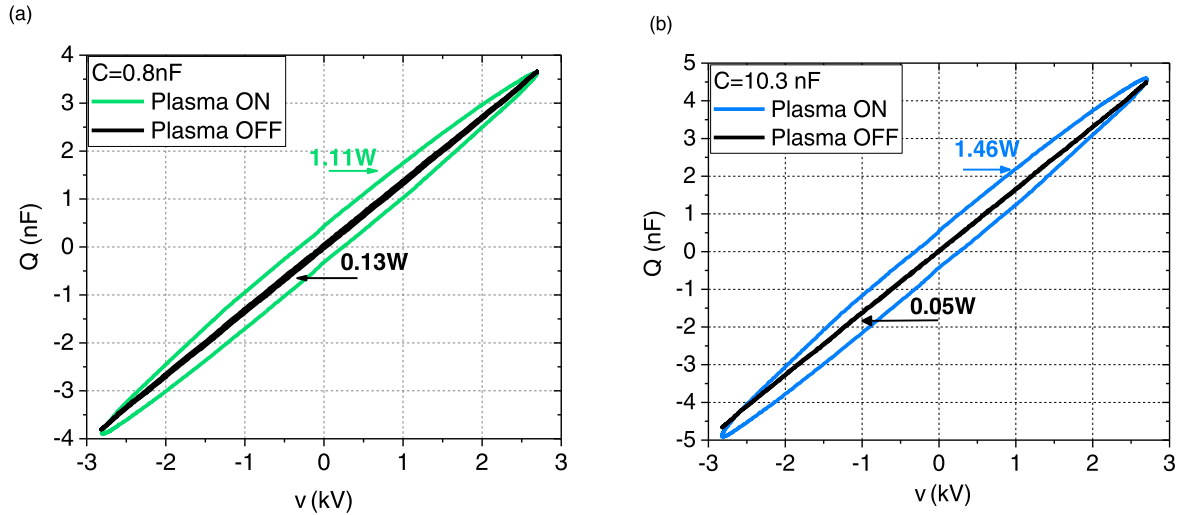


Figure 10. Charge-voltage plot (Lissajous figure) and consumed power P_{mean_G} for the applied peak-to-peak voltage of 5.5 kV and a signal frequency of 330 kHz. Data in (a) green and (b) blue represent state with plasma and data in black (a) and (b) represent state without plasma (without He flow). Capacitors of (a) 0.8 nF and (b) 10.3 nF were used for measurements.

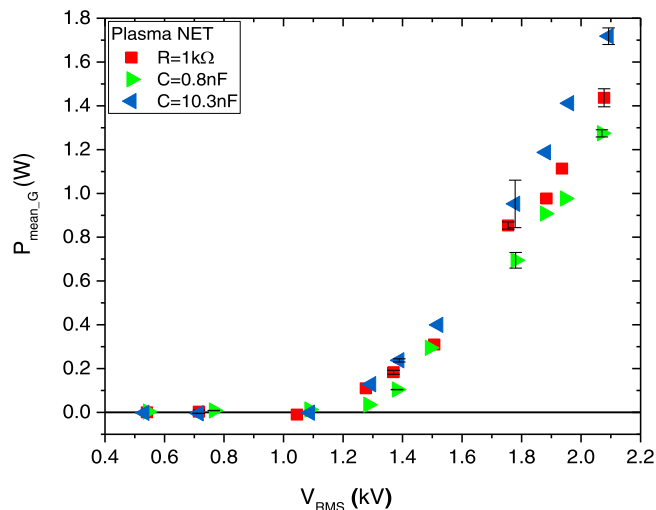


Figure 11. Mean power consumed by plasma P_{mean_G} (NET) for three different measuring elements in the grounded line.

grounded line. It can be seen in figure 11 that the power dependence on V_{RMS} calculated by the Lissajous figure (triangles) is in excellent agreement with those values obtained by the V - I method (squares). The error bars were estimated as standard errors from three different measurements. In the V_{RMS} range from 0.5 to 1.1 kV, the calculated difference between powers measured in the presence and absence of plasma is close to zero. However, the plasma could be observed with the naked eye as a small bright region at the tip of the electrode, even in this range of applied voltages. Therefore, these power values were lower than the sensitivity of the methods.

3.5. Difficulties in measurements of power

The most common problems in the power measurement are unwanted losses in the circuit that affect repeatability of the measurements. The losses in the plasma jet system occur due

to the connections of the circuit elements, their quality i.e. characteristics, cables and also measuring equipment (especially when approaching the high frequency range). In the ground line of the electrical circuit, we noticed that any change in connections, such as the change of cables, connectors or a grounding technique, will affect the measured values. To minimize the power losses and ensure reproducible results, the cables of appropriate length, connection and quality should be selected before measurement.

Several cautions need to be noted regarding the use of the Q - V method. The most common difficulties to pay attention to are: the selection of the appropriate type of capacitor, the use of proper cables for grounding the capacitor and the appearance of nodes or discontinuities in the curve.

First, the selection of an appropriate capacitor is considered as the most important part of the successful application of this method. It was found that different types of capacitors with the same capacitance yield different results. The use of film capacitors has been found to lead to non-physical results. When using a 10 nF metalized polyester (PET) capacitor, we measured more dissipated power when the plasma was OFF than when the discharge was ignited. In some cases when a PET capacitor was used, we observed a self-intersecting loop in the part of the figure where the charge and voltage change sign. The appearance of node points in the curve indicates different oscillation frequencies of the two signals—the applied voltage and capacitor voltage. These findings suggest that PET capacitors should not be used in the Q - V technique for power determination, at least not with high-frequency signals.

The capacitor size should be chosen with respect to the plasma jet system capacitance, i.e. a capacitor should be selected that can store all of the charge that has passed through the discharge during one half-cycle of the applied voltage. This can be fulfilled if the capacitor size is at least one order of magnitude higher than the plasma source capacitance. In this work, the capacitance size was selected to be

three orders of magnitude greater than the plasma jet system capacitance.

As already mentioned, the connections in the grounded branch of the circuit greatly influence the repeatability of measurements. Small changes in the position of the cable between the grounded electrode and the monitor capacitor could cause changes in the inclination and width of the Q - V elliptic loop. A suitable cable which does not influence the characteristics of the Lissajous curve should be selected for the measurement.

Finally, despite its type and capacitance, every capacitor gives open loops. The Q - V plot is discontinuous because the curve after one period of applied voltage does not return to the starting point. That is, the charge and voltage values are different in points $t = 0$ and $t = T$. This feature of the Lissajous figure has already been observed for DBD plasma reactors [41, 56]. Despite this, the integration of the figure area is still possible, although there is a gap in the curve. This issue can be diminished when using a high-resolution oscilloscope that provides waveforms with a large number of points.

The main challenge with the use of the V - I method is resistor selection. It should be large enough to limit the discharge current. This is especially important in the case of low resistivity targets (conductive targets). An uncontrolled increase in discharge current can lead to a change in the type of discharge and then to heating and evaporation of the liquid target. However, the measurement resistor must be small enough to measure the voltage across the resistor with a voltage probe. Moreover, there are certain problems with using the V - I method when a pulsed power source is used in the experiment. In that case, when calculating the power, it is necessary to perform integration over the entire pulse duration period. Integration over one part of the period of the input signal which is shorter than the pulse duration, gives an inaccurate result for the power deposition, as it could include only the positive or negative part of the waveform oscillation. This would lead to an underestimation or overestimation of the measured power.

4. Conclusion

In this paper, we presented the results of electrical diagnostics of the He plasma jet powered by continuous sine HV signal and operating in contact with a liquid sample. The main goals were to use data from systematically performed measurements of the electrical parameters of such a system for the determination and comparison of the mean power consumed by plasma P_{mean_G} obtained by using two different methods - V - I and Q - V . It must be emphasized that the displacement current can be significant, especially in the case of atmospheric pressure plasma sources, and it needs to be taken into account. Since it is mainly a result of the capacitive characteristics of the plasma systems, it does not influence the mean power deposited to (consumed by) the plasma. This is evoked by the fact that the plasma jets in the configuration with the pin electrode are employed in different applications in contact with the liquid target, but without thorough

investigation of the power measurement techniques, especially for the case of a continuous wave power supply.

For that purpose, voltage and current waveforms were monitored and V - A characteristics of the plasma jet and the discharge were established. The phase difference between the current and voltage signal showed mainly the capacitive nature of the system, regardless of the plasma presence in the system. As the obtained V - A characteristics were linear, the data were used to determine the impedance of the whole system from the linear fitting of the plot. The impedance was almost the same regardless of the ground circuit configurations.

The mean power delivered to the plasma jet was determined by using the V - I method and found to be up to 6 W for a V_{RMS} range between 0.5 kV and 2 kV. It was shown that plotting the power values using either voltage or current as an independent parameter results in the same power dependence.

The mean power consumed by the plasma was determined by using two different methods. In both case when the capacitor was inserted in the ground line of the electrical circuit, an elliptical shape of the Lissajous figure was obtained. However, the measured power observed with two different capacitors differs by 23%, suggesting a slightly different adjustment of the plasma operating point due to the system conditions. Also, it shows that the properties of the capacitor should not be neglected when calculating the power delivered to the plasma, as the capacitor should not be considered as an ideal element of the circuit. Nevertheless, this study has found that the discharge power dependence on V_{RMS} calculated by the V - I method is consistent with those observed by the Lissajous figure (Q - V method).

Nevertheless, proper utilization of the power measurement method is necessary if one performs an optimization of the energy efficiency of the plasma system. The mean power delivered to plasma calculated by both methods ranges from 0 to 1.7 W for a V_{RMS} range between 0.5 kV and 2 kV. Therefore, this means that less than 30% of the power delivered to the plasma jet is consumed by the plasma.

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Article

Plasma-Activated Water Against Carbapenem-Resistant *Klebsiella pneumoniae* and Vancomycin-Resistant *Enterococcus faecalis*

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Article

Plasma-Activated Water Against Carbapenem-Resistant *Klebsiella pneumoniae* and Vancomycin-Resistant *Enterococcus faecalis*

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Abstract: The scope of the antibacterial effects of plasma-activated water (PAW) is not yet fully comprehended. We investigated the activity of PAW produced by the in-house 3-pin atmospheric pressure plasma jet against carbapenem-resistant *Klebsiella pneumoniae* and vancomycin-resistant *Enterococcus faecalis*, with a focus on PAW's potential to promote susceptibility to conventional antibiotics in these bacteria. Bacterial inactivation was determined by the colony count after 15 and 60 min PAW treatments. Minimum inhibitory concentrations (MICs) measured following repeated exposures to PAW across multiple generations of bacteria enabled the assessment of changes in susceptibility to antibiotics. The PAW's efficacy was also analyzed through the detection of intracellular reactive oxygen and nitrogen species in treated bacteria. Time-dependent significant inactivation efficiency against *K. pneumoniae* was observed (log reduction 6.92 ± 0.24 after 60 min exposure), while effects on *E. faecalis* were limited. PAW demonstrated potential to decrease the MICs of crucial antibiotics. Namely, a 50 to 62.5% decrease in the MICs of colistin against *K. pneumoniae* and a 25% reduction in the MICs of vancomycin against enterococci were recorded. We found a significant increase in the superoxide anion concentration in *K. pneumoniae* and *E. faecalis* cells after PAW treatments. This study indicates that PAW's inactivating efficacy coupled with the capacity for the potentiation of antibiotic effects is a promising combination against multidrug-resistant bacteria.

Keywords: plasma-activated water; antibacterial efficacy; multidrug resistance; *Klebsiella pneumoniae*; *Enterococcus faecalis*



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1. Introduction

Antimicrobial resistance (AMR) is one of the most pressing global health threats we face today [1,2]. The resistance of bacteria to antibiotics is of particular importance, as multiple recent surveys and surveillance studies show alarmingly high and still increasing levels of bacterial AMR with devastating consequences [1–5]. It has been shown that bacterial AMR, associated with 4.95 million deaths, was a leading cause of death globally in 2019 [4]. According to the most recent forecasts, further increases in the counts are

expected [5]. The estimated numbers of annual deaths attributable to and associated with bacterial AMR in 2050 will be 1.91 million and 8.22 million, respectively.

It is therefore clear that responding to bacterial AMR with novel therapeutics is of paramount importance [2,5,6]. The World Health Organization (WHO) addressed the challenge by classifying the antibiotic-resistant bacterial pathogens into the critical, high, and medium categories of priority [7]. The major purpose of this list of priority pathogens, which was first published in 2017 and updated in 2024, is to guide research and the development of new antibacterial agents for control, prevention, and treatment. While the latest review of antibiotics in clinical and preclinical development reveals some promising candidates, the overall finding is a lack of new antibiotics to effectively combat drug-resistant bacteria recognized as priority pathogens [6,8].

Novel non-traditional agents capable of overcoming multiple drug resistance mechanisms in bacteria offer the potential to tackle bacterial AMR. Cold atmospheric plasma (CAP), a non-thermal ionized gas generated at ambient temperature and pressure, is an innovative technology that has emerged as a highly promising option targeting antibiotic-resistant bacteria. The antibacterial activity of CAP has been shown in multiple studies and is attributed primarily to the mixture of reactive oxygen and nitrogen species (RONS) that are created in the gas phase of the discharge [9–17]. During CAP treatments, bacteria can be in direct contact with the plasma gas phase and its rich chemistry containing short- and long-lived RONS, ions, electrons, UV radiation, and metastable and excited species or indirect contact, when CAP is used to treat suspensions containing bacteria or liquid to produce plasma-activated liquid (PAL), including plasma-activated water (PAW) [18–26].

CAP is used for treatments of deionized, distilled or tap water, then stored for some period or applied to microbial samples immediately after the CAP treatments. The characteristics of PAW vary depending on the type of CAP source that is used, its electrode geometry, working gas, frequency, the type of the applied voltage (sine, pulsed), and if the discharge is created above the water or in bubbles in the water [27–33]. The chemistry responsible for bacterial inactivation primarily includes short-lived RONS (like hydroxyl radicals ($\text{OH}^\bullet$), ozone (O_3), superoxide (O_2^-), peroxyxynitrite (ONOOH), etc.) and long-lived RONS (nitrate (NO_3^-), nitrite (NO_2^-), hydrogen peroxide (H_2O_2), etc.) [18,34–37]. In the literature, characteristics of PAW are usually reported by the measured pH, oxidation reduction potential (ORP), conductivity, and, more importantly, by measured concentrations of H_2O_2 , NO_3^- , and NO_2^- , which are the main reasons for the bactericidal properties of PAW. The complete understanding of mechanisms leading to bacterial inactivation is still lacking, but it has been shown that the cell membrane and cell wall, intracellular proteins, and DNA are key bacterial structures and components affected by RONS contained in PAW [19–21,25].

Many studies have reported PAW's potential to inactivate bacteria [38–43], but also showed high variability in the results obtained due to processing parameters for PAW generation and application as well as bacterial characteristics. We, therefore, aimed to assess the inactivation efficiency of PAW produced by the in-house 3-pin atmospheric pressure plasma jet (3-pin APPJ) against selected model multidrug-resistant (MDR) bacteria, carbapenem-resistant *Klebsiella pneumoniae* and vancomycin-resistant *Enterococcus faecalis*. Both bacteria are among the leading causes of healthcare-associated infections (HAIs) [44,45], and their resistance patterns have been recognized as particularly challenging targets for conventional antimicrobials [1–3,7]. Nevertheless, they have not been extensively investigated as specific targets for the antimicrobial activity of PAW, which particularly applies to *K. pneumoniae*. Several previous studies showed CAP and plasma-activated saline treatments to result in enhanced antibiotic activity [46–48], but these data for the effects of PAW treatments are lacking. Hence, the second goal of our research

was to determine PAW's potential to promote the effects of antibiotics against selected MDR bacteria. In addition, the PAW's efficacy was also analyzed through the detection of intracellular RONS in treated bacteria.

2. Materials and Methods

2.1. Experimental Setup and Plasma Treatment of Water Sample

The 3-pin APPJ was used as a plasma source for the production of RONS in a water sample (Figure 1). The system operated with argon (Argon 5.0) as a working gas at a flow rate of 2.5 slm, regulated by a mass flow controller (Mass View MV-304, Bronkhorst High-Tech B.V., Ruurlo, The Netherlands). A detailed description of the 3-pin APPJ has been provided in a previous study [49]. The 3-pin APPJ was constructed by using three syringe needles, functioning as powered electrodes, enclosed within transparent quartz tubes. The needle tips were positioned 5 mm from the endings of the quartz tubes.

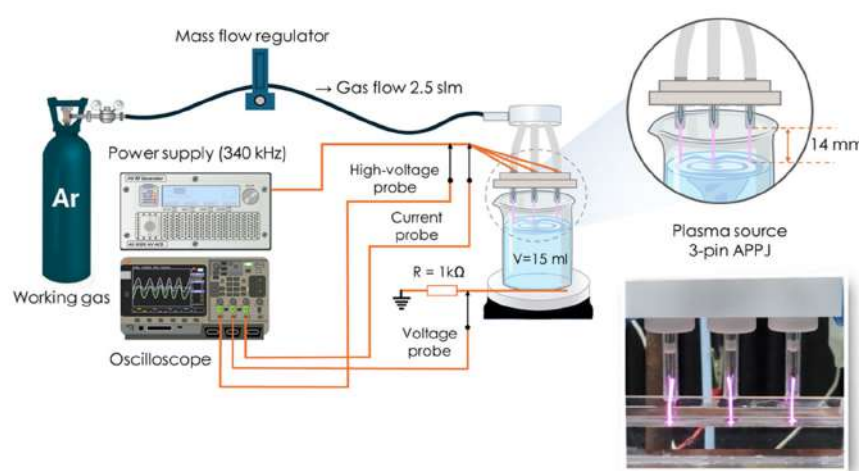


Figure 1. Three-pin atmospheric pressure plasma jet (3-pin APPJ) configuration.

The grounded electrode was placed below a treated water sample, which was placed in a plastic Petri dish under the 3-pin APPJ. A copper tape was attached to the bottom of Petri dish and grounded through a 1 kΩ resistor. The distance between the needle tips and the water surface was maintained at 14 mm.

The 3-pin APPJ was powered by a high voltage power supply (AG 0201 HV-ACD RF HV GENERATOR, T&C Power Conversion, Inc., Rochester, NY, USA) operating at a continuous sinusoidal wave signal of 340 kHz. Electrical parameters, including the high voltage applied to the 3-pin APPJ and the current through it, were continuously monitored and controlled to ensure consistent treatment conditions. A high-voltage probe (P6015A, Tektronix, Inc., Beaverton, OR, USA) and a current probe (N2783B, Agilent Technologies, Tokyo, Japan) were used for these measurements. Additionally, the current passing through the grounded line was determined by measuring the voltage drop across the 1 kΩ resistor (by using probe TPP0201, Tektronix, Inc., Beaverton, OR, USA). The discharge power, calculated as described previously [50], was maintained at an average value of 14 W during all treatments.

To create PAW, which was used for all experiments on bacteria, plasma treatments were conducted on 15 mL of deionized water (Milli-Q, pH 7.3) for 10 min. Prior to treatment, 25 mg of ZnO was added to prevent sample acidification caused by plasma exposure [51].

2.2. Characterization of Plasma-Activated Water

To investigate the effects of plasma–liquid interaction, after filtering the sample to remove remaining ZnO, the pH value (HI1131 pH electrode, HI5521 controller, Hanna

Instruments Inc., Woonsocket, RI, USA) and concentrations of the hydrogen peroxide, nitrate, and nitrite of treated water were determined. All measurements were made in triplicate immediately after the plasma treatment.

The concentrations of three reactive species (H_2O_2 , NO_3^- , and NO_2^-), in PAW were determined photometrically by using a UV-VIS spectrophotometer (UV/VIS Lambda 365, PerkinElmer, Inc., Waltham, MA, USA). Prior to the experiments, calibration curves were made for each reactive species. RONS concentrations exceeding the measurement range of the methods were quantified by diluting the samples with deionized Milli-Q water prior to analysis.

The hydrogen peroxide concentration in treated liquid was quantified using the titanium oxysulfate (TiOSO_4) assay (Titanium(IV) oxysulfate - sulfuric acid solution, Sigma Aldrich Chimie S.a.r.l, St. Quentin Fallavier Cedex, France), which measures the absorbance of yellow colored pertitanic acid formed in the reaction between H_2O_2 and titanyl ions in acidic conditions [52]. One volume of reagent per ten volumes of aqueous solution containing H_2O_2 was added [52] and the absorption peak value at 407 nm was measured. Calibration curves were prepared by using seven different H_2O_2 concentrations (0–50 mg/L) obtained by diluting a 30% stock solution (1.07210.1000 Hydrogen peroxide 30%, Merck KgaA, Darmstadt, Germany).

Nitrate (NO_3^-) concentrations were determined with the Spectroquant Nitrate Test (1.09713.0002, Merck KgaA, Darmstadt, Germany), based on the nitration of 2,6-dimethylphenol to 4-nitro-2,6-dimethylphenol with an absorption maximum at $\lambda = 357$ nm [33]. A calibration curve (0–110 mg/L) was constructed using dilutions of a nitrate stock solution (200 mg/L NO_3^- -N, Merck KgaA, Darmstadt, Germany).

Nitrite (NO_2^-) concentrations were measured by using the Spectroquant Nitrite Test (1.14776.0001, Merck KgaA, Darmstadt, Germany), where NO_2^- reacts with Griess reagent in acidic conditions to form a red-violet azo dye ($\lambda = 525$ nm) [53]. Calibration was performed using dilutions of a nitrite stock solution (40 mg/L NO_2^- -N, Merck KgaA, Darmstadt, Germany) in the range of 0.07–3.28 mg/L.

The pH value of obtained PAW was 6.8 ± 0.1 , and concentrations of H_2O_2 , NO_2^- , and NO_3^- were 56 ± 4 ppm, 55 ± 3 ppm, and 74 ± 4 ppm, respectively. All PAW samples used were checked for sterility by inoculation on tryptic soy agar (TSA).

2.3. Bacterial Strains and Growth Conditions

The reference carbapenem-resistant *K. pneumoniae* ATCC BAA-2814 and the reference vancomycin-resistant *E. faecalis* ATCC 51299 strains were used in the study. The *K. pneumoniae* ATCC BAA-2814 strain displays resistance to aminoglycosides, fluoroquinolones, and beta-lactams, and is considered a model strain for carbapenem-resistance in enterobacteria, while *E. faecalis* ATCC 51299 is high-level gentamicin and vancomycin resistant.

The master stocks were maintained at -20°C with 40% of glycerol. The strains were plated on TSA and incubated aerobically for 24 h at 37°C . A single colony was transferred from the plates into 10 mL of fresh tryptic soy broth (TSB), and following incubation at 37°C for 16 h, a culture was diluted to obtain a final concentration of 1×10^8 CFU/mL determined by using a McFarland densitometer (DensiCHEK plus, bioMerieux, Marcy-l'Étoile, France).

2.4. PAW Treatment and Assessment of Bacterial Inactivation

For each bacterial species tested, an aliquot of 100 μL of the 1×10^8 CFU/mL bacterial suspension was added to 900 μL of PAW in microtubes and homogenized. The samples containing bacterial load and PAW were held at room temperature for two exposure times

(15 min and 60 min) before they were used for further testing. In all experiments, we used bacterial suspensions (100 µL) added to saline (900 µL) as control untreated samples.

The colony forming unit (CFU) assay was used for the assessment of bacterial inactivation by PAW treatment. After the treatments, bacterial suspensions were serially diluted 10-fold in saline. One hundred microliters of each dilution were evenly spread using a bent inoculation loop as a cell spreader on Luria Bertani (LB) agar (for *K. pneumoniae*) or TSA (for *E. faecalis*) plates. After overnight incubation at 37 °C, colonies were counted to determine the number of viable bacterial cells (CFU/mL) and log reduction was calculated using the following equation: $\text{Log}_{10}\text{Reduction} = \log_{10}(\text{CFU}_{\text{control}}/\text{CFU}_{\text{treated}})$. All experiments were carried out in triplicate and repeated three times.

2.5. Determination of Minimum Inhibitory Concentrations (MICs)

Bacteria were grown and then treated with PAW as described above for the assessment of bacterial inactivation. Following the treatments, an aliquot of 100 µL of bacterial suspensions was used to subculture the surviving bacteria and inoculated onto LB or TSA. After overnight incubation at 37 °C, the grown colonies were collected for the preparation of a 1×10^8 CFU/mL next-generation bacterial suspension, which was then exposed to PAW under the aforementioned conditions. The initial culture before the first PAW treatment was considered as generation 0, followed by generation 1 after the first plasma treatment. In total, the procedure was repeated four times, so that four generations of bacteria were treated with PAW and examined for their susceptibility to antibiotics after the first and fourth exposure to PAW.

The procedure for the determination of MICs was identical in both generations, 1 and 4, of the bacterial strains tested as well as for the untreated bacteria, which served as the control. The 1×10^8 CFU/mL suspensions were prepared from untreated or PAW-treated bacteria, and an aliquot of 100 µL of the suspensions was plated on Mueller Hinton (MH) agar plates. The Liofilchem® MIC Test Strips (Liofilchem, Roseto degli Abruzzi, Italy) were used as a quantitative assay for determining the MICs of selected antibiotics for the bacteria tested. The following MIC test strips were used (the antibiotic gradient scale is given in parentheses): for *K. pneumoniae*, ceftazidime (0.016–256 µg/mL), cefotaxime (0.016–256 µg/mL), gentamicin (0.016–256 µg/mL), levofloxacin (0.002–32 µg/mL), imipenem (0.002–32 µg/mL), and erapenem (0.002–32 µg/mL), and for *E. faecalis*, vancomycin (0.016–256 µg/mL), teicoplanin (0.016–256 µg/mL), and imipenem (0.002–32 µg/mL). After 24 h incubation, the MIC values were read directly from the scale, at the point where the edge of the inhibition ellipse intersects with the MIC test strip. In addition, the automated Vitek® 2 (bioMérieux, Marcy-l'Étoile, France) system was used for measuring the MICs of colistin against *K. pneumoniae*. The experiments were performed in triplicate and repeated two times.

2.6. Detection of Intracellular RONS Generated by the PAW Treatment

Two fluorescent probes, MitoSox™ Red (Thermo Fisher, Waltham, MA, USA) mitochondrial superoxide indicator and the OxiSelect™ in vitro ROS/RNS assay kit (Cell Bio Labs, San Diego, CA, USA) were used to detect intracellular reactive species generated by the PAW treatments applied as previously described.

The detection of superoxide anion ($\text{O}_2^{\bullet-}$) by using MitoSOX™ Red reagent was performed in accordance with the procedure described by Guo et al. [46]; namely, the indicator was incubated with each bacterial sample at a final concentration of 5 µM for 30 min at 37 °C. To detect highly reactive oxygen/nitrogen species, the OxiSelect™ probe, prepared according to the manufacturer's recommendations, was incubated with bacterial strains for 40 min in the dark at room temperature. Bacteria were then collected by

centrifugation, washed three times in saline and 200 μ L per treated and untreated bacterial suspensions were transferred to a black 96-well microtiter plate (ThermoNunc 96F, Thermo Scientific, Roskilde, Denmark). Fluorescence measurements were performed using a microplate reader Fluoroskan (Thermo Fisher, Waltham, MA, USA), with an excitation wavelength of 515 nm and an emission wavelength of 578 nm for the MitoSOX™ probe, while the excitation and emission wavelengths of 485 nm and 538 nm, respectively, were used for dichlorodihydrofluorescein (DCF) in the OxiSelect™ probe.

All fluorescence intensities were normalized to the baseline value and results are presented as means $\pm$ standard deviation. Untreated bacterial suspensions in saline were used as controls. Each sample was read in three wells and replicated twice.

2.7. Statistical Analyses

Data were analyzed in the IBM SPSS Statistics for Windows, version 22.0 (IBM, Armonk, NY, USA). Numerical data were described by using average and standard deviation. The Kolmogor–Smirnov test was used to test the distribution of normality of the data. For parametric data, a one-way ANOVA was used to compare more than two groups, and Bonferroni was used for post hoc analyses. Nonparametric data for comparing the two groups were processed with the Mann–Whitney U test. Statistical significance was set at 0.05.

3. Results

3.1. Inactivation Efficacy of PAW

The bacterial species included in the study exhibited different behaviors in the colony count trials, as is depicted in Figure 2. The most notable reduction of bacterial CFU count was observed after a 60 min PAW exposure of the *K. pneumoniae* strain (log reduction = 6.92 ± 0.24), while the log reduction after a 15 min treatment was 1.23 ± 0.39 ($p < 0.001$) (Figure 2a). In contrast, the overall antimicrobial effects of PAW against the *E. faecalis* strain were limited (Figure 2b), regardless of the duration of treatment ($p = 0.073$).

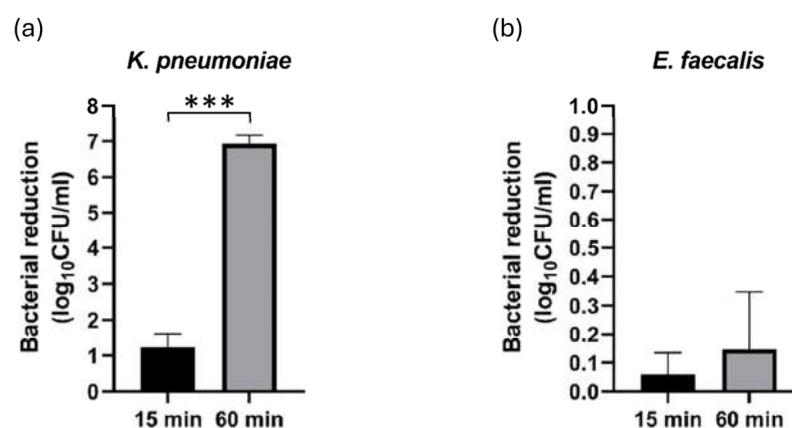


Figure 2. Growth reduction after plasma-activated water (PAW) treatments for 15 and 60 min against (a) *K. pneumoniae* and (b) *E. faecalis*. Data are representative of three independent experiments carried out in triplicates. Statistical significance is denoted as *** $p < 0.001$.

3.2. Effects of PAW on Susceptibility to Antibiotics

By exposing bacteria to repeated PAW treatments across multiple generations, we aimed to create conditions similar to those encountered during real-world PAW application in parallel with the continuous multiplication of bacteria. The summarized readings of MIC values are presented in Figure 3. The European Committee on Antimicrobial

Susceptibility Testing (EUCAST) breakpoints for categorizing *K. pneumoniae* and *E. faecalis* strains as susceptible (S and I) and resistant (R) to the antibiotics were not applied. Only the differences in MIC values of antibiotics included in the testing against untreated and PAW-treated bacterial samples were of interest as indicators of increase or decrease in susceptibility.

Species	Antimicrobial agent	MIC (μg/mL)*								
		Untreated	First PAW treatment				Fourth PAW treatment			
			15 min	Change	60 min	Change	15 min	Change	60 min	Change
<i>Klebsiella pneumoniae</i>	Ceftazidime	32	24	−25%	24	−25%	24	−25%	24	−25%
	Cefotaxime	1	1.5	+50%	1.5	+50%	1	nc	1.5	+50%
	Gentamicin	6	6	nc	6	nc	8	+33%	6	nc
	Levofloxacin	0.5	0.5	nc	0.5	nc	0.5	nc	0.5	nc
	Imipenem	0.25	0.19	−24%	0.19	−24%	0.25	nc	0.25	nc
	Ertapenem	0.47	0.64	+36.2%	0.64	+36.2%	0.32	−32%	0.47	nc
	Colistin	2	0.75	−62.5%	1	−50%	0.75	−62.5%	0.75	−62.5%
<i>Enterococcus faecalis</i>	Vancomycin	64	64	nc	64	nc	48	−25%	48	−25%
	Teicoplanin	0.75	0.38	−49.3%	0.5	−33%	0.5	−33%	0.5	−33%
	Imipenem	2	1	−50%	1	−50%	1.5	−25%	1	−50%

Figure 3. Heat-map plot of minimum inhibitory concentrations (MICs) of selected antibiotics against *K. pneumoniae* ATCC BAA-2814 and *E. faecalis* ATCC 51299 following exposure to PAW for 15 and 60 min across four generations. The change is given with respect to the untreated data. * Data are representative of two independent experiments carried out in triplicates. Nc, not changed.

The effects exhibited by the PAW treatments on *K. pneumoniae* susceptibility to antibiotics varied from antibiotic to antibiotic, even within the same group. This was shown for the third-generation cephalosporins and carbapenems tested. While the susceptibilities to cefotaxime either decreased or remained the same as for the controls, MICs of ceftazidime were reduced by 25% under all treatment conditions compared to the untreated control. The effects of PAW treatments on susceptibility to ertapenem were inconsistent, while MICs of imipenem decreased after the initial PAW treatments (0.19 $\mu\text{g/mL}$). Sensitivities to an aminoglycoside representative, gentamicin, and a fluoroquinolone, levofloxacin, were not affected by PAW treatments. On the other hand, the effects of PAW treatment on *K. pneumoniae* susceptibility to colistin were positive. All modalities of PAW treatments led to a significant decrease in the MIC values of colistin, in comparison to the MIC of 2 $\mu\text{g/mL}$ in untreated control.

The MIC of vancomycin measured against the untreated *E. faecalis* was 64 $\mu\text{g/mL}$, and remained unchanged after the first treatment with PAW. However, after the fourth treatment, the MIC decreased to 48 $\mu\text{g/mL}$ for both 15 and 60 min exposures, which is a noteworthy result. The *E. faecalis* susceptibilities to teicoplanin and imipenem were also positively affected by the PAW treatments, regardless of the conditions applied (Figure 3).

3.3. Assessment of Intracellular RONS Generated by the PAW Treatment

Gram-negative and Gram-positive bacteria differed in the intracellular levels of the reactive species detected by the assays employed (Figure 4). For the *K. pneumoniae* strain (Figure 4a), the fluorescence intensities of the MitoSOXTM probe increased by 2.6-fold ($p = 0.002$) and 3.1-fold ($p < 0.001$) compared to control, after 15 and 60 min of PAW exposure, respectively. In *E. faecalis*, the intracellular concentration of $\bullet\text{O}_2^-$ was positively correlated with the time of exposure (Figure 4b). The increase in fluorescence achieved with 60 min exposure was significantly higher than that measured after 15 min treatments ($p = 0.015$). As compared to the control, the highest fluorescence levels were recorded in *E. faecalis*

cells after PAW treatment for 60 min ($p < 0.001$). As far as long-lived RONS detected with the OxiSelect™ probe are concerned, the fluorescence increases in both *K. pneumoniae* (Figure 4a) and *E. faecalis* cells (Figure 4b) remained below the level of significance, and no correlation with the treatment time was observed.

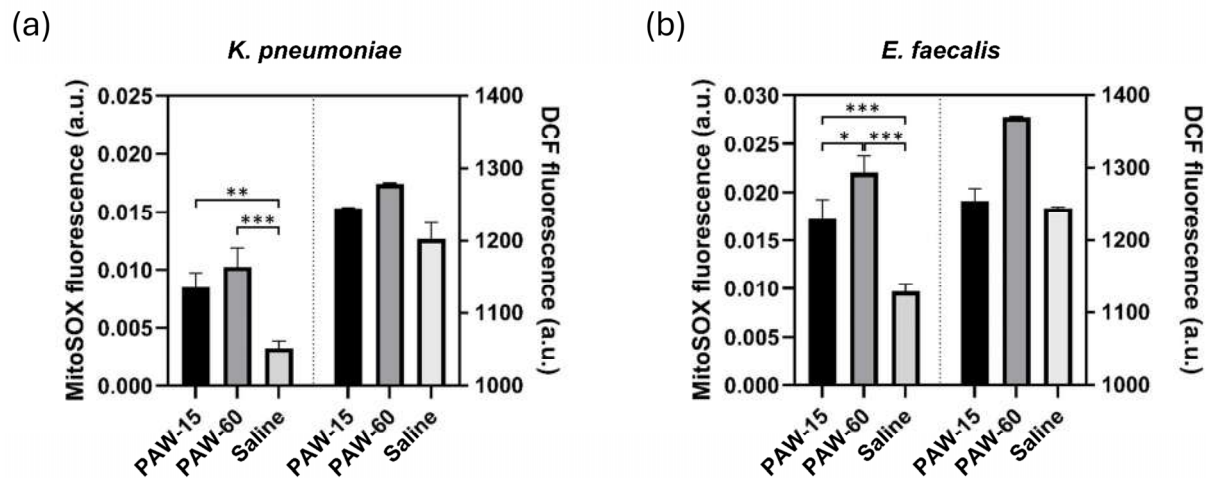


Figure 4. Superoxide anion radical $\bullet\text{O}_2^-$ levels detected by MitoSOX™ and hydrogen peroxide (H_2O_2), peroxy radical ($\text{ROO}\cdot$), nitric oxide (NO), and peroxyxynitrite anion (ONOO^-) levels detected by OxiSelect™ assay (DCF) in (a) *K. pneumoniae* ATCC BAA-2814 and (b) *E. faecalis* ATCC 51299 cells following exposure to plasma-activated water (PAW) for 15 and 60 min. Untreated bacterial cells suspended in saline served as control. Data are representative of two independent experiments. Statistical significance is denoted as * $p < 0.05$; ** $p < 0.01$; and *** $p < 0.001$.

4. Discussion

PAW as an antimicrobial agent is considered environmentally friendly and cost-effective, and its important advantages over direct CAP treatment are convenience and safety in use [35,54,55]. Accordingly, the scope of promising implementations in medicine, agriculture and environmental applications is growing [15,36,56,57]. The medical and dental fields offer broad opportunities for the clinical implementation of PAW as an alternative antimicrobial strategy, ranging from the decontamination of medical and dental devices to in vivo disinfection and infection control [39,55,58,59]. While PAW is already being described as a novel disinfectant, a validated application as an antimicrobial technology has not been realized so far.

The reference strains used in our study were selected for their multiple drug resistance determinants, primarily resistance to carbapenems in *K. pneumoniae* and resistance to vancomycin in *E. faecalis*. Both patterns of resistance have been recognized as major health threats [2,4,5,7], and are therefore adequate targets for testing PAW as a tool in combating MDR bacteria.

K. pneumoniae is a Gram-negative opportunistic pathogen that can cause a variety of HAIs, including pneumonia, sepsis, urinary tract infections, and wound infections [44]. Infections caused by carbapenem-resistant strains are particularly concerning due to their high mortality rates, especially among immunocompromised patients and patients in critical conditions [60]. Therefore, there is a growing necessity for alternative antimicrobial strategies as the effectiveness of last-resort antibiotics against *K. pneumoniae* continues to decline. It is also noteworthy that the species is capable of persisting on various surfaces, and is difficult to eradicate due to resistance to extreme conditions and antimicrobial agents [44]. *E. faecalis* is another example of an opportunistic pathogen, which successfully adapts to and thrives in harsh environments. This Gram-positive bacterium has also been recognized as a major causative agent of HAIs [45]. The species is characterized both by intrinsic

resistance to a variety of antibiotics and by its ability to easily acquire resistance to further antibiotics, such as high-level aminoglycoside resistance, and vancomycin resistance [61].

The PAW tested showed inactivation efficiency against Gram-negative *K. pneumoniae*, which increased with contact times to the samples. The effects on Gram-positive *E. faecalis*, on the other hand, were limited regardless of the exposure time. It has already been shown that Gram-positive bacteria display elevated resistance to plasma treatments either when the bacterial load is in contact with plasma (gas phase or as a suspension) [62–64] or in case of treatments with PAW [41,65,66]. On the other hand, Nicol et al. [67] showed that both Gram-positive and Gram-negative bacteria were similarly affected by the CAP treatments, while Tipa et al. [68] demonstrated a higher efficacy of CAP against Gram-positive than against Gram-negative bacteria. Increased protection against both CAP and PAW treatments in Gram-positive bacteria has been primarily attributed to the robust cell wall, which is characterized by a thick layer of peptidoglycan reaching a size of between 30 and 100 nm [69]. Given that we used a vancomycin-resistant *E. faecalis* strain, it is noteworthy that reduced susceptibility to plasma treatment was reported in strains of enterococci resistant to vancomycin in comparison to the vancomycin-susceptible counterparts [70]. However, the causality of this finding was not demonstrated. The recent study by Droste et al. [42] investigated whether Gram-positive bacteria are generally robust or truly tolerant to PAW treatment, and established that the complete inactivation of multiple species of Gram-positive bacteria was achievable with longer contact times and a higher concentration of RONS in applied PAW. These mixed findings suggest that different susceptibility to PAW treatments between Gram-positive and Gram-negative bacteria is determined by multiple factors encompassing CAP generation and PAW activation settings, operational parameters of the treatments applied to the bacterial samples, and the complex structure of the cell wall and its appendices in bacteria as well as antioxidant mechanisms in different bacterial species and even strains. Given that this list is not exhaustive, there is at least one direction for our further work based on the results obtained so far. The stark difference between the effects exerted on Gram-negative *K. pneumoniae* viability and the effects on the survival of Gram-positive *E. faecalis* observed in our study indicates a need for the further optimization of the PAW's antibacterial activity through the optimization of water activation settings and PAW treatment parameters. We have already shown that the effectiveness of PAW in inactivating bacteria improves by increasing the concentration of RONS (by decreasing the water volume used for PAW preparation) and extending the incubation time of the bacteria in PAW [71]. The inconsistencies in the results presented in the literature, including ours, also show that further studies of bacterial inactivation achieved by different PAWs generated by different systems are needed.

The influence of treatment with CAP and, in particular, PALs on the susceptibility of pathogenic bacteria to antibiotics has not been extensively studied. Hoon Park et al. demonstrated that argon plasma, generated by a dielectric barrier discharge plasma device, increased susceptibility to β -lactams in *Staphylococcus aureus* strains, including methicillin-resistant *S. aureus* (MRSA) [47]. In a more recent study, researchers reported that CAP pretreatment increased the sensitivity of both planktonic and biofilm cells of *Pseudomonas aeruginosa* to several antibiotics [48]. However, it has also been shown that CAP pretreatment could lead to increased resistance to antibiotics [72], probably through the induction of mutations and adaptive changes. In the case of PALs, a study showed that treatment with plasma-activated saline positively affected the sensitivity of MRSA to five different classes of antibiotics [46]. As far as the effects of PAW treatments on bacterial susceptibility are concerned, the data are lacking, while carbapenem-resistant *K. pneumoniae* and vancomycin-resistant enterococcus have not been investigated in this context so far.

We showed that the effects of PAW on bacterial susceptibility to conventional antibiotics are similar to those achieved with CAP and other PALs [46–48,72]. Namely, the effectiveness of PAW treatments in terms of enhancing antibiotic activity against the bacteria tested was not fully consistent, but nevertheless relevant both in Gram-negative and in Gram-positive bacteria. Effects on *K. pneumoniae* susceptibility to third-generation cephalosporins and carbapenems were of interest as enterobacteria resistant to these groups of antibiotics pose the highest estimated burden among all MDR Gram-negative bacteria [4], and are included in the critical priority category in the latest WHO priority pathogen list [7]. With the exception of the MICs of the third-generation cephalosporin ceftazidime that decreased following PAW exposure and, thus, were positively affected, the results obtained with another cephalosporin, cefotaxim, as well as with carbapenems were inconsistent. Nonetheless, the result of particular importance is the decrease in the MICs of colistin as treatment with this antibiotic is considered a salvage therapy for patients infected with drug-resistant *K. pneumoniae* [73]. The MICs of colistin were positively affected by the PAW treatments for both 15 and 60 min, with the decrease ranging from 50 to 62.5%. In contrast to the mixed results obtained for *K. pneumoniae*, PAW positively changed the activity of all antibiotics tested against *E. faecalis*. The reduction in the MIC of imipenem ranged from 25% to 50%, with two-fold reduction achieved in generation 1 regardless of the treatment time. The effects on MICs of glycopeptide antibiotics, which are critically important in the treatment of enterococcal infections, were also favorable. Namely, the MICs of teicoplanin against *E. faecalis* were consistently reduced by over 30% regardless of the PAW treatment parameters, while both 15 and 60 min exposures caused a decrease of 25% in the MIC of vancomycin in generation 4. The summarized findings indicate a possible role of PAW applied as a support to antibiotics in the treatment of MDR infections, but the effectivity of this combinatory approach needs to be tested and verified in further experiments.

We found a significant increase in the superoxide anion concentration in *K. pneumoniae* and *E. faecalis* strains after the PAW treatments. Intracellular superoxide anion is a short-lived free radical which rapidly interacts with different biological and inorganic compounds that could potentially damage and alter the functioning of living cells [74–76]. Even though it is well documented that superoxide anion could have direct detrimental effects on different cellular structures [75–77], the complexity of oxidative stress reactions and interplay of superoxide anion with other cellular components and reactive species should not be taken out of this context. Changes in levels of total RONS were not detected. As far as the total intracellular RONS accumulation is concerned, our findings are similar to those obtained with *S. aureus* cells treated with PAW [46]. Although the findings may at least partially be related to the limitations of the testing methodologies we used [78], they are in line with the RONS present in the PAW used in our study, with the highest content of hydrogen peroxide.

For example, in the process of dismutation, hydrogen peroxide is produced from superoxide anion, which further transforms to hydroxyl radicals and is responsible for extensive damage to various biomolecules [74–77]. In addition, superoxide anion can interact with nitric oxide and form a peroxynitrite, another short-lived and highly reactive oxidant which leads to lipid peroxidation and bacterial membrane disruption [74]. The presumed importance of superoxide anion and its downstream reactive species for the antibacterial properties of PAW indicated in our study is supported in the literature, although for different bacteria samples. The role of these species was proven essential in *Escherichia coli* biofilm removal by PAW treatment [79]. A recent preprint from the same research group stressed the importance of superoxide anion in intracellular RONS accumulation, and linked the finding with 478 up-regulated and 186 down-regulated genes in the *E. coli* genome, which highlights the complexity of the cellular response to PAW expo-

sure [80]. Rothwell et al. [43] also showed the critical relevance of superoxide against both Gram-negative *E. coli* and Gram-positive *Listeria monocytogenes*. Based on our results, the demonstrated effects of PAW on bacterial viability and susceptibility to antibiotics cannot be ascribed solely to superoxide anion and its downstream reactions, but also to oxidative stress during exposure to PAW. The cocktail of RONS both produced and accumulated in bacterial cells post-PAW exposure as well as the damaging effects of PAW-induced oxidative stress are complex and interlinked. It can only be presumed that the oxidative damage of the membrane and intracellular biomolecules resulted in either a loss of viability or in more subtle changes at the level of DNA and proteins, which led to changes in susceptibility to antibiotics [22,42,46,66]. However, more detailed mechanistic studies to understand the effects observed, particularly how PAW specifically alters susceptibility to antibiotics, are needed.

There are several limitations to the present study. Firstly, only laboratory-adapted reference strains that may not represent the full ecological and functional diversity of the species were included. Nevertheless, the presumed genetic differences between reference *K. pneumoniae* and *E. faecalis* strains and their clinical and environmental counterparts are of limited relevance in studies like ours, which are not focused on properties such as genomic diversity or virulence. Secondly, only two species of MDR bacteria were included as representatives of Gram-positive and Gram-negative bacteria, which limits the generalizability of the findings. Most of the other relevant Gram-positive and Gram-negative MDR bacteria have already been presented in the literature related to the antibacterial properties of CAP and/or PAL. The species that we have selected for this study have not been extensively investigated as specific targets for the antimicrobial activity of PAW and, especially, related changes in antibiotic sensitivity. This particularly applies to *K. pneumoniae*, which gave us an added impetus to include this species in our study. Thirdly, to obtain a deeper understanding of the processes involved in the antibacterial efficacy of PAW, the panel of testing procedures used for the detection of intracellular RONS needs to be expanded.

5. Conclusions

This study demonstrated, on the one hand, the inactivation activity of PAW against the superbug *K. pneumoniae*, and, on the other hand, the need for further optimization of water activation and treatment settings in order to achieve the inactivation of the Gram-positive bacterium *E. faecalis*. We investigated the effects of PAW treatments on bacterial susceptibility to traditional antibiotics, as an underexplored aspect of the activity of this emerging biocidal agent. The PAW-induced decreases in the MIC values of critical antibiotics, such as colistin against *K. pneumoniae*, and glycopeptides against *E. faecalis*, were observed. The overall findings indicate that PAW's inactivating efficacy coupled with its capacity for the potentiation of antibiotic effects is a promising combination against MDR bacteria, while also underscoring the importance of further research into the exact mechanisms and reaction pathways that influence the bacterial response to PAW. The optimization of PAW properties, as one of the next steps in the research, should be performed in the context of particular bacterial species, represented by clinical and environmental isolates in addition to the reference laboratory strains.

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Abbreviations

The following abbreviations are used in this manuscript:

PAW	Plasma-activated water
MDR	Multidrug-resistant
MICs	Minimum inhibitory concentration
AMR	Antimicrobial resistance
WHO	World Health Organization
CAP	Cold atmospheric plasma
RONs	Reactive oxygen and nitrogen species
ORP	Oxidation reduction potential
PALs	Plasma-activated liquids
HAIs	Healthcare-associated infections
APPJ	Atmospheric pressure plasma jet
slm	Standard liters per minute
ATCC	American Type Culture Collection
TSA	Tryptic soy agar
TSB	Tryptic soy broth
LB	Luria Bertani
CFU	Colony forming unit
MH	Mueller Hinton
DCF	Dichlorodihydrofluorescein
SPSS	Statistical Package for the Social Sciences
EUCAST	European Committee on Antimicrobial Susceptibility Testing
MRSA	Methicillin-resistant <i>S. aureus</i>

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RESEARCH ARTICLE

Experimental Study and Modeling of a KHz Atmospheric Pressure Streamer Jet in Contact With Water: Application for PAW Generation and Toxicity Evaluation

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ABSTRACT

We present characterization of atmospheric pressure Ar streamer jet, diagnostics of physico-chemical properties of Plasma Activated Water (PAW), and modeling of Reactive Oxygen and Nitrogen Species (RONS) deposited in PAW. Additional focus is on the toxicity of the created PAW to model plant *Lemna minor*. The streamer jet is characterized by analyzing electrical properties, gas-phase characteristics through plasma emission, and laser-induced fluorescence of OH(X) radicals. Measured OH densities are in the range of 10^{20} m^{-3} depending on the mean power deposited to plasma. Gas phase species are linked to measured RONS concentrations, while modeling results confirmed the production mechanisms of H_2O_2 , NO_2^- and NO_3^- species. Experiments with *Lemna minor* show both favorable and negative effects of PAW depending on the PAW concentration.

1 | Introduction

Non-equilibrium plasma interacting with liquids has emerged as a promising approach in the development of sustainable agriculture, where the generation of plasma-activated water (PAW) offers new opportunities for supporting agriculture with minimal environmental impact [1–5]. The importance of PAW in plasma agriculture is reflected in the wide spectrum of applications explored so far, including seed germination and priming [6–10], plant fertilization [11–13], inactivation of enzymes and pathogens [14–17], and food processing [18].

In the first place, plasma/liquid interaction can be controlled by positioning the active plasma volume with respect to the liquid

surface. Plasma sources operated directly in liquids are highly efficient for treating large water volumes and degrading dissolved pollutants [19, 20]. However, their applicability in PAW synthesis is limited by low cost-effectiveness and high temperature, suppressing the formation of long-lived active species [21]. Correspondingly, discharges initiated in the gas phase above a liquid are more beneficial approach when it comes to application. In those systems, tuning of PAW properties is associated both to the changes in the liquid target (treated volume, thickness of plasma/liquid interface, pH, conductivity, etc.) and plasma properties. Among these parameters, the conductivity and grounding of the target have a significant influence on the gas-phase composition and, therefore, on the

concentrations of reactive species in the liquid samples [22–25]. The interlinking that exists between plasma operating parameters and liquid parameters makes this task complicated and necessitates comprehensive diagnostics of both plasma and liquid to analyze important processes and establish control over the generation of PAW.

Previous studies have employed a variety of plasma sources for this purpose, including dielectric barrier discharges (DBD)/DBD-like discharges [26–28], corona discharges [29], microdischarges [30], microwave discharges [31, 32], and plasma jets [33, 34]. The latter are often used to provide fundamental understanding of the processes induced by the discharge in liquid phase due to very controlled action [35, 36]. Studies have demonstrated that plasma jets possess significant potential for transferring highly reactive species from the gas to the liquid phase, facilitated by the gas flow in the jet [33, 37]. The working gas composition, together with water vapor present in the active plasma volume, governs the chemistry and reactivity of the discharge above water. Apart from the jet configuration, both the driving frequency and the discharge type strongly influence plasma chemistry. Consequently, the spectrum and concentrations of reactive species generated in PAW and the energy input result in distinct reactions occurring in both gas and liquid phases [38]. Various diagnostic techniques are used to characterize gas-phase plasma behavior from electrical characterization [22, 39], optical emission spectroscopy (OES) [24, 38], laser-induced fluorescence (LIF) [35, 40], mass spectrometry [41–43], etc.

The presence of a liquid interface makes gas-phase chemistry a complex system with numerous reactive species. A way to deal with this is employing modeling together with experimental diagnostics to resolve chemical kinetics, estimate concentration, and formation pathways of the reactive species [44, 45]. Recent review studies [46] emphasize the need to link short-lived reactive species in the gas phase with the formation of long-lived species in PAW. However, studies combining experimental and modeling approach that directly connect gas-phase diagnostics with liquid-phase chemistry remain limited. In this work, we combine gas- and liquid-phase diagnostics with a one-dimensional gas–liquid model to observe the spatial and temporal evolution of OH radicals and their role in the formation of H_2O_2 , NO_2^- , and NO_3^- .

For successful application in plasma agriculture, PAW should contain high concentrations of the long-lived reactive species (H_2O_2 , NO_3^- , NO_2^-) that remain stable over time, since immediate use is not always possible [47]. However, for some organisms, water with high concentrations of RONS can cause toxic effects due to unfavorable conditions. In order to investigate, we have selected a well-established model plant. *Lemna minor* (Common Duckweed) is an aquatic plant widely used as a model plant in the literature for evaluating water quality and environmental toxicity—for wastewater treatment and phytoremediation [48], biomonitoring of aquatic habitats [49], as a food source [50], and as a biofuel [51]. This freshwater plant is employed in different ecological and toxicity experiments with various pollutants due to its traits, including fast growth, easy maintenance in various experimental setups, and easy extraction of results [52]. Considering existing data in the literature about toxic effects to *Lemna minor* and the straightforwardness of biological experiments, we have chosen this plant for testing the toxicity of PAW.

This work presents an experimental and modeling study of mechanisms of PAW synthesis by using the atmospheric pressure streamer jet (APSJ). The research aimed to gain a deeper understanding of the reaction pathways occurring in both gas and liquid phases. A working hypothesis was that OH radicals play an important role in gas-phase processes, influencing subsequent reactions in the liquid phase responsible for the formation of long-lived species in PAW (H_2O_2 , NO_2^- , and NO_3^-). To verify this, the APSJ was thoroughly characterized by examining its electrical properties, gas temperature, and the presence of short-lived reactive species in the gas phase. Moreover, the chemistry in the liquid phase was tackled by using a one-dimensional gas–liquid model, employing discharge parameters and experimentally determined OH concentrations in the gas phase as input data. Finally, the potential phytotoxic effects of PAW containing these long-lived species were evaluated using Common Duckweed (*Lemna minor*) as a model plant system. Importantly, natural river water was used as the liquid matrix, allowing us to study PAW effects in conditions representative of real aquatic environments. As the use of PAW with complex and variable matrix of natural river water has been explored to a very limited extent, the present study provides a new insight into this application.

2 | Materials and Methods

2.1 | Atmospheric Pressure Streamer Jet (APSJ)—Experimental Setup

A schematic illustration of an experimental setup with a streamer-like discharge in contact with a water sample is shown in Figure 1. The APSJ consisted of a stainless-steel powered electrode (diameter 1 mm) with a sharpened tip positioned along the axis of the glass tube (inner diameter 4 mm, and outer diameter 6 mm) and covered with a ceramic insulating tube. A detailed description of the APSJ can be found in [39, 53]. The plasma was generated between a powered electrode and a water surface separated by a gap of 10 mm. For this, a commercial HV high-frequency power source (T&C Power Conversion AG 0201HV-OS 140 W–500 kHz) was used to provide a continuous high-voltage sinusoidal signal at 330 kHz. A 4 mL water sample was placed in a well of a six-well microtiter plate (MTP6, TC-Platte 6 Well, Standard, F, Sarstedt, Germany) positioned below the APSJ was considered as the grounded electrode, and the bottom of it was grounded from the outer side using a copper tape. For gas phase analysis via optical diagnostics techniques, the sample was placed in a quartz vessel (height: 15.6 mm; diameter: 25 mm; wall thickness: 2.3 mm) to prevent cutoff of the UV spectral range. Pure argon (Ar 5.0) was used as the working gas at a fixed flow rate of 1 slm. The flow was regulated by a mass flow controller (Omega FMA5400/5500).

2.2 | Electrical Characterization of APSJ

Optimization of the electrical parameters of the APSJ by examining the discharge regimes enables efficient transfer of the electrical energy into plasma and control of the discharge power. The electrical circuit was designed to allow recording of both the total load applied to the APSJ and the current flowing through the discharge gap and liquid sample separately. This

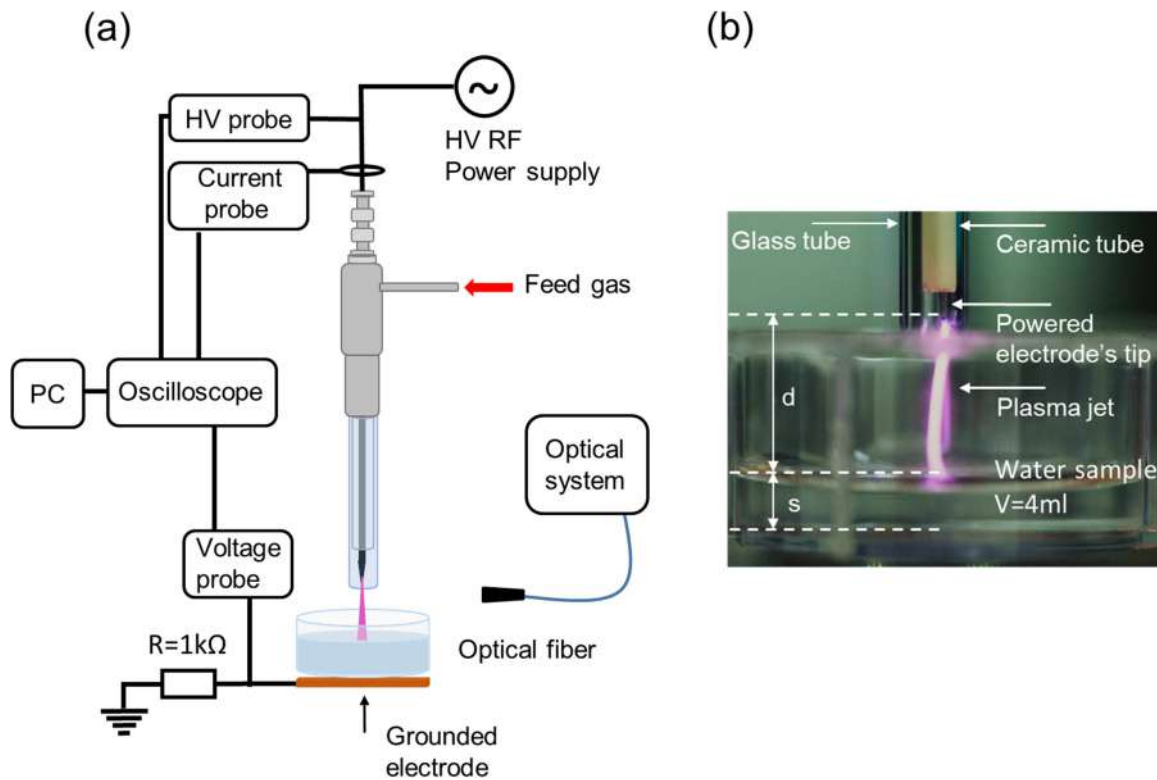


FIGURE 1 | (a) Schematic diagram of experimental setup used for water treatment, electrical and optical measurements, (b) image of plasma in contact with water: $d = 10$ mm—distance between the powered electrode's tip and water surface, $s = 6$ mm—thickness of the distilled water layer in a well of MTP6.

allowed obtaining volt-ampere (V - I) characteristics in both parts of the circuit (high-voltage and ground) and to provide insights into the system behavior.

A commercial HV high-frequency power source (T&C Power Conversion AG 0201HV-OS 140 W-500 kHz) with a continuous sinusoidal signal at 330 kHz was used to generate the plasma. The power supply used in the experiment acted as a source of power, whereas V - I waveforms were monitored at the powered electrode in order to determine APSJ electrical properties. A HV (Tektronix P6015A) probe was utilized to measure the instantaneously applied voltage signal $v(t)$ at the power electrode. The current waveforms $i(t)$ were monitored by a current probe (Tektronix TCP0030A) attached to the main line of APSJ.

Electrical measurements in the ground path were performed to determine the current passing through the discharge gap and the liquid sample $i_G(t)$. By using a voltage probe (Agilent N2863B), connected to a Tektronix MDO3024 oscilloscope, we measured the voltage drop $v_G(t)$ across a $R = 1$ k Ω resistor connected in series to the ground line of the electrical circuit, including the water sample (see Figure 1a). The bandwidths of all probes were sufficient to capture additional fluctuations due to streamer discharge behavior in the recorded signals. Current flowing through the sample was calculated from the measured voltage drop, as follows:

$$i_G(t) = \frac{v_G(t)}{R}. \quad (1)$$

Monitoring of voltage and current waveforms during plasma treatment allowed us to determine an optimal set of parameters

at which the discharge regime is stable and reproducible. Discharge power has been measured to ensure steady conditions of plasma treatment. The mean power dissipated in the ground line of APSJ was evaluated as follows:

$$P_{\text{MEAN}_G} = \frac{1}{nT} \int_0^{nT} v(t) \cdot i_G(t) dt, \quad (2)$$

where n is the number of periods T of the calculated signal (usually 3–5 periods).

In order to make precise calculations of the consumed power, the power was measured in the plasma-ON and plasma-OFF regimes. This approach allowed us to distinguish the power dissipated in the discharge (NET) from contributions associated with parasitic capacitances, leakage currents, and capacitive coupling to surrounding components. To account for these baseline losses, the NET plasma power is obtained by subtracting the mean power measured without plasma from that measured under plasma ignition:

$$P_{\text{MEAN}_G(\text{NET})} = P_{\text{MEAN}_G(\text{ON})} - P_{\text{MEAN}_G(\text{OFF})}. \quad (3)$$

2.3 | Gas Phase Analysis of APSJ

2.3.1 | Optical Emission Spectroscopy

OES served two purposes: to detect excited species in the gas phase and to determine the gas temperature from the rotational structure of the electronically excited $\text{OH}(A^2\Sigma^+ - X^2\Pi)$. A schematic representation of the optical system is shown as an

inset in Figure 1. The time-integrated broadband emission spectra of the APSJ were recorded in the wavelength range from 200 to 900 nm by using Maya2000 Pro-UV-NIR Ocean Insight High-Sensitive Spectrometer with optical resolution of 0.5 nm. The spectrometer was equipped with a 2 m long optical fiber (M114L02) with a 600 μm core diameter, coupled with a collimating lens (Ocean Optics UV74), positioned perpendicular to the APSJ axis at a distance of 5 cm from the discharge region, to capture an integrated emission signal across the discharge gap. Spectra were recorded with an integration time of 10 ms and averaged over 10 measurements.

The rotational structure of the electronically excited OH ($A^2\Sigma^+ - X^2\Pi$) was measured using Zolix Omni- λ 750i spectrometer equipped with an Andor Solis iStar DH334 ICCD camera, providing a high-resolution spectrum in the optical range from 304 to 312 nm. Optical emission was collected with an optical fiber and analyzed by a monochromator (2400 grooves mm^{-1} grating, 250 nm blaze) using a 15 μm entrance slit. The spectra were used to estimate the rotational temperature (T_{rot}) of OH($A^2\Sigma^+ - X^2\Pi$), assuming a Boltzmann distribution of rotational levels. In turn, T_{rot} was assumed to be equal to the gas temperature (T_{gas}) owing to the fast rotational-translational energy transfer (< 1 ns), thus fulfilling the requirement for the translational-rotational equilibrium: $T_{\text{rot}} \approx T_{\text{trans}} (= T_{\text{gas}})$ [54, 55]. The OH($A^2\Sigma^+ - X^2\Pi$) rotational structure was fitted to a simulated one using the MassiveOES software [56, 57]. All emission spectra were background-corrected, and the intensity calibration of the spectrometers was performed using a tungsten-halogen lamp (250–2400 nm, NIST).

2.3.2 | Laser-Induced Fluorescence Spectroscopy

The ground state density of OH($X^2\Pi$) was measured in the middle of the discharge gap using laser-induced fluorescence (LIF). More information on the experimental arrangement can be found elsewhere [40]. In brief, the fluorescence of OH($A^2\Sigma^+ - X^2\Pi$) was generated using a frequency-doubled dye laser (Sirah Cobra-Stretch) with Rhodamine 6 G dye, pumped by a Nd:YAG laser (Quantum-Ray Lab-Series). The output at 283 nm, produced after a second harmonic generation unit, was focused on the center of the discharge gap with a 500 mm bi-convex lens.

The LIF measurements involved exciting the f1(4) rotational level of OH $X(v'' = 0)$ at 283.463 nm to the F1(1) rotational level in OH $A(v' = 1)$, corresponding to the P1(4) transition. The resulting fluorescence was observed at 308.9 nm and 314.5 nm, which represent the OH $A(v' = 1) \rightarrow OH X(v'' = 0)$ and OH $A(v' = 1) \rightarrow OH X(v'' = 1)$ transitions, respectively. The fluorescence signal was monitored using an ICCD camera (Hamamatsu C8484-05G) equipped with a UV-sensitive macro lens (UV-VIS-IR 60 mm 1:4 Apo Macro). Additionally, a bandpass optical filter centered at 310 nm with a FWHM of 10 nm was used to filter out interfering emissions from other transitions. LIF images were acquired with a 500 ns camera gate width, with 5 on-chip integrations per image and 20 images recorded per condition. The mean signal intensity and its standard deviation were determined and used for calibration. To account for the plasma-produced background OH(A-X) signal, a background image was captured by shifting the laser away from the transition while keeping the same laser energy, and this was later subtracted from the image containing the LIF

signal. The measurements of the LIF signal intensity as a function of laser energy were performed, and the slope obtained from the log-log plot was found to be 0.97 ± 0.04 in the laser energy range from 1.3 to 35 μJ .

Absolute density calibration was performed using a four-level kinetic model calibrated with Rayleigh scattering at 566 nm in air. OH calibration followed a published procedure [40, 58] and employed LIFBASE software [59] to evaluate temperature-dependent level populations, spontaneous emission, and absorption. Experimentally measured T_{gas} was used to determine the quenching rate and Boltzmann population of the rotational states. The gas composition in the probed point was considered to be Ar and H_2O only. The water concentration was determined by analyzing the decay of the fluorescence signal [60] and found to be 2.5%. Vibrational energy transfer and quenching rate coefficients of OH($A^2\Sigma^+$) by Ar and H_2O were the same as those used in [58, 61].

2.4 | Plasma Treatments, PAW Characterization, and Toxicity Testing

Initially, distilled water was treated to investigate how parameters such as treatment time and discharge power influence the formation of long-lived reactive species (H_2O_2 , NO_2^- , NO_3^-) in PAW. These treatment parameters were also used as input for the numerical model, enabling a direct comparison between experimental results and model predictions. During the treatment, the temperature of the water sample was monitored, while physicochemical properties were determined before and after treatment, including pH (Hanna Instruments, HI1131 pH electrode, HI5521 controller), electrical conductivity (Hanna Instruments, HI76312 electrode, HI5521 controller), and the concentrations of H_2O_2 , NO_2^- , and NO_3^- . All measurements were performed in triplicate. In addition, the reduction of sample volume caused by gas flow and heating was measured after each experiment, and the PAW results were normalized accordingly.

The concentrations of three reactive species (H_2O_2 , NO_2^- , NO_3^-) in PAW were determined photometrically by using a UV-VIS spectrophotometer (Perkin Elmer Lambda 365) [62]. Briefly, H_2O_2 was quantified by the TiOSO_4 assay based on absorbance at 407 nm [63], while NO_2^- and NO_3^- were detected by using commercial test kits (Spectroquant, Merck) with absorbance measured at 525 and 357 nm, respectively.

Temperature in the treated liquid was monitored using an optic GaAs-based sensor positioned at the end of an optical fiber (Opsens Solutions, IMP00114 OTG-Q, IMP0073 Tempmonitor controller). The method is applicable in the temperature range from -40°C to $+250^\circ\text{C}$ with an accuracy of $\pm 0.8^\circ\text{C}$. The probe was immersed in the water from above (see Figure 2), and measurements were performed at three distances from the APSJ axis: 7, 10, and 17 mm. Average temperatures were calculated from three independent measurements.

To address more practical conditions relevant to plasma agriculture, experiments aimed at assessing the potential toxicity of PAW under realistic environmental conditions, such as its possible release into natural or groundwater systems, were performed. To assess the toxicity of PAW, its effects on the model plant *Lemna minor* (*L. minor*) were examined [48].

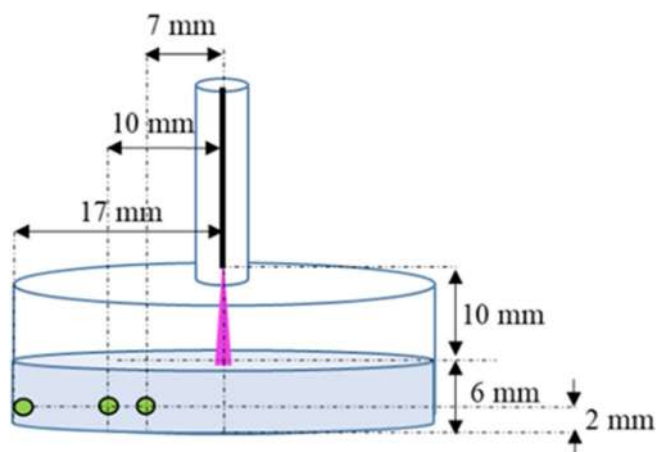


FIGURE 2 | Positions of the thermo-optical probe used for water temperature measurements during plasma treatment. Green circles indicate the measurement points within the sample and their corresponding distances from the APSJ.

Before the experiment, the plants were cultivated in a glass container with TW. The medium was supplemented with a commercial nutrient solution (Easy-Life Profito, 100 μL per 1 L). The container was kept in a climate chamber with a 12 h/12 h light–dark cycle. The temperature was maintained at 20°C–21°C, with a relative humidity of 80% $\pm$ 5%.

For these experiments, PAW was also generated from tap water (TW), which was chosen as a convenient intermediate case between distilled water and realistic water systems [64, 65]. The tap water used in our study is certified as potable according to national standards, and its composition is routinely monitored by the local water supply authority. This implies that almost all harmful precursors are absent or present only at trace levels [46]. Carbonic compounds and residual chlorine are the only those that could be involved in the interaction chemistry [66]. PAW was obtained from 15 ml of TW treated for 10 min, and RONS concentrations, pH value, and electrical conductivity were measured. Then, two different matrices were used for diluting the PAW: tap water (TW) and river water (RW). The Danube River was used for dilution due to its importance as a major European river. RW was collected from the Danube River in Belgrade on the day of the experiment. It was subsequently filtered through standard filter paper. In each matrix, four PAW dilutions were prepared with final concentrations of 10%, 30%, 60%, and 100%. TW and RW without PAW were used as controls.

For each treatment, 25 mL of water was transferred into 50 mL laboratory beakers containing 20–25 *L. minor* plants. The water was supplemented with 2.5 μL of nutrient solution. The beakers were placed in a climate chamber under the same conditions as used during pre-cultivation. The exposure period lasted 17 days. Each treatment was performed in triplicate (three beakers per sample). To evaluate the impact of PAW on plant growth and physiology after the exposure period, one of the most common measured endpoints in *L. minor* experiments, the frond area, the root length, and chlorophyll content were measured.

After 17 days of exposure, the plants were photographed in order to determine frond area and root length. A Nikon D200 camera equipped with a Micro-NIKKOR 105 mm f/2.8 lens was

used for image acquisition. The images were processed with the software package ImageJ 1.54 d. Mean values of frond area and root length were calculated, along with standard errors, based on three independent measurements. Statistical comparisons were conducted by using the Standard Student's *t*-test (OriginLab), with a significance level of $p < 0.05$.

Chlorophyll content was determined using a modified method [67, 68] with dimethyl sulfoxide (DMSO, Merck, 99.5%). Plant samples were vortexed (VELP Scientifica ZX3) in DMSO for 10 s and incubated at 60°C for 7 h in an ultrasonic bath. After centrifugation (Cole Parmer #17250-10, 30 min, 15 mL tubes), absorbance was measured at 433 nm and 665 nm, wavelengths linearly correlated with chlorophyll content [69–71]. The measurements were performed in duplicate. The error bars for the determination of chlorophyll content were estimated to be approximately 30%. Chlorophyll a and b contents were determined using the formulas:

$$C_a = 12.19A_{665} - 3.45A_{649}, \quad (4)$$

$$C_b = 21.99A_{649} - 5.32A_{665}. \quad (5)$$

2.5 | Water Chemistry Model

The influence of plasma-induced reactive species on liquid-phase chemistry using a time-dependent one-dimensional reaction–diffusion model was implemented [72]. To gain a more detailed understanding of the experimental results, we clarify the temporal evolution of liquid-phase species concentrations at a depth of 1 μm near the gas–liquid interface and at a depth of 1 mm within the bulk water region. In the model, the components flux into the water surface and the plasma-induced chemical species flux are determined by Henry's law, which governs the equilibrium between the gas-phase and liquid-phase concentrations of the chemical species. Here, the plasma-derived reactive oxygen species and nitrogen species under consideration are selected as OH radicals (measured experimentally), along with NO , NO_2^- , and NO_3^- . A numerical study on the effect of humid air impurities on plasma jet chemistry [73] suggests that the concentrations of these species in the afterglow region are similar. To simplify the discussion, the gas-phase concentrations of all these species are set to $1.0 \times 10^{20} \text{ m}^{-3}$ (this value is of the same order of magnitude as the experimentally measured OH radical concentration).

3 | Results and Discussion

3.1 | Electrical Characterization

The electrical characterization of the APSJ system was carried out in order to analyze the discharge behavior and to determine the operating conditions most suitable for subsequent treatments. Figure 3 shows the acquired signals of applied voltage $v(t)$ (solid line), current $i(t)$ through the APSJ's main line (dotted line), and current $i_G(t)$ through the sample and the grounded line (dash-dotted line). Each signal was obtained by averaging 16 consecutive waveforms recorded by the oscilloscope. This allowed us to record and analyze the waveforms of both currents, as well as to obtain V – I characteristics in the main and ground parts of the electrical circuit for a better understanding of system behavior after plasma ignition.

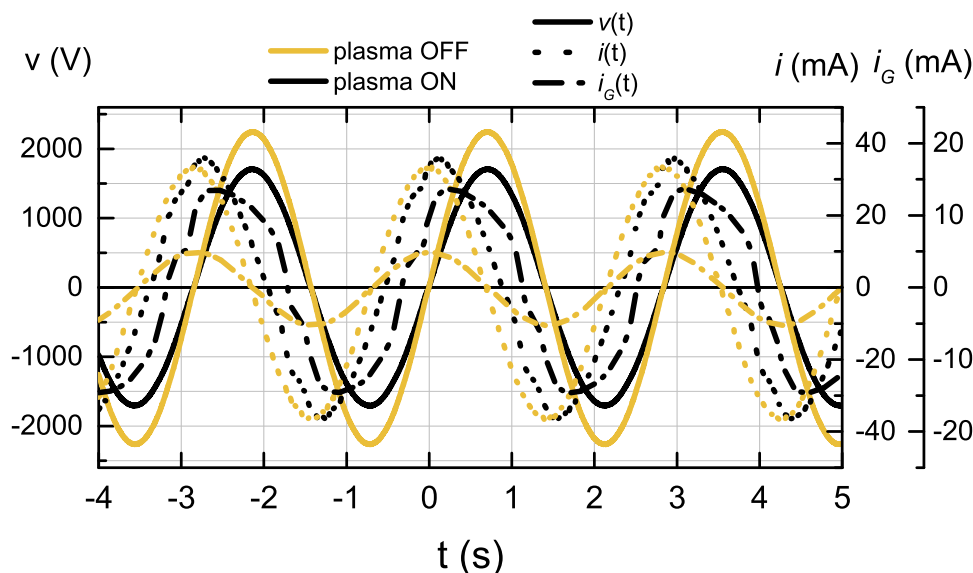


FIGURE 3 | Measured high voltage $v(t)$ (solid line), current $i(t)$ (dotted line) at APSJ's main line, and current $i_G(t)$ through the grounded sample (dash-dotted line). The applied peak-to-peak voltage was 3.4 kV, with a frequency of 330 kHz. Measurements were performed using Ar at a total flow rate of 1 slm (plasma-ON). Yellow lines represent the plasma OFF (without gas flow), with an applied peak-to-peak voltage of 4.5 kV.

The applied high-voltage signal $v(t)$ maintained a stable sinusoidal waveform, independent of the presence of plasma. In the absence of gas flow (yellow lines), the applied voltage remained below the breakdown threshold in air, and the resulting current waveforms, $i(t)$ (yellow dotted line) and $i_G(t)$ (yellow dash-dotted line), retained a sinusoidal shape. Without plasma, the system showed capacitive behavior, with the current $i(t)$ leading the voltage by 86° . This phase shift deviated from the ideal 90° , indicating the presence of parasitic losses in the electrical circuit. The existence of such losses, even in the absence of plasma, suggests that the power delivered to the system should be calculated in all regimes to quantify energy losses accurately.

After plasma was ignited, both currents deviated from the sinusoidal form and exhibited four peaks—two in each half-cycle of the applied voltage that were due to the streamer-type discharge [39].

With plasma ignited, the phase shift between current and voltage signals decreased. Current signal $i(t)$ led voltage by 66° , while current signal measured in the grounded line, $i_G(t)$, led voltage by 41° . The observed difference in phase shift between $i(t)$ and $i_G(t)$ can be attributed to the fact that they were measured in different parts of the discharge circuit. The current measured at the powered electrode included part of the circuit with dominantly capacitive-based impedance. In contrast, the grounded current $i_G(t)$ passes through the entire plasma channel, the plasma-liquid interface, and the external resistor, which together present a higher and more resistive impedance. Consequently, these impedance differences result in a smaller phase lead of $i_G(t)$ compared to $i(t)$.

For each of the three time-dependent signals recorded during the experiment, root mean square values were calculated: V_{RMS} - high-voltage applied to the main line of APSJ, I_{RMS} - current through the main line of APSJ, and I_{RMS_G} - current through the sample and the ground line. The discharge power P_{MEAN_G} (NET) was calculated by using recorded voltage and current signals (see Equations 1, 2, and 3). V - I characteristics obtained

for the main line and for the ground line of APSJ are shown in Figure 4a,b, respectively.

Applied RMS voltage was varied in the range from 0.9 to 1.7 kV by increasing the power at the output of the power supply. Before plasma inception (RMS voltage below 1.7 kV), V - I characteristics in both lines of the electrical circuit were linear (indicated as “without plasma” in Figure 4a). The current I_{RMS} was between 13 and 26 mA, while the discharge current I_{RMS_G} ranged from 2 to 5 mA. Impedances derived from the V - I characteristics of I_{RMS} and I_{RMS_G} were 68 k Ω and 364 k Ω , respectively.

The sudden voltage drop following plasma ignition reflects the fast breakdown of the gap with a drop in total impedance. This part of the V - I characteristic is marked as “with plasma” in Figure 4a. The voltage and current initially decreased to 1350 V and 19.5 mA (red circled data point in Figure 4a). I_{RMS_G} decreased to 3.6 mA (marked with a red arrow in Figure 4b) corresponding to creation of corona discharge at the electrode tip, which did not cause significant changes in the impedance. A further increase in power resulted in a transition to the streamer discharge mode. A conductive streamer channel was established between the pin of the electrode and the liquid surface, with visible propagation about 3 mm above the water. Numerous smaller filaments then spread laterally along the surface. A steady increase in both currents was observed, manifested through the broadening of the streamer channel. P_{MEAN_G} (NET) increased with voltage, reaching 0.6 W at $V_{\text{RMS}} = 1.7$ V before discharge was established. With corona discharge measured power was 0.02 W. After formation of the streamer regime, P_{MEAN_G} (NET) generally increased with increasing power delivered to the APSJ, despite minor fluctuations in voltage and current. Determination of power allowed us to monitor the stability of the discharge, and for selected working conditions of the APSJ, we performed OES to obtain an insight into plasma chemistry and the generation of reactive species.

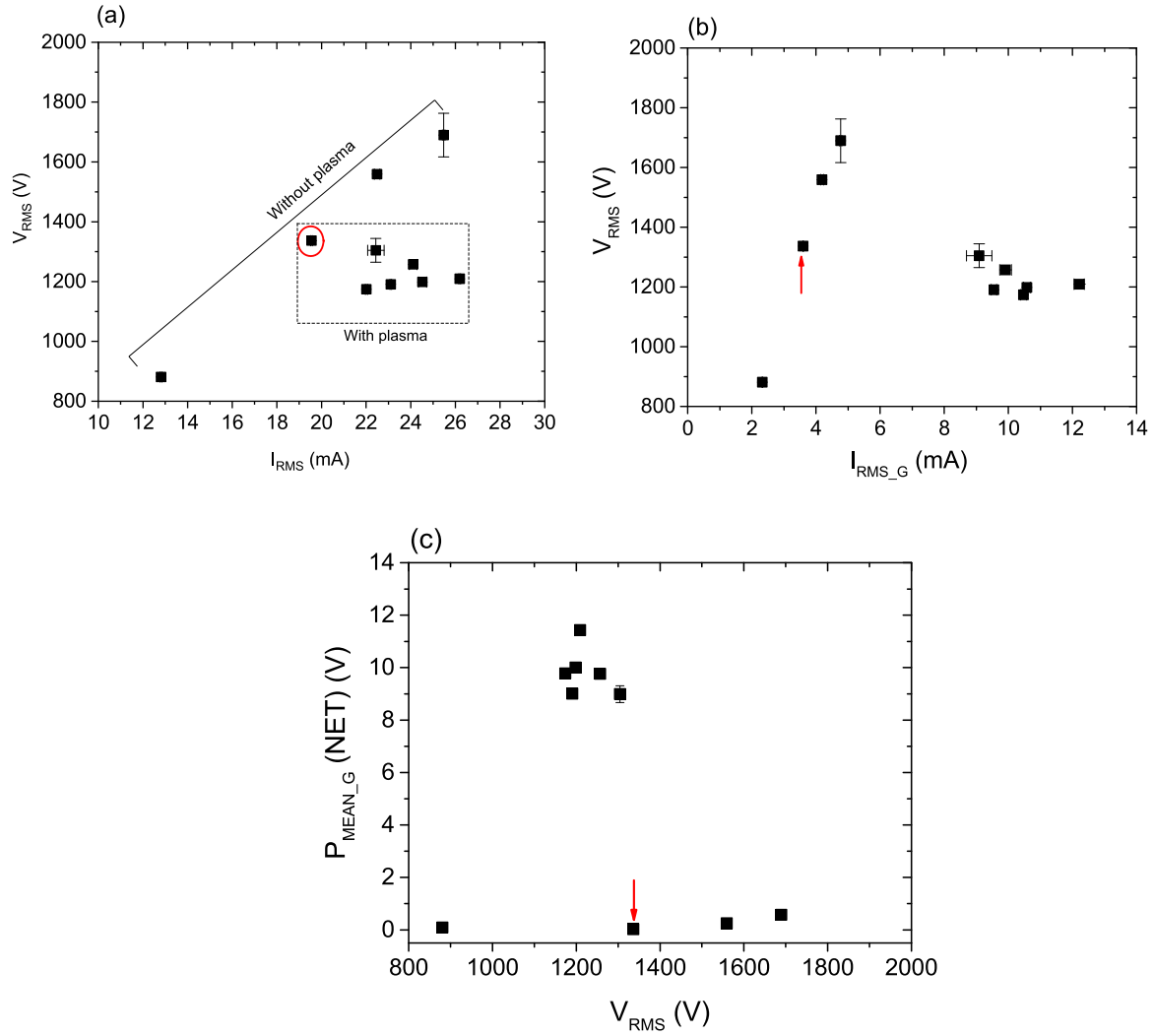


FIGURE 4 | V - I characteristics of the APSJ, dependence of RMS voltage on the RMS current (a) through APSJ main line - I_{RMS} , (b) through the sample and ground line - I_{RMS_G} , and (c) dependence of mean power dissipated in the ground line P_{MEAN_G} (NET) on RMS voltage. The circled point (red circle) in (a) indicates the first data point in the regime with plasma. The corresponding operating point is marked by a red arrow in (b) and (c). Standard measurement errors from three independent measurements are shown. Only errors exceeding 3% of the measured value are displayed.

3.2 | Spectroscopic Study of the Gas Phase

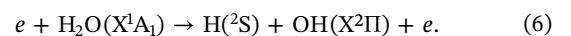
3.2.1 | Broadband Radiation Analysis

Plasma emission recorded in the wavelength range from 200 nm to 875 nm is shown in Figure 5. In the UV part of the spectrum, NO_γ ($A^2\Sigma - X^2\Pi$) molecular band in the wavelength range from 215 to 272 nm [74–76], with a maximal emission at 236 nm, and hydroxyl radical $OH(A^2\Sigma - X^2\Pi)$ at 307 and 310 nm [77, 78] were detected. Additionally, an intensive band corresponding to the transition of electronically excited N_2 molecule in the $C^3\Pi_u$ state to the $B^3\Pi_g$ state (second positive system) was captured [79] with additional emission line of the N_2^+FNS ions ($B^2\Sigma_u^+ \rightarrow X^2\Sigma_g^+$) detected at 357 and 390 nm. Emission lines at 777 nm and 844 nm were assigned to atomic oxygen, corresponding to the 3s–3p transitions [80]. In the 690–950 nm region, the observed lines were attributed to argon excited species corresponding to 4s–4p transitions. The most intense emissions were detected at 751, 763, 811, and 841 nm [81].

The formation of excited argon species in the discharge occurs predominantly through collisions between electrons and ground-state argon atoms. In gas mixtures containing air, molecular nitrogen and oxygen are also subjected to electron impact, leading to their dissociation into atomic nitrogen and oxygen. Additionally, atomic oxygen can be generated through the dissociation of O_2 molecules induced by collisions with metastable argon species [82].

Due to the direct contact between the APSJ and the water surface, water evaporation leads to the presence of additional water vapor in the discharge region. Under these conditions, the formation of OH radicals in the ground state ($X^2\Pi$) can proceed via several reaction pathways involving H_2O molecules. The most relevant reactions include [78, 83–86]:

Electron-impact dissociation of water molecules:



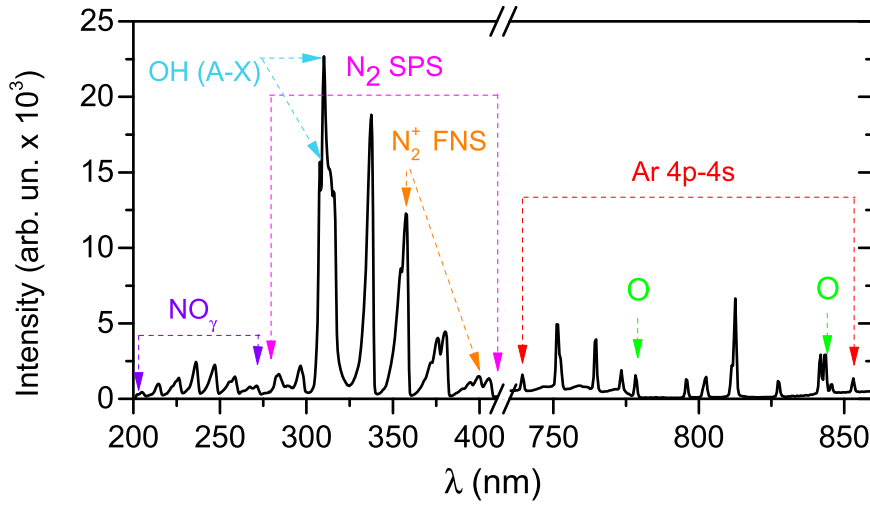
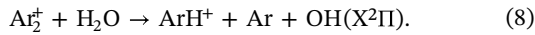
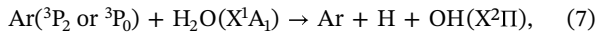
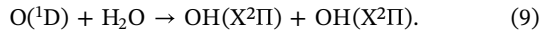


FIGURE 5 | Optical emission spectra of the APSJ at the discharge power $P_{G\_MEAN}(NET)$ of 11.7 ± 0.3 W and Ar flow of 1 slm.

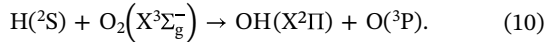
Reaction of metastable argon atoms ($^3P_2$, $^3P_0$) or Ar_2^+ with water:



Reaction of excited atomic oxygen with water:

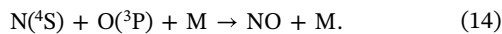
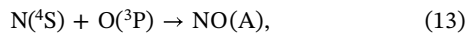
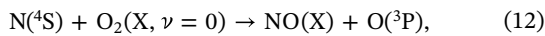
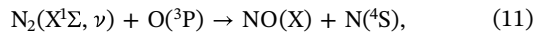


Recombination of a hydrogen atom with molecular oxygen (O_2):



The presence of atomic and molecular nitrogen in the discharge region above the liquid enables the formation of nitric oxide (NO) through several gas-phase reactions with primary oxygen-containing species. These reactions are initiated by electron impact dissociation of nitrogen and oxygen molecules and have been widely reported in non-thermal plasma systems interacting with ambient air or oxygen admixtures [87, 88].

The $NO\gamma$ bands detected in the spectrum (see Figure 5) originate from the $NO(A-X)$ transition. The most relevant reactions leading to the formation of ground-state $NO(X)$ and excited $NO(A)$ include [89–91]:



In reaction 14, which proceeds in the presence of a third body M (N_2 , O_2 , Ar), NO can be formed both in the ground state (X) and in the excited state (A). In addition, the presence of water vapor in the plasma–liquid interaction zone facilitates further NO production [90]:

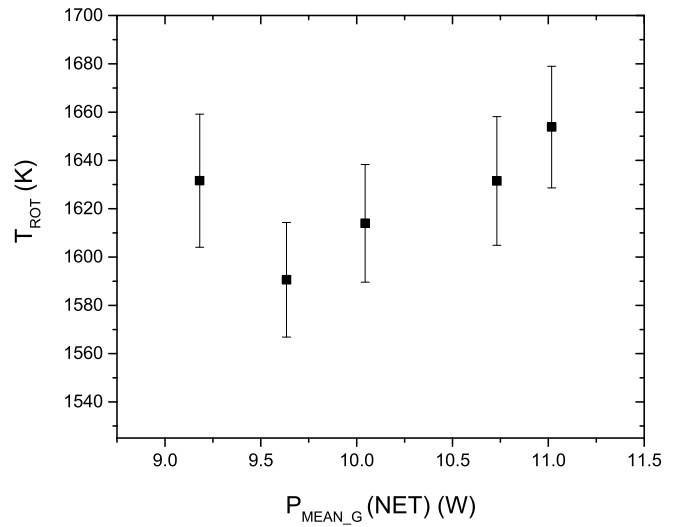
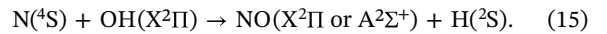


FIGURE 6 | Gas temperature values obtained from the rotational structure of the $OH(A^2\Sigma^+-X^2\Pi)$ emission, averaged over the plasma gap. The error bars represent the uncertainty resulting from the mismatch between the experimentally measured and simulated spectra. Each measurement was repeated three times, and the plotted values correspond to the average of these repetitions.



3.2.2 | Gas Temperature Determination

Figure 6 presents the dependence of the gas temperature on plasma power. The temperature remains nearly constant at 1630 ± 60 K over the investigated power range. Given that the applied power varied only by about 2 W, this behavior indicates that the discharge operates in a stable regime, where the energy partitioning and heat losses remain nearly unchanged. The relatively high temperature reflects intense gas heating, suggesting that a considerable fraction of the input energy is converted into translational energy of the gas along with excitation or chemical processes. Such efficient heating is typical for atmospheric-pressure argon streamer jets interacting with humid environments, where collisional relaxation of excited

argon states and quenching of $\text{OH}(\text{A}^2\Sigma^+)$ by water vapor contribute significantly to thermalization [92]. Since the collected emission represents an integrated signal across the discharge gap, the reported temperature corresponds mainly to the region of highest $\text{OH}(\text{A}^2\Sigma^+)$ density—that is, the plasma core.

3.2.3 | OH Ground State Concentration in the Gas Phase

Figure 7 shows the evolution of the $\text{OH}(\text{A}^2\Sigma^+)$ LIF signal in the middle of the discharge gap above deionized water (water surface at $Y = 0$ mm) with increasing discharge power, $P_{\text{MEAN\_G}}$. LIF signal is strongest at the center of the streamer channel and gradually decreases toward its edges. As the discharge power increases, the streamer channel broadens, resulting in a wider region contributing to the LIF signal. In addition, the fluorescence intensity increases with power, particularly in the central region of the channel.

Figure 8 presents the absolute number density of $\text{OH}(\text{X}^2\Pi)$ radicals in the gas phase as a function of the discharge power. The results show that the $\text{OH}(\text{X}^2\Pi)$ density increases with increasing discharge power. In the range from 9.2 W to 10.0 W, the density rises nearly linearly—from $4.8 \times 10^{20} \text{ m}^{-3}$ to $6.0 \times 10^{20} \text{ m}^{-3}$. At higher powers (10.7–11.0 W), the increase saturates, and the maximum measured value reaches $6.5 \times 10^{20} \text{ m}^{-3}$. These values are in good agreement with those previously reported for atmospheric-pressure argon plasma jets operated in humid or water-containing environments [93–96].

The increase in discharge power enhances the electron density, which in turn promotes OH radical formation via electron-impact dissociation of water molecules [97]. At elevated power levels, however, depletion of water vapor—originating from ambient humidity and evaporation at the plasma–liquid interface—may limit further OH production, leading to the observed saturation.

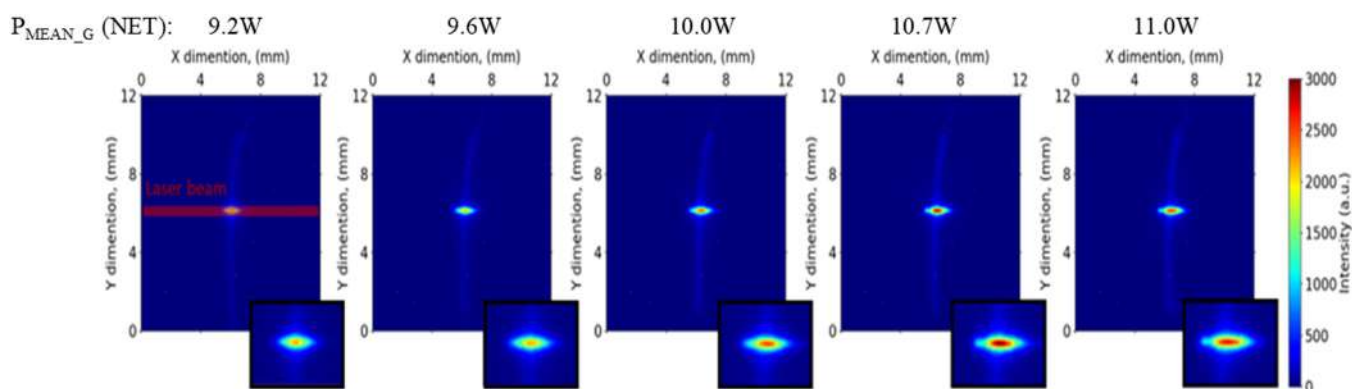


FIGURE 7 | Evolution of $\text{OH}(\text{A})$ fluorescence signal from discharge in Ar (1 slm) above deionized water with increasing the discharge power $P_{\text{MEAN\_G}}$ (NET). The laser is positioned 4 mm from the jet's nozzle.

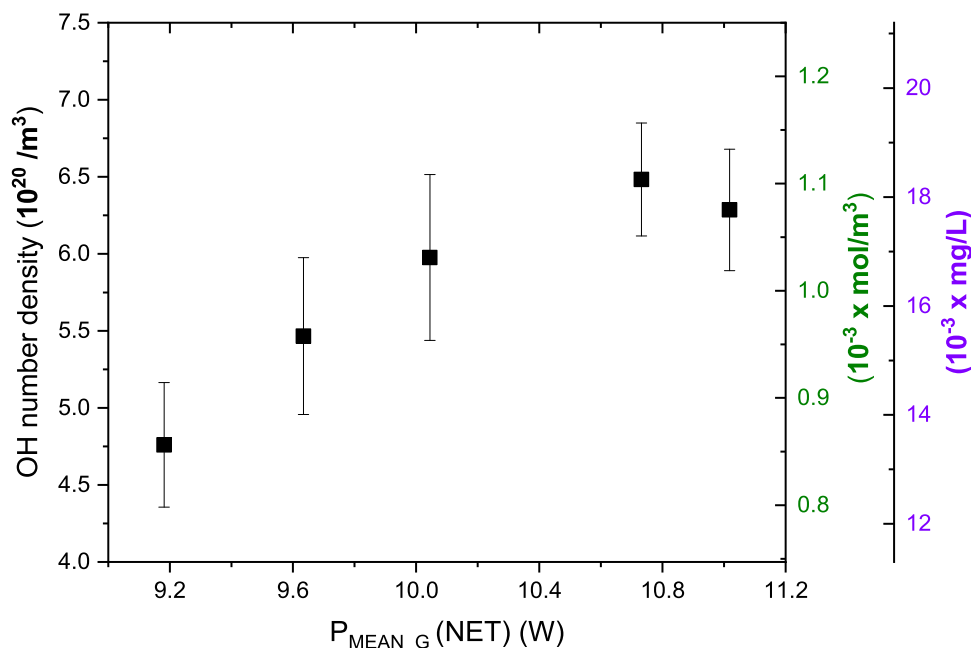


FIGURE 8 | Absolute $\text{OH}(\text{X})$ density in the Ar (1 slm) discharge as a function of $P_{\text{MEAN\_G}}$ (NET), measured 4 mm from the glass tube, with distilled water as the target.

The OH radical is one of the key reactive oxygen species in atmospheric-pressure plasmas and serves as the primary precursor for H_2O_2 formation. Given the high gas temperature estimated in the streamer, H_2O_2 formation in the bulk plasma is unlikely, as it would readily decompose under such thermal conditions. Therefore, the plasma–liquid interface is expected to be the main reaction locus for H_2O_2 generation, where gas-phase OH radicals can recombine in the cooler boundary layer or dissolve into the liquid phase and participate in secondary reactions leading to H_2O_2 formation.

In summary, the emission spectrum of the discharge revealed OH, N_2 , and N_2^+ as the dominant species, with additional detection of NO and O. The reactive species OH, NO, and O are of particular importance, as they participate in reactions leading to the formation of long-lived species in the liquid phase, which will be discussed in the following subsection. Furthermore, OH rotational temperature measurements showed high gas temperature in the active zone of the discharge, leading to fast evaporation of water in the plasma/liquid interface, the process making a strong contribution to the pathways of chemical reaction in the streamer discharge. The presence of water vapor in the gas phase results in the formation of dissociation products, including the OH radicals with a concentration that increases up to 10 W and then saturates at higher powers.

Characterization of the gas phase was an important step towards establishing a link between discharge conditions and obtained liquid-phase reactivity. Both experimental measurement and global modeling were used to further understand the chemical kinetics in the liquid phase.

3.3 | Characterization of PAW

The sample temperature was measured during the treatment by using a thermo-optical probe (see Figure 2) for $P_{\text{MEAN}_G}(\text{NET}) = 10.7 \pm 0.1$ W. Figure 9 presents the temperature

evolution in the sample during a 10-min treatment. In all three positions (7, 10, and 17 mm), the water temperature rapidly increased from an initial 18.5°C to approximately 50°C during the first 3 min of treatment, followed by a slower rise over the next 2 min. Subsequently, it stabilized at approximately 56°C at 7 mm and 10 mm, and at around 52°C at 17 mm, until the end of the treatment.

Although the rotational temperature obtained from the OH spectra could suggest a high gas-phase temperature of the plasma, the measured temperature in the treated water remained below 60°C. The rapid rise during the first 3 min of treatment was consistent with the initial phase of heat transfer due to thermal conduction. After that, the temperature rise slowed down and the water temperature stabilized, suggesting that heat transfer approached equilibrium. Apart from thermal conduction, the heat transfer from the gas to the liquid also occurs, primarily due to evaporation since the gas is not saturated with vapor. Simultaneous heat and mass transfer involves a balance between the energy delivered to the liquid and the rate of water vapor transfer into the gas phase [98].

During the treatment, the sample volume decreased from the initial 4 mL to 3.1 ± 0.1 mL after 5 min and to 2.2 ± 0.1 mL after 10 min, corresponding to approximately 78% and 55% of the initial volume. High temperature increase might occur locally at the point of streamer touching the liquid surface and lead to non-equilibrium evaporation of water, as overall heating was insufficient to explain the observed volume reduction. Gas purging was another effect responsible for evaporation, as continuous argon gas flow (1 slm) directed over the liquid surface facilitated evaporation by enhancing vapor removal. Evaporation of water caused by the Ar flow (in the absence of discharge) reduced the sample volume by 0.5% and 1.8% after 5 and 10 min, respectively.

A series of treatments of distilled water was performed to evaluate the efficiency of RONS production under varying

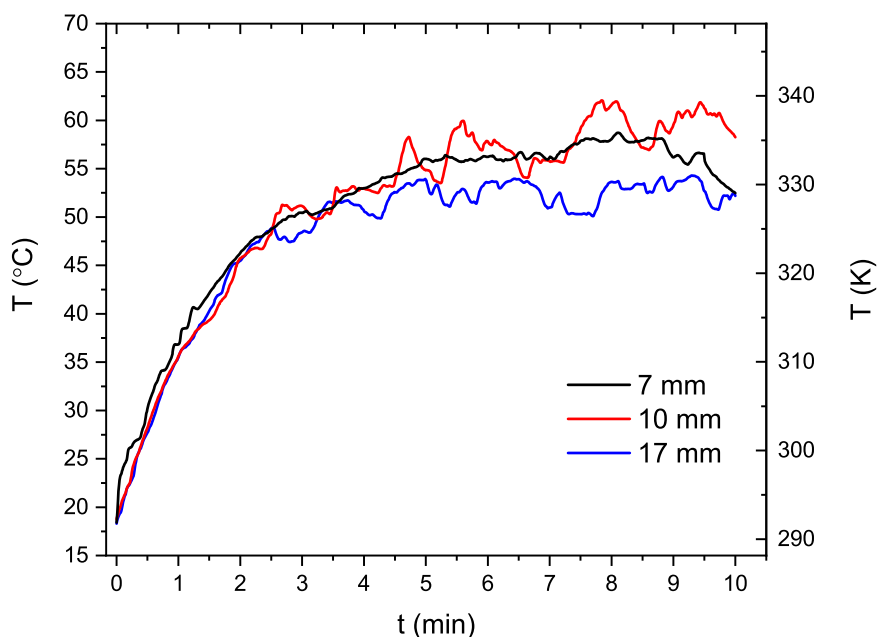
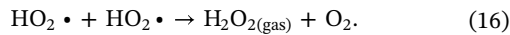


FIGURE 9 | Water sample temperature measured during treatments of 10 min, at three different positions (at 7 mm, 10 mm, and 17 mm) from pin jet and at $P_{\text{MEAN}_G}(\text{NET}) = 10.7 \pm 0.1$ W.

experimental conditions. The experiments were carried out at three discharge powers (10.0, 10.7, and 11.7 W) and two treatment durations (5 min and 10 min), with an initial sample volume of 4 mL. In all cases, acidification of the treated water was observed, with the pH decreasing from the initial value of 6.4 ± 0.2 to 2.9 ± 0.1 after 5 min and to 2.4 ± 0.1 after 10 min of treatment, regardless of the applied discharge power.

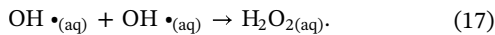
Figure 10 presents the measured concentrations of H_2O_2 and NO_3^- in PAW. The concentration of NO_2^- was also measured, but it was below the detection limit, as discussed later in this section. H_2O_2 concentrations (Figure 10a) increased with longer treatment, rising from 3.3 to 3.7 mol/m³ after 5 min to 4.5–5.3 mol/m³ after 10 min (Figure 10a). This trend has been previously observed and attributed to the formation of OH reactive species during plasma treatment [45, 99].

Hydrogen peroxide detected in PAW may originate from two pathways. It can be generated in both gas and liquid phases. In gas phase, H_2O_2 can be formed through reactions of hydroperoxide radicals $\text{HO}_2 \cdot$:



These radicals are created in gas phase in reaction $\text{H} + \text{O}_2 + \text{M} \rightarrow \text{HO}_2 \cdot + \text{M}$, in the presence of a third body M, such as Ar, N₂, O₂, H₂, H₂O [100, 101].

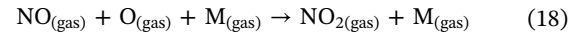
Hydrogen peroxide formed in gas phase can diffuse into the liquid, contributing to its concentration in PAW. In addition, H_2O_2 can be generated directly in liquid phase through the recombination of OH radicals [98]:



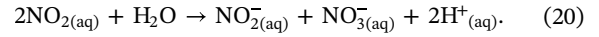
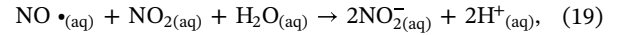
In addition to hydrogen peroxide, the concentrations of nitrate ions (NO_3^-) in PAW were also determined. Measured NO_3^- concentrations were within the range of 1.0 mol/m³ and 2.7 mol/m³ (see Figure 10b).

Nitric oxide (NO) and other reactive nitrogen species (RNS) formed in the gas phase can dissolve into the liquid and undergo subsequent reactions, leading to acidification of the liquid

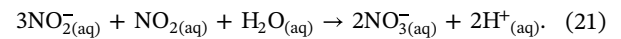
phase and nitrate accumulation [102, 103]. These processes include the oxidation of NO to NO_2 in gas phase:



followed by the hydrolysis reactions:



At the initial stage of plasma treatment, both NO_2^- and NO_3^- are expected to form, as indicated by reaction (20). However, as experimentally observed, the progressive acidification of the solution ($\text{pH} < 3.5$) promotes the rapid oxidation of NO_2^- into NO_3^- , leading to its complete conversion and non-detection in the final PAW composition, as represented by reaction (21) [90]:



Both longer treatment time and higher discharge power contributed to increased concentration of measured species. Extending the treatment time from 5 to 10 min resulted in approximately a 20%–45% increase in H_2O_2 and a 95%–125% increase in NO_3^- concentrations for all three power levels. In contrast, after increasing the discharge power from 10.0 to 11.7 W, the concentrations of H_2O_2 and NO_3^- in the liquid phase remained comparable on the order of 10%–20%. This behavior was consistent with the gas-phase OH measurements, which showed a power-dependent increase (up to 10.7 W) and then saturated (see Figure 8). This was further investigated with one-dimensional modeling to better understand the underlying chemical pathways leading to the formation of long-lived species in the liquid phase.

3.4 | Liquid Chemistry Modeling

As previously indicated, the concentration of gas-phase OH radicals between the plasma device and the liquid surface has

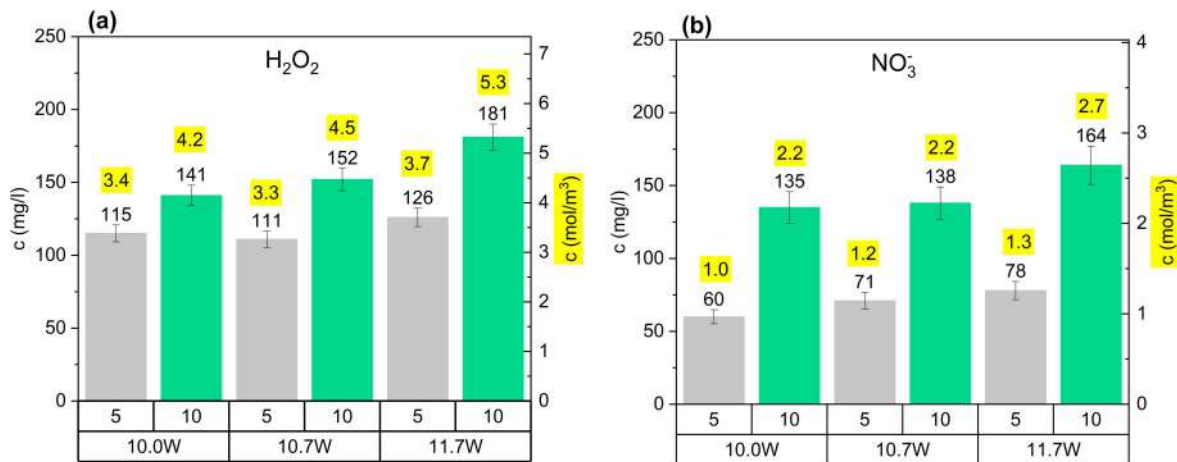
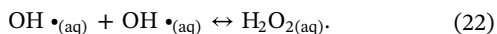


FIGURE 10 | Measured concentrations of (a) H_2O_2 and (b) NO_3^- in distilled water (initial volume: 4 mL) treated with Ar (1 slm) plasma, at two treatment durations (5 and 10 min) and three powers P_{MEAN_G} (NET) (10.0 ± 0.2 W, 10.7 ± 0.1 W, and 11.7 ± 0.3 W). All concentrations are normalized to the final volume of the sample. Bars are labeled with concentrations in mg/L and mol/m³ (yellow). Error bars represent the standard error of the mean.

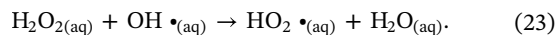
been experimentally measured. Furthermore, as already mentioned, since the plasma device operates in air, it is anticipated that RNS are also generated in the gas phase. Here, we considered the case where, in addition to the measured OH radicals, typical RNS species such as NO, NO₂, and NO₃ are irradiated onto the liquid surface. We investigated the behavior of liquid-phase chemical reactions induced as these RONS species enter as triggered and diffused into the depths of the liquid. A one-dimensional model directed toward the liquid interior is one of the most suitable models for understanding reaction-diffusion behavior from the gas-liquid interface into the liquid. Below, we presented numerical analysis results focusing on the behavior of H₂O₂ and NO₃⁻, that is, experimentally observed chemical species, at a depth of 1 μm near the gas-liquid interface and at a depth of 1 mm, considered to be deep within the liquid.

Figure 11 shows the simulated concentrations of various hydrogen-oxygen species ((a) and (b)) and hydrogen-oxygen-nitrogen species ((c) and (d)) at depths of 1 μm ((a) and (c)) and 1 mm ((b) and (d)). The simulation modeled 1.5 min of active chemical behavior during the RONS impact time, followed by 15 min of passive chemical behavior.

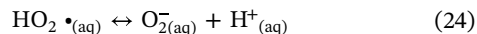
As shown in Figure 11a, highly reactive and thus short-lived OH radicals rapidly dissipate within a depth of 1 μm near the gas-liquid interface via a reversible reaction (the reversible form of Equation (17)), which is one of the main pathways:



The disappearance of OH radicals effectively activates chemical reactions in the liquid, generating H₂O₂. As H₂O₂ is rapidly produced, OH is consumed in its reaction with H₂O₂, generating hydroperoxyl radicals HO₂• through the following reaction:

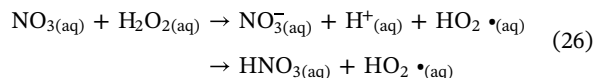
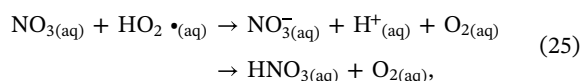


Simultaneously, the reversible reaction



also proceeds, resulting in the generation of superoxide anion O₂⁻. As evident from the behavior of H⁺ and OH⁻, the inflow of RONS acidifies the water, causing the pH to change from 7.0 to approximately 4.5. A decrease in pH is observed not only near the gas-liquid interface but also extending to a depth of 1 mm. This can be understood because, as shown in Figure 11b, long-lived H₂O₂ permeates into the bulk region while being consumed, thereby causing changes in the concentrations of H⁺, OH⁻, and HO₂.

Figure 11c,d indicate the significant presence of nitrate ions and nitrate NO₃⁻/HNO₃ in the bulk region from the gas-liquid interface. This is due to reactions such as:



as well as the reactions (19) and (20). The concentration of nitrite ions NO₂⁻ is lower than that of nitrate ions NO₃⁻. This is due to the following reaction:

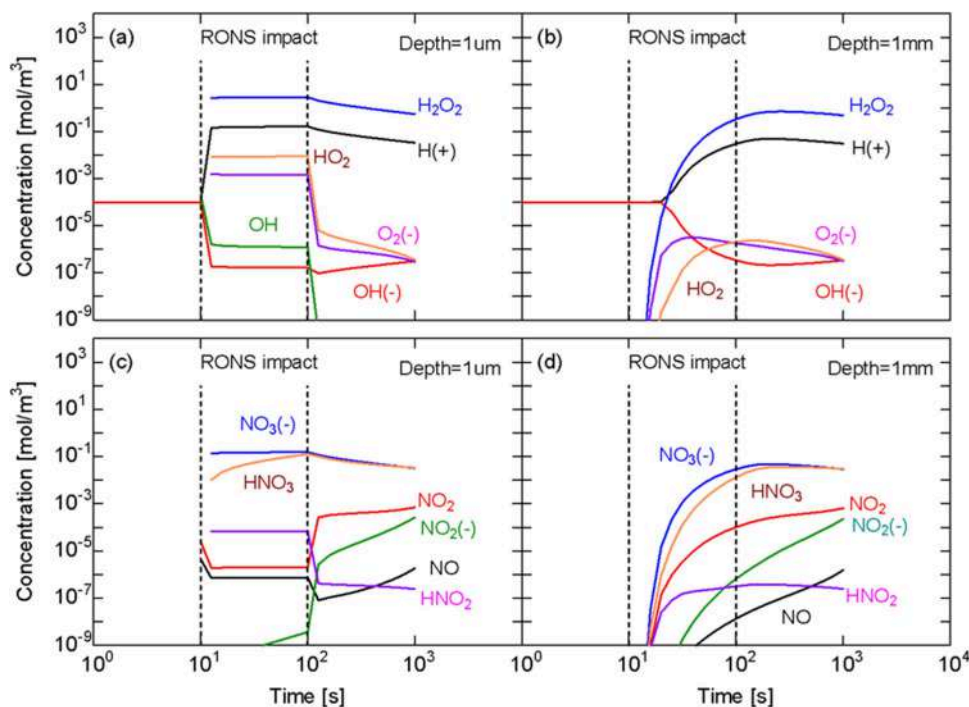
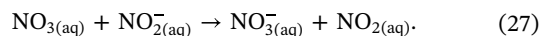


FIGURE 11 | RONS impact (1.5 min) + 15 min. Hydrogen-oxygen species: (a) depth = 1 μm, (b) depth = 1 mm. Hydrogen-oxygen-nitrogen species: (c) depth = 1 μm, (d) depth = 1 mm.

It is not easy to reproduce the absolute values of H_2O_2 and NO_3^- concentrations obtained experimentally (Figure 10) using this one-dimensional model. However, numerical simulations explain the mechanism by which measured OH radicals and RNS, expected to be generated by the plasma, increase the concentrations of H_2O_2 and NO_3^- in the liquid. Furthermore, it captures the trend where H_2O_2 concentrations are highest, NO_3^- concentrations are lower than that, and NO_2^- concentrations are even lower.

3.5 | PAW Toxicity Test

The phytotoxicity of PAW was evaluated on Common duckweed (*Lemna minor*) as a model plant. To mimic realistic environmental scenarios, PAW was tested at various dilutions (10%, 30%, 60%), as the lower concentrations were expected upon release of PAW into natural aquatic systems. TW was also included as a transitional model toward more realistic conditions relevant for agricultural applications. Produced PAW was diluted with TW and river water (RW)—the latter was used for dilution to provide a more realistic test matrix. Furthermore, the treated water volume was increased from 4 to 15 mL to maximize the obtained PAW volume while still generating appropriate concentrations of RONS to induce observable biological effects. The treatment parameters were as follows: $V_{\text{RMS}} = 1.2$ kV, $I_{\text{RMS\_G}} = 10.5$ mA, $P_{\text{MEAN\_G}} = 10$ W, and the properties of PAW are shown in Table 1.

The pH of the PAW obtained from TW remained unchanged after plasma treatment due to the buffering capacity of carbonic compounds present in TW. Carbonates and bicarbonates act as buffers during plasma treatment and limit pH reduction [66, 104–106]. Electrical conductivity decreased by ~20%, from 583 to 457 $\mu\text{S}/\text{cm}$. Similar short-term reductions have been

reported for DBD-treated water [64], followed by an increase with prolonged treatment. This variation is attributed to concurrent changes in concentrations of long-lived ionic species—most notably, an increase in nitrate levels and a decrease in (bi) carbonate content due to localized heating and CO_2 release [107]. In contrast to distilled water, in PAW produced from TW, we obtained comparable concentrations of NO_3^- and NO_2^- . In this case, since no change in pH occurred, NO_2^- was not subsequently converted to NO_3^- .

Additionally, the appearance of reactive chlorine species (RCS), such as hypochlorous acid (HOCl) and hypochlorite ions (OCl^-), has been reported in PAW generated from tap water that induced bactericidal effects under certain conditions [66, 108, 109]. As the tap water in our experiments is potable, the maximum permissible concentration of residual chlorine is 0.5 mg/L [110, 111]. With the concentration of residual chlorine well below the legal limit and pH measured around 8, the conditions for HOCl formation were not favorable in PAW. Therefore, a possible toxicity effect of RCS present in the PAW on plants is negligible.

We divided our results from experiments with *L. minor* into two groups. The first group refers to morphological changes in *L. minor* (total frond area and root length), while the second group covers physiological monitoring of chlorophyll content (chlorophyll a, chlorophyll b, and total) under different combinations of PAW, TW, and RW. Figure 12a shows the mean frond surface area per plant, S_{frond} , for the different cultivation matrices. We observed an increase in frond area in both TW- and RW-based mixtures with PAW. In TW dilutions, the maximum was reached at 10% PAW, with ~50% larger area compared to the TW control, while higher concentrations ($\geq 60\%$) reduced growth. In RW dilutions, the peak occurred at 30% PAW, after which further increases in PAW content slowed

TABLE 1 | Physicochemical properties of TW and PAW: V – sample volume, σ – electrical conductivity, c – concentration.

Sample	V [mL]	pH	σ [$\mu\text{S}/\text{cm}$] _i	c(H_2O_2) [mg/L]	c(NO_3^-) [mg/L]	c(NO_2^-) [mg/L]
TW	15.0	7.8 ± 0.1	583 ± 30	0	0	0
PAW	13.5 ± 0.1	8.1 ± 0.2	457 ± 30	36 ± 1	39 ± 1	29 ± 1

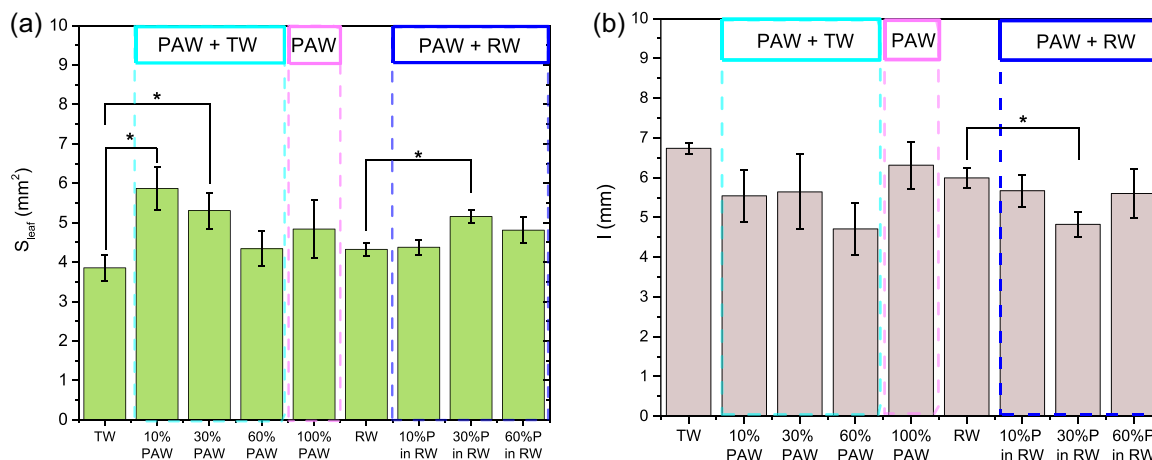


FIGURE 12 | Effect of plasma-activated water (PAW) and its dilutions in TW and RW on (a) S_{frond} - mean frond surface area and (b) l_{root} - mean root length of *L. minor* after 17 days of cultivation, for different PAW concentrations (10%–100%) and control samples (TW, RW). Error bars represent the standard error of the mean. * indicates a statistically significant difference compared to the corresponding control ($p < 0.05$, two-sample t-test).

growth. In addition, the root length (l_{root}) of this plant (see Figure 12b) in both experimental setups shows different patterns. In TW-based samples, root elongation generally decreased with increasing PAW content, with a slight increase in elongation observed at 30% PAW. In the mixture of PAW and the RW elongation was decreasing with the increase of added PAW to the samples.

According to the Standard Student t -test analysis, the increase in frond surface area observed in TW dilutions was statistically significant at 10% PAW and 30% PAW compared to the TW control (Figure 12a). In RW-based mixtures, the t -test confirmed a significant difference at 30% PAW relative to the RW control. For root length (Figure 12b), a statistically significant reduction was detected between RW and 30% PAW in RW samples.

Our results in root elongation of *L. minor* correlate with the investigation of zinc oxide nanoparticle effects on the same plant, with root elongation increasing with the concentration of zinc oxide nanoparticle from 0.3 to 1 mg/L and decreasing following higher concentrations of this nanoparticle [112]. A study performed by Xue and colleagues [113] researching the effects of Cadmium and its forms on *L. minor* pointed out that the root elongation and frond number are decreasing following the increase of Cadmium solution in the medium. Another study investigating the effects of fluorides on *L. minor* [114] observed a gradual decrease in frond area with the increase of fluoride (2, 4, 6 mg/L). Similar effects on the frond area were observed following the effects of zinc selenide nanoparticles on *L. minor*, with the frond size gradually decreasing with the increase of concentration of this nanoparticle from 1 to 80 mg/L, with the highest frond area observed when treating the plant with the concentration of 5 mg/L [115].

Figure 13 shows the concentration of chlorophyll a, chlorophyll b, and total chlorophyll ($C_{\text{total}} = C_a + C_b$) content in plants after PAW treatment. For clarity, the 10%, 30%, and 60% PAW labels refer to dilutions prepared with both TW and RW. For TW-based samples, chlorophyll content increased at 10% and 30% PAW, then declined at higher PAW concentrations. A similar trend was observed for RW-based samples, though overall chlorophyll levels were higher compared to TW-based media.

The decrease in chlorophyll content for PAW concentrations above 30% is much more pronounced than the reduction in frond area. Chlorophyll content provides a more accurate indication of the quality of the leaves and the overall plant condition than frond area measurements alone. However, both parameters indicate that lower PAW concentrations in water are not only non-toxic but also promote plant improvement, with PAW-derived nitrogen species acting as a fertilizer in this case.

Monitoring the chlorophyll content in *L. minor* in both combinations of water with PAW, we observed an increase in chlorophyll content with the maximum at 30% PAW and gradually decreasing with the PAW concentration increasing (see Figure 13). In the study performed by Chen and colleagues [112], they recorded the decrease in chlorophyll content gradually with an increase in concentration of zinc oxide nanoparticles applied to *L. minor* in concentrations of 0.03, 0.3, and 3 mg/L. Similar results regarding chlorophyll a were reported in a study investigating the effects of fluorides on *L. minor*, with the absorbance at 433 and 665 nm decreasing with increased concentration of fluorine with selected concentrations of 2.10, 4.19, and 4.90 mg/L [114]. Another case study regarding the effects of zinc selenide nanoparticles on *L. minor* also observed a gradual decrease in chlorophyll content with increased

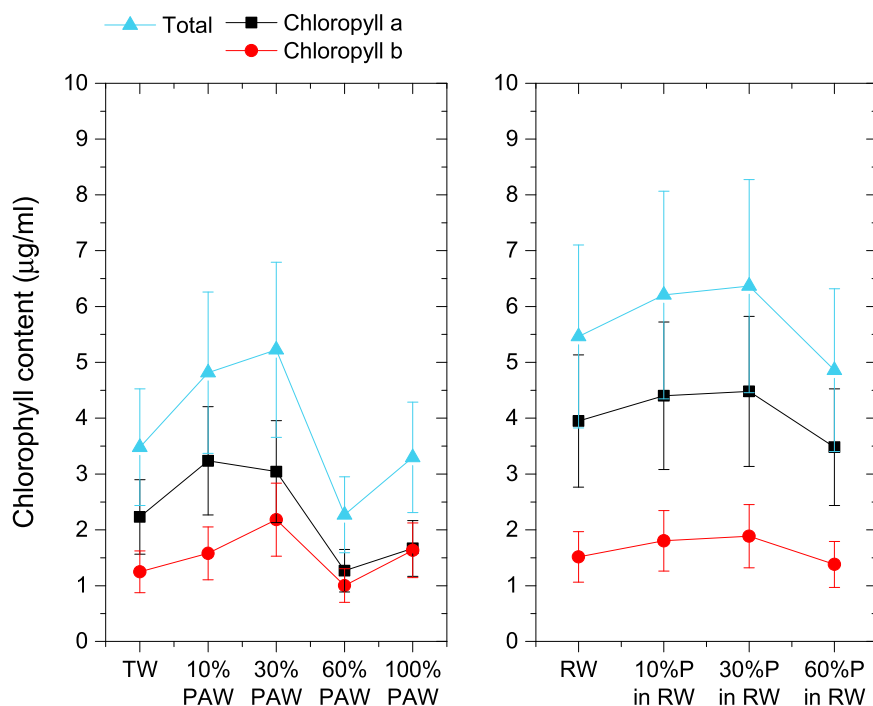


FIGURE 13 | Chlorophyll content (chlorophyll a, chlorophyll b, and total chlorophyll) measured in plants treated with different liquid samples. Treatments included PAW and PAW dilutions (10%–60%) prepared in two aqueous matrices: TW and RW. Control samples were untreated TW and RW. The uncertainty of chlorophyll determination was estimated to be approximately 30%.

concentration of this nanoparticle [115], and similar results were found while investigating the effects of herbicide fluoro-chloridone on *L. minor* [103].

Chlorophyll content is regarded as an indicator of Photosystem II protein activity in plants exposed directly or indirectly to some stress [116]. The increase in chlorophyll content in our study could be explained in two ways. First, by the formation of shade-type chloroplasts, which have some ultrastructural modifications to accommodate more pigments with an increase in pigment content in *L. minor* exposed to PAW. On the other hand, the higher increase in chlorophyll content at lower concentrations of PAW could be related to hermetic responses, where the plants exposed to low stress conditions increase growth and enzyme activity. Such hormesis was previously observed in *L. minor* treated with different concentrations of various pesticides [117].

Approaches used for toxicity experiments here were in line with the methods previously used for the investigation of different potentially toxic chemicals. In that sense, this allowed comparison of PAW influence with the studies where the effects were thoroughly investigated at the biochemical level, and also to link to the mechanisms induced in plants. Our results on toxicity testing showed that, in general, PAW influence on *L. minor* could be both positive and negative, depending on the concentration of PAW and also on other water impurities. Thus, a larger range of PAW properties should be tested in order to fully understand the effects of reactive species from PAW to plant physiological mechanisms. Furthermore, the observed dependence on both the liquid matrix and PAW concentration was obtained using a standard plant toxicity model. This indicates that PAW safety cannot be generalized from a single application or exposure route but should be evaluated with respect to its broader environmental impact, particularly after its release into natural water systems.

4 | Conclusion

In this study, we performed experimental measurements together with modeling to study the main processes responsible for the generation of PAW by using APSJ that operates with the addition of Ar flow in ambient air. We used electrical diagnostics, optical emission spectrometry, and LIF techniques to study the discharge in the gas phase above water sample. *V-I* and power measurements showed two distinctive regimes of operation, with streamer regime powers around 10 W that were found to be optimal for stable operation and water treatment. Optical emission spectrum was typical for discharges working in Ar-air mixtures. Rotational temperatures obtained from OES were in the order of 1600 K and were similar to the temperatures obtained from the literature for the streamer type of discharge. The concentration of OH radicals had a weak dependence on the power delivered to the discharge and was in range from 4.8×10^{20} to $6.5 \times 10^{20} \text{ m}^{-3}$.

To determine the chemistry produced in the liquid phase we measured long-lived species formed in PAW (H_2O_2 , NO_2^- , and NO_3^-) employing colorimetric techniques and used a one-dimensional gas-liquid model with experimentally determined OH concentrations in the gas phase as input data. The model helps in assessing the most important reactions in the liquid phase responsible for the formation of long-lived species in

PAW. Comparing experimental RONS measurements and model results after 10 min showed agreement in the trend for concentrations of H_2O_2 and NO_3^- . Despite the streamer's high temperatures determined from LIF temperatures of water measured during the treatment in bulk liquid never exceeded 60°C, indicating that thermal effects are localized to the narrow region of plasma-liquid interaction. The treatments led to pronounced acidification of the samples and the formation of H_2O_2 and NO_3^- , while NO_2^- was not detected. The concentrations of the long-lived reactive species increased with treatment time, whereas variations in discharge power had no significant influence on their levels.

Finally, potential phytotoxic effects of PAW were evaluated on Common Duckweed (*Lemna minor*) as a model plant system. We made dilutions of PAW by using tap and Danube river water to test an environment closer to realistic. We showed both positive and negative morphological changes in leaves, roots, and chlorophyll concentrations with respect to control samples. The most adverse effects to *L. minor* were found for 40% of dilution (60% of PAW). Our results, gathered from the *L. minor* experiment, are valuable since they provide rare data combining the effects of PAW, the selected aquatic macrophyte, and could be used in similar experiments regarding Danube river water pollution or *L. minor*'s response to PAW treatment in combination with different aquatic pollutants in the future.

Author Contributions

Olivera Jovanović, Nevena Puač, Nikola Škoro, and Anton Nikiforov: conceptualization. **Olivera Jovanović, Nevena Puač, Nikola Škoro, Tomoyuki Murakami, Arian Morina, Mikhail Gromov, and Anton Nikiforov:** methodology. **Olivera Jovanović, Mikhail Gromov, Anđelija Marković, and Tomoyuki Murakami:** investigation. **Olivera Jovanović, Nevena Puač, and Nikola Škoro:** writing – original draft. **Nevena Puač, Nikola Škoro, Tomoyuki Murakami, Arian Morina, Mikhail Gromov, and Anton Nikiforov:** writing – review and editing. **Anton Nikiforov, Nikola Škoro and Nevena Puač:** supervision. **Nevena Puač:** funding acquisition.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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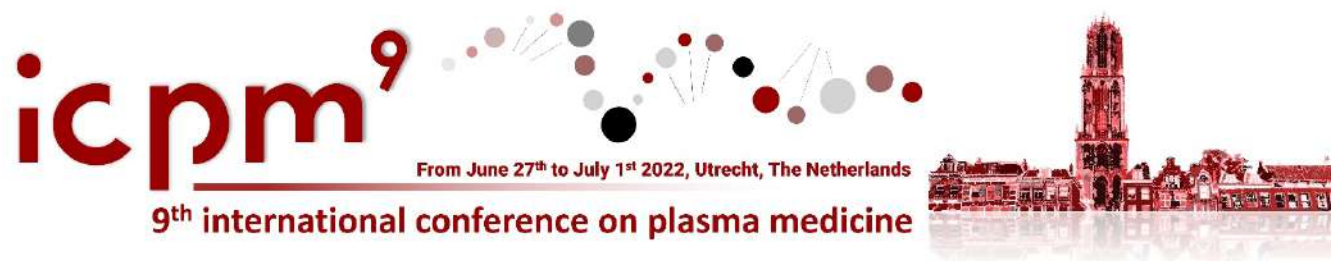
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Plasma pin-jet for treatment of water: production of reactive species in distilled and tap water

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In two increasingly important fields of plasma application – biomedicine and plasma agriculture, Plasma Activated Liquids (PALs) play a key role [1,2]. These applications caused the development of various designs of atmospheric pressure plasma jets (APPJs) that operate in contact with liquid samples. Despite a significant number of successful investigations involving the use of PALs [3], there are still lot of unknowns that need to be clarified. The first is to completely understand the interaction mechanisms between plasma and liquid, and the creation of a huge number of chemical species. The second, equally challenging problem, is how to standardize a device's performance to produce PAL of required physico-chemical characteristics for different applications.

In this work, we will present the results of the treatment of pure distilled and tap water in order to create Plasma Activated Water (PAW). We have used an atmospheric pressure plasma jet (APPJ) in pin electrode configuration operating at a frequency of 340kHz in contact with aqueous sample. The plasma jet was used with pure argon and an admixture with synthetic air as working gas. Total gas flow was kept constant at 1 slm in all experiments. The distilled water and tap water samples were exposed to the streamer discharge under several treatment conditions in order to investigate the influence of plasma parameters and the target properties on characteristics of the produced PAW. In order to determine the excited species produced in plasma above the liquid, optical emission spectroscopy was employed. Diagnostics of liquid samples were performed to assess the effectiveness of plasma treatment. The analysis of physicochemical parameters was conducted by measuring conductivity, pH, temperature and the amount of deposited RONS (H_2O_2 , NO_3^- , NO_2^-).

The results of optical emission spectroscopy showed the existence of oxygen and nitrogen reactive species in the spectrum of argon discharge. Besides the dominant OH radical band, NO γ emission was also detected. Measurements of RONS created in plasma and deposited in aqueous samples showed that the sample properties, treatment time, and working gas were parameters that influenced both the type and the amount of produced reactive species.

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OP-8

LIQUID TREATMENTS BY ATMOSPHERIC PRESSURE PLASMA PIN-JET

O. Jovanović, N. Puač, N. Škoro

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Numerous designs of atmospheric pressure plasma jets (APPJs) that function in contact with liquid samples have been developed in the last decade. When aqueous solutions are exposed to non-thermal plasma, reactive oxygen and nitrogen species (RONS), such as OH, O, O₃, H₂O₂, NO₂⁻, NO₃⁻, formed in several reactions in the gaseous phase are introduced into the aqueous phase. These reactive species are physiologically significant chemicals that are involved in metabolic activities of the plant cells. Consequently, plasma-treated water (PTW) can be used as a complex chemical reactant in the treatment of numerous biological systems.

A plasma system was investigated with an APPJ with sharpened-end powered electrode that generates a streamer discharge above the liquid sample. The plasma source was powered by a continuous kHz signal. As working gases, we used He, Ar, and Ar/air admixtures. We conducted electrical characterization and discharge power measurements to acquire additional information about the treatment stability and plasma properties. Spectrally resolved imaging and optical emission spectroscopy were used to determine the type and the spatial distribution of the excited species created in the plasma-liquid interaction. In order to create PTW, liquid samples placed below the APPJ were treated for different duration times and in different sample volumes. The device was used for treating both clean and contaminated water. Detailed physico-chemical analysis of the treated liquid samples, including reactive oxygen and nitrogen species detection, pH, temperature, and electrical conductivity measurements, was performed after treatments. The treated samples were further used in experiments with biological materials.

The results showed significant differences in the properties of Ar and He plasma and enabled us to link the effects of the treatments with the liquid samples properties. Hence, we were able to produce PTW with particular properties suitable for different applications in the fields of biotechnology and plasma agriculture.

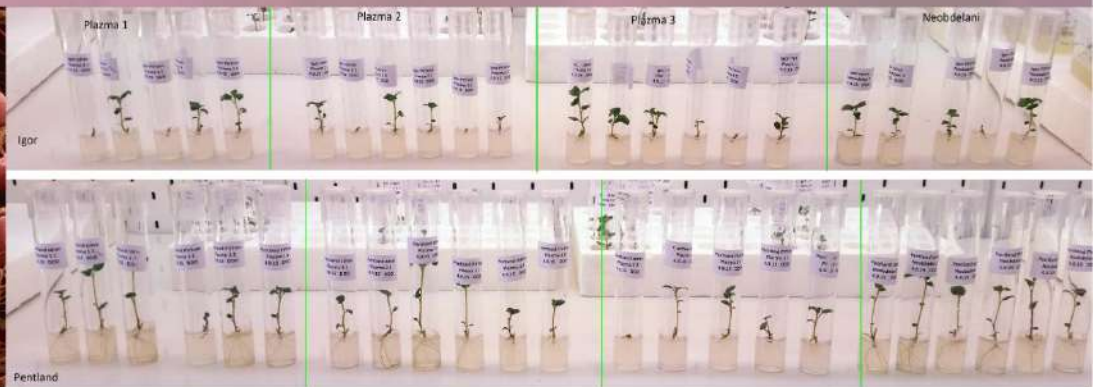
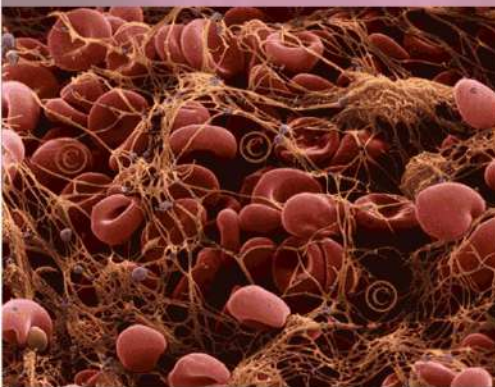
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8th International Conference on Advanced Plasma Technologies 3rd Workshop on Plasma Applications for Smart and Sustainable Agriculture



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Session chair: Monica Magureanu	
16:00	Tom Field (UK); A method to control the energy of free electrons in atmospheric pressure plasma discharges; STSM
16:15	Thomas Vazquez (Slovakia); Real scale treatment of indoor air by cold atmospheric plasma and photocatalysis; Oral
16:30	Mia Ivanov (Croatia); Degradability improvement of the olive mill wastewater by high voltage electrical discharge plasma (HVED); Oral
16:45	Domenico Aceto (Italy); The effect of PAW on the growth and gene activation of healthy and tomato-mottle-mosaic-virus inoculated tomato seedlings; Oral
17:00	Olivera Jovanović (Serbia); Argon plasma pin-type jet for water treatment and biological applications; Oral
17:15–17:30	Zdenko Machala (Slovakia); Future agriculture with green fertilizers produced by non-thermal plasma-activated water technology; Oral
18:30	Dinner break
20:00	PLAgri networking and discussions / Poster session

Tuesday, 16th May

Session chair: Zdenko Machala	
9:00	Tomoyuki Murakami (Japan); Numerical simulation and chemical network analysis of plasma-treated water; Invited
9:30	Anna Dzimitrowicz (Poland); Plasma-based technologies for the efficient and economic removal of pharmaceuticals from contaminated water; Invited
10:00	Vit Jirašek (Czech Republic); Direct and imprinted chemical reactivity of plasma-treated solutions; Invited
10:30	Coffee break
Session chair: Joanna Pawlat	
11:00	Živko Čurčić (Serbia); Plasma in agriculture from agronomist perspective; Invited
11:30	Liutauras Marcinauskas (Lithuania); Extraction of valuable compounds from microalgae using plasma and pulsed electric field treatment; Oral
11:45	Mark Zver (Slovenia); Low-pressure plasma irradiation for water disinfection; Oral
12:00	Lunch break
Session chair: Tomislava Vukušić Pavičić	
14:00	Changtao Chen (Belgium); Degradation of micropollutants in secondary wastewater effluent using nonthermal plasma-based AOPs; Oral

Argon plasma pin-type jet for water treatment and biological applications

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A non-thermal Ar plasma jet operated in the kHz regime was used to generate reactive oxygen and nitrogen species (RONS) in water and produce plasma-activated water (PAW). We have developed a plasma jet in pin electrode configuration that generates a streamer discharge. The distance between the powered electrode and water placed in a vessel below the plasma device was 10 mm. As working gases, we used argon and an admixture of argon and synthetic air (3%). Total gas flow was kept at 1 slm. During treatments of 5, 10, and 20 minutes, 15 ml of distilled water was treated. In order to identify and determine the spatial distribution of excited reactive species produced by plasma-liquid interaction, we used spectrally resolved imaging and optical emission spectroscopy. To examine the efficacy of plasma treatment, liquid diagnostics, including electrical conductivity, pH, and RONS measurements, were carried out immediately after treatments. Concentrations of nitrate and hydrogen peroxide were determined photometrically by using a spectrophotometer. The findings of the optical characterization and measurement of the physicochemical properties of PAW indicated a correlation between RONS formation in the gas phase and in treated water. The obtained results demonstrated that the quantities of measured reactive species in water may be adjusted for the specific application in biotechnology by varying the treatment parameters and employing a single plasma jet.



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INFLUENCE OF PLASMA PROPERTIES ON REACTIVE SPECIES IN PAW

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We have used a pin-type of atmospheric pressure plasma jet (APPJ) for treatment of distilled water samples and production of plasma activated water (PAW). Electrical characterization of the APPJ and plasma power measurements were performed in order to obtain information about stability of the treatment conditions, the plasma properties, and the influence of plasma parameters on PAW. In order to investigate the influence of working gas on the PAW chemistry, we have performed treatments with helium or argon streamer discharges. Diagnostics of treated liquid samples were carried out to evaluate the effectiveness of plasma treatment. Results show that chemically reactive gaseous environment of helium discharge favours the production of nitrates and nitrites. At the same time, the argon discharge produces an order of magnitude higher values of hydrogen peroxide and nitrates, but the amount of nitrites is quite low.

1. Introduction

Cold atmospheric pressure plasmas have been intensively investigated over the past decade due to their great potential for various applications. In the area of plasma agriculture, gaseous plasma treatment of aqueous solutions has multiple advantages such as reduction or elimination of organic contaminants and antimicrobial effects [1, 2]. The exposure of water to plasma induces a number of reactions occurred in the gaseous phase and introduces reactive oxygen and nitrogen species (RONS) species in aqueous phases. The resulting “plasma-activated water” was shown to remain active long after the plasma is turned off [3]. Among the most commonly detected chemical species in PAW are hydrogen peroxide, nitrite and nitrate due to their relative stability. These reactive species play a key role in the reactions involving dissolved organic species in water and can effectively inactivate bacteria or microorganisms [4]. Proper and detailed diagnostics of plasma sources will help to achieve better understanding and establish the correlation between the plasma processes and the treatment effects which is of crucial importance for further applications. From the point of view of plasma physics, the central issue is to standardize the performance in RONS production. Here we will present the results of the influence of the type of the working gas used (He or Ar) on the production of RONS in PAW.

2. Experimental setup

In the study, we assessed the performance characteristics of the pin-type configuration of an APPJ powered by a continuous high voltage signal at frequency 330 kHz and operated with He and Ar as working gases. Schematics of APPJ and experiment set up are provided in Fig.1. Determination of concentrations of three reactive species (H_2O_2 , NO_2^- , NO_3^-) in liquid and pH of treated aqueous solution was performed. Plasma source consists of a metal cylindrical case, a glass tube with concentrically placed powered sharpened electrode. The copper tape at the bottom of the microtiter plate was connected to the ground through a 1 k Ω resistance for monitoring the discharge current flowing through the plasma. The electrical volt–ampere characteristics as well as the power consumption were analysed with electrical probes.

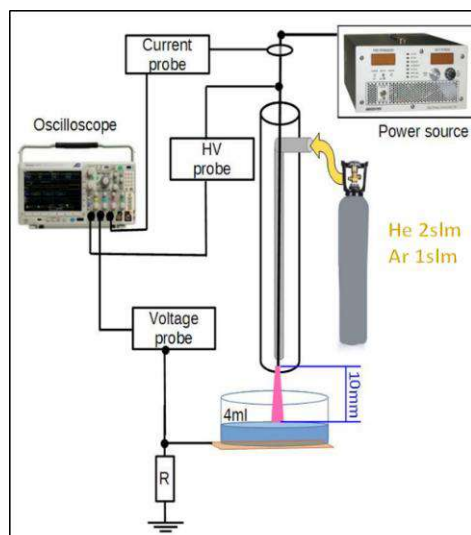


Fig. 1. Schematic overview of the experimental set up.

Two types of plasma treatments were done: with He as working gas with flow rate of 2 slm and Ar with flow rate of 1 slm. Treatment times were 5 and 10 minutes. The volume of treated samples placed in the wells of 6-well microtiter plate below the APPJ was 4ml. The distance between the water surface and wire was 10 mm in all treatments. In order to characterize PAW we used colorimetric methods for measuring the concentrations.

3. Results and Discussion

In Fig. 2. we show concentrations of long-lived reactive species obtained after 5 and 10 minutes treatment of water sample by a pin-electrode jet. We measured completely different concentrations of all reactive species depending on the working gas. By changing the working gas, concentrations of produced H_2O_2 and NO_3^- in PAW are measured to differ by an order of magnitude. For both gases the results show an increase in concentration of H_2O_2 and NO_3^- with treatment time. The amount of nitrite generated by He plasma is almost same after 5 and 10 min while in case of Ar plasma the concentrations of nitrite in liquid are negligible. This result may also reflect a significant difference in power dissipation under different feed gas composition.

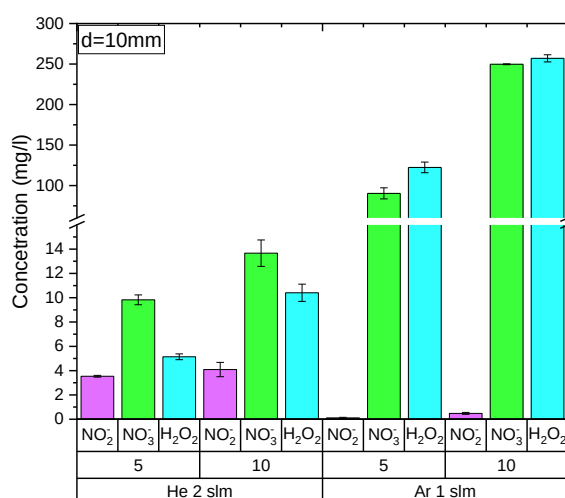


Fig. 2. Concentrations of nitrite, nitrate, and hydrogen peroxide in PAW samples after 5 and 10 minutes treatments using He and Ar as working gases.

4. Conclusions

Plasma treatments of distilled water were done using an APPJ in a pin-electrode configuration. These experiments demonstrated that changing the working gas produced different amounts of measured reactive species in PAW while in both cases filamentary type of plasma was established. Correlating measurement of plasma parameters with liquid sample properties will enable to investigate influence of different plasma parameters on changes in the properties of treated samples. Furthermore, it will be important to explore the potential use of these samples for the particular application in plasma agriculture field.

5. Acknowledgments

This research has been mainly supported by MESTD Republic of Serbia projects III41011 and ON171037. Part of the research was funded by IPB through grant by MESTD.

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ON MECHANISM OF REACTIVE NITROGEN SPECIES FORMATION IN NEGATIVE POLARITY HIGH PRESSURE GLOW DISCHARGE

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Mass spectroscopy (MS) was used for detection of oxygen and nitrogen species produced by negative polarity high pressure glow discharge (HPGD). Atomic O and N species as well as nitrogen oxides NO and NO₂ were detected. It was not possible to detect ions generated in the discharge directly. As shown by chemical kinetic model, concentration of negative ions is negligible in HPGD because of elevated temperature. Chemical kinetic model was also used to study formation pathways of species detected by MS.

1. Introduction

Electrical discharges can generate chemically active non-equilibrium plasmas, where electrons have significantly higher energy than ions and neutral particles. Among the many types of electrical discharges, low-pressure glow discharges are one of the most common and fundamental, often used for illumination [1]. While less common and well-known than their low-pressure counterparts, high pressure glow discharges (HPGD) offer the possibility of removing organic pollutants from exhaust gases [2]. Furthermore, their stability and efficiency in generating nitric oxide from air make them useful for nitrogen fixation [3].

HPGD can be ignited between a high-voltage metal electrode and a water surface, with the second electrode submerged [4]. This configuration, in combination with the generation of nitrogen oxides in the gas phase, makes HPGD suitable for generating plasma-activated water [5].

Plasma-activated water (PAW) is water that has been exposed to plasma. This exposure infuses the water with reactive oxygen and nitrogen species (RONS), such as hydrogen peroxide, nitrates, and nitrites, which temporarily alter the water's chemical properties and make it useful in many applications in food, agriculture, and biomedicine [6, 7]. These possibilities have made PAW a hot topic in the low-temperature plasma community in recent years.

Despite many studies and obtained results, further research is crucial for a better understanding of the formation mechanisms of reactive species, such as nitrogen oxides, and for assessing the role of different gas-phase species in the formation of aqueous RONS in PAW. From a practical point of view, this knowledge will allow for increased energy efficiency and selectivity with respect to the desired products when generating PAW. For this reason, the formation of RONS by HPGD is studied in this paper, using mass spectrometry (MS) and chemical kinetic modelling. For MS measurements we used molecular beam mass spectrometer (MBMS) that can sample from atmospheric pressure enabling to directly access chemical species created in the plasma. In principle the MBMS can detect both neutral and ionic species but in this study we focused to neutrals.

2. Experimental setup

A schematic diagram of the experimental apparatus is shown in Figure 1. The high pressure glow discharge was generated by a DC high voltage (HV) power supply (Glassmann PSIWH 20R25) with negative polarity output, capable of delivering up to 20 kV. The power supply was connected to the cathode via a 1 MΩ series resistor (R) to limit current. This power supply can also operate as a stabilized current source, providing up to 30 mA.

The cathode consisted of a stainless steel needle with a flat tip and an outer diameter of 0.7 mm. The discharges were generated in dry air from the pressure cylinder (purity 5.0), flowing along the cathode

towards the anode with the gas flow rate of 0.3-0.45 slm, controlled by mass controller (Bronkhorst F201-EV).

For diagnostics of neutral species, a grounded steel ring with an inner diameter of about 4 mm and an outer diameter of about 8 mm served as the anode. This ring was positioned 5 mm in front of the entrance to the mass spectrometer, which had an orifice diameter of 100 μm . For the measurements the mass spectrometer front plate with the orifice was grounded. The cathode-anode gap distance was maintained at 3 mm.

The electrical potential between the cathode and ground was measured using a high voltage probe (Tektronix P6015A) connected to a digital oscilloscope (Keysight MSOX 3024T).

The MBMS used for measurements (Hiden Analytical HPR 60) was operating in two modes- RGA (Residual Gas Analyzer) mode, that provides data on the mass spectra of neutrals in the range 0–100 amu; and MID scan, when the device is set to monitor the temporal changes of selected species. In both cases, for the detection of neutral species a ionization chamber was active with the electron energy set to 70 eV. The MBMS has an internal shutter – Swagelok that allows recording of the background signal coming from the gas phase inside the device.

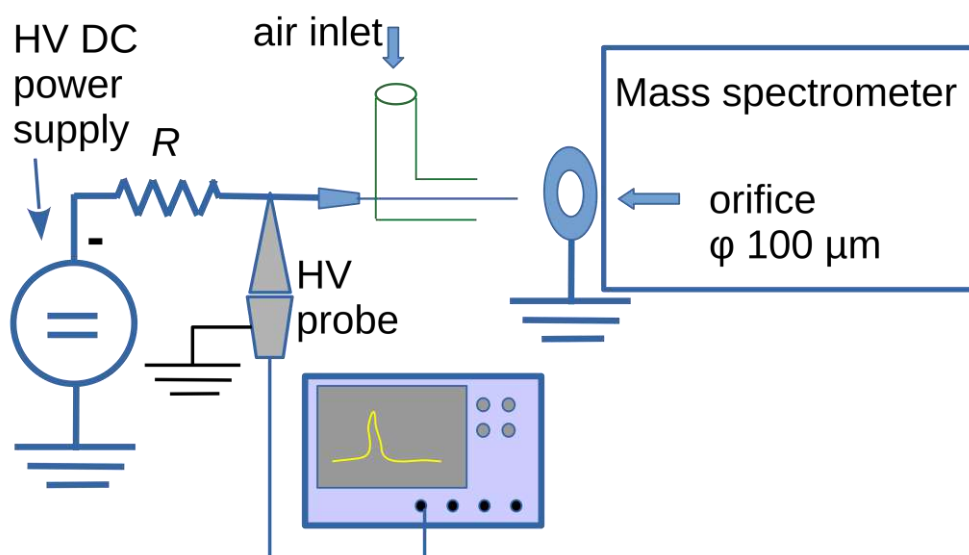


Fig. 1. A schematic diagram of the experimental apparatus.

3. Chemical kinetic model

The aim of the chemical kinetic model is to calculate density evolution of studied species interacting via defined set of chemical reactions. For this purpose we used ZDPlasKin module [8] that includes a Fortran 90 version of the VODE solver for numerical solution of system of ordinary differential equations [9]. Authors of ZDPlasKin also provide a ready-to-use list of plasma chemical processes in nitrogen-oxygen mixtures with all necessary rate coefficients [10]. This set of reactions (version 1.03) includes ~650 chemical reactions among 53 species.

The rate constants of reactions between heavy species from this list are calculated from the thermodynamic gas temperature T_g . The rate constant for electron impact reactions must be calculated from electron energy distribution function (EEDF) obtained by solving the Boltzmann equation for free electrons. The ZDPlasKin package includes a Bolsig+ solver [11] for this purpose. A set of required electron scattering cross sections was taken from the LXCat project database [12]. Finally, ZDPlasKin module requires use of additional subroutines written by user for comprehensive control of simulation conditions, e.g., changes in the gas temperature, pressure and reduced electric field. Our physical model has two parts, glow discharge and afterglow. The afterglow part has to be included because in some experiments, the gas from the discharge did not enter the mass spectrometer directly, but there was a 5 mm gap between the grounded ring electrode and orifice.

For modeling of GD we used constant temperature $T_g = 2000$ K, constant pressure of 1 atmosphere and constant reduced electric field strength $E_n = 60$ Td. We also used constant electron density $n_e = 10^{12}$ cm⁻³. These values were estimated based on previous experimental observations of HPGD [13]. In order to take into account diffusion of species out of the discharge plasma channel and mixing with the surrounding ambient air, we included a primitive diffusion model in our code. After each calculation step with duration Δt , concentration of each heavy particle n_i is decreased by Δn_i calculated as

$$\Delta n_i = -\alpha_{diff} n_i \Delta t, \quad (1)$$

where α_{diff} is coefficient representing diffusion of particles out of the plasma channel. To keep constant pressure (total density of particles), the removed particles were replaced by N₂ and O₂ molecules (ratio 4:1). This simulates mixing with the ambient air.

In the second part of the model, an afterglow period of 0.2 s, the reduced electric field strength decreased exponentially to 3 Td with a time constant of 20 ns. Electrons concentration was calculated dynamically along with the densities of all other species, rather than being held constant. Mixing with the surrounding air continued during the afterglow phase, leading to a calculated decrease in gas temperature. Electron diffusion was also incorporated, with a diffusion coefficient ten times higher than that of the heavy particles.

4. Results and Discussion

HPGD was generated with discharge current of either 2.1 mA, or 3 mA. The applied voltage was -3 kV and -4 kV, respectively. The discharge voltage (across the gap) decreased from ~1.3 kV to ~1.15 kV when the HPGD current increased from 2.1 mA to 3 mA. Mass spectra of produced neutral species were measured for both discharge currents. The obtained spectra had the same species visible for both currents so in Figure 2 we present the data recorded for 3 mA.

The data shows only species created in the plasma as the background signal, recorded in plasma off conditions, was subtracted. The most abundant species created in the discharge were atoms of H, N and O and reactive species OH, H₃O, NO, H₂O₂ and N₂O.

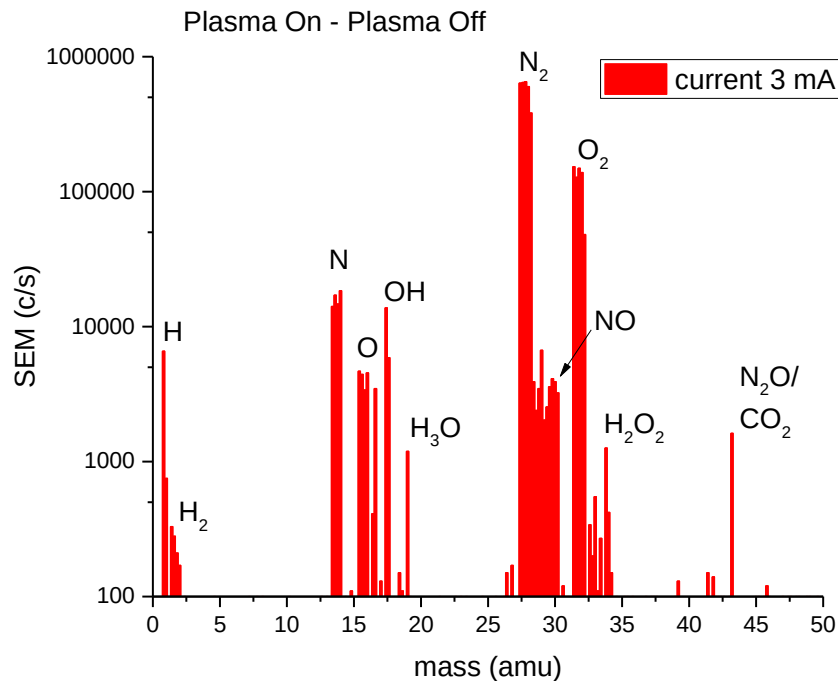


Fig. 2. Mass spectra of neutral species measured for current 3 mA.

In the MID scan mode, we monitored temporal changes in the nitrogen species NO, N₂O and NO₂ important for PAW generation. In Figure 3 we show recorded signals for these species with respect to different measurements conditions. Measurements were conducted with Swagelok open and with Swagelok closed. Swagelok open represents the sum of foreground and background species, while Swagelok closed corresponds to background species only. For the first 6 minutes of recording the discharge was off so variations in the signal is only due to processes inside the device. Once the discharge is ignited, increase in NO and NO₂ signals was due to the species created in the discharge. Obviously, creation of N₂O species was not large so the signal did not change after discharge inception.

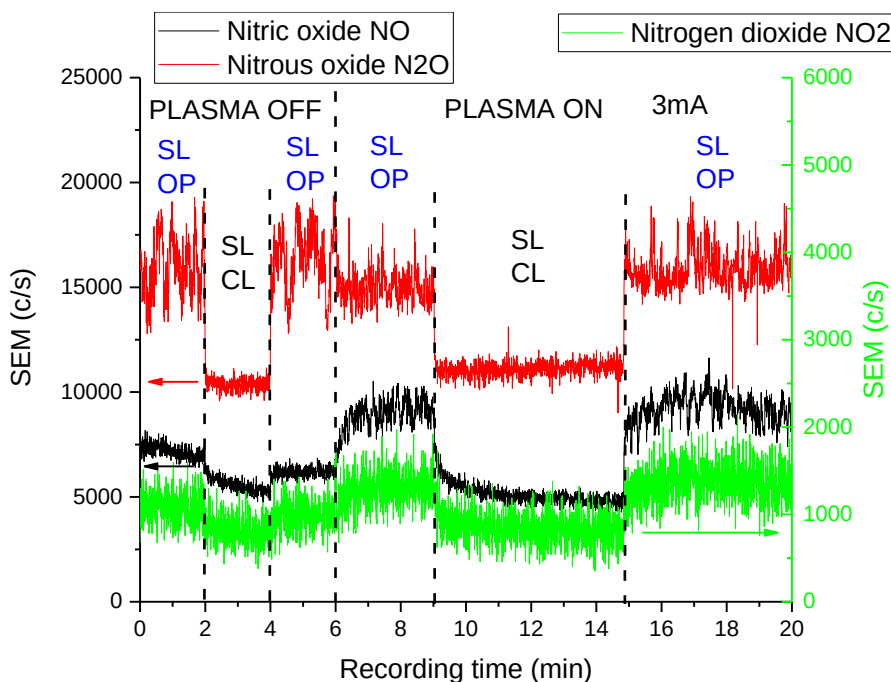


Fig. 3. MID-scan of NO, NO₂ and N₂O species without and with the discharge operating in front of the MS orifice with addition of synthetic air. Discharge current was 3 mA. Measurements were performed with Swagelok closed (SL CL) and open (SW OP).

In summary, the experimental data confirmed generation of N, O, NO and NO₂ by HPGD. Chemical kinetic model using ZDPlasKin module was used to explain their formation. As for example, Figure 4 shows time evolution of N, O, NO, NO₂ and O₃ species concentrations in the glow discharge. This calculation was performed with $\alpha_{diff} = 2 \text{ s}^{-1}$.

Our model incorporates diffusion, but the employed calculation approach is simplified, and the exact value of the diffusion coefficient remains unknown. Instead, we utilize a parameter, α_{diff} , to represent the diffusion rate. We performed several calculations with α_{diff} ranging from 0.1 to 10 s⁻¹. Since α_{diff} influences the calculated steady-state concentrations of species in the plasma, we cannot definitively determine the actual concentrations of the studied species in the HPGD. However, we observed that α_{diff} in studied range does not significantly affect the ratio of concentrations of various RONS or their production pathways.

Figure 4 demonstrates that the concentrations of all studied RONS reach a steady state after approximately 0.2 ms, remaining constant thereafter. This steady state arises from a balance between production (through chemical reactions) and removal (via chemical reactions and diffusion). Consequently, we analyzed the reaction pathways separately for the initial phase of the simulation and for the subsequent steady state.

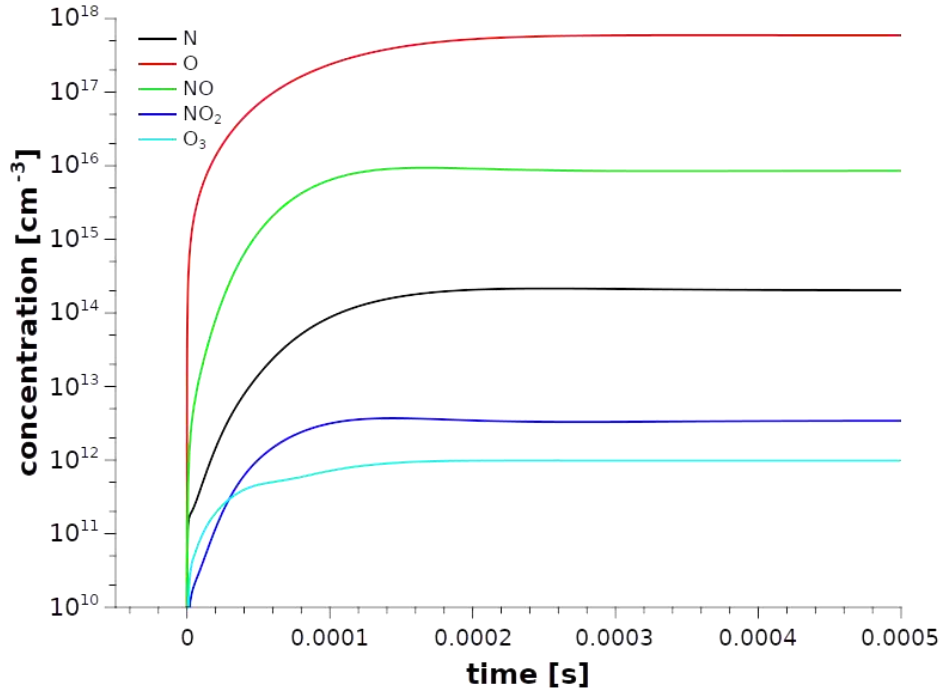


Fig. 4. Time evolution of N, O, NO, NO₂ and O₃ species concentration; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

In the initial phase (Figure 5), O atoms are mainly produced by reactions of O₂ with electronically excited molecular nitrogen species N₂* (the most important being N₂(B³Σ), and by electron impact dissociation of O₂ molecules:



Contribution of other reactions, such as



on O atoms production is already quite small.

In the later steady state phase, the production reactions (2-4) are compensated by O atoms recombination reactions



In this simplified O production/removal mechanism we omitted reactions between O, O(¹D) and O(¹S) species. An equilibrium between these species is achieved quickly, with O representing more than 99.9% of them.

N atoms are produced mostly by these two reactions:



There is also a third important reaction producing N atoms, electron impact dissociation of N₂, but it plays an important role at the very beginning of the simulation ($t < 50 \text{ } \mu\text{s}$), when there is still not enough NO molecules for N generation by equations (6) and (7).

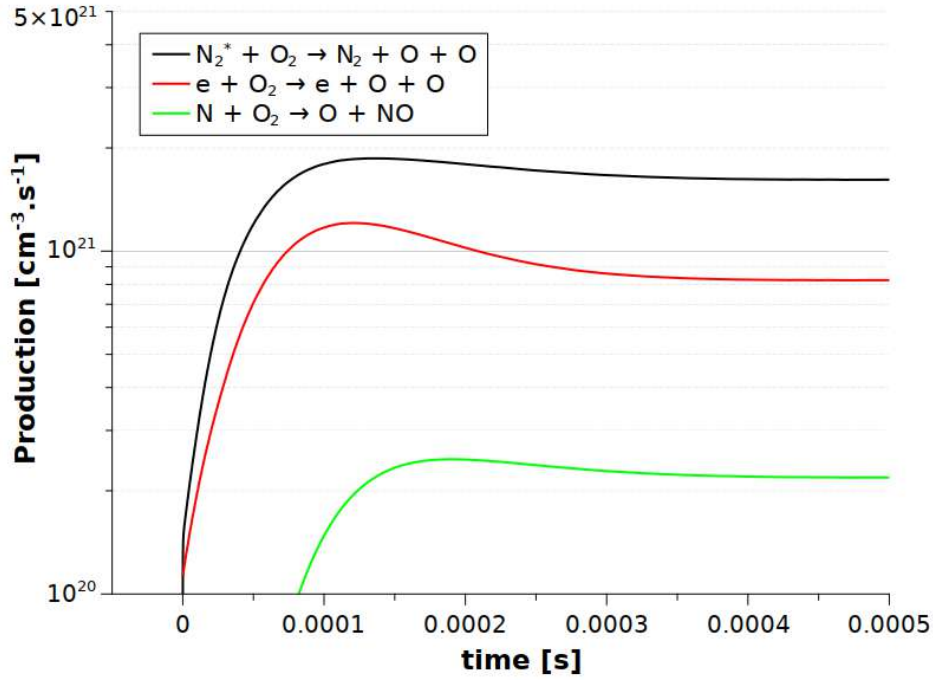


Fig. 5. Production of O atoms during the initial phase of the simulation; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

In steady state, the removal of NO molecules by N (equations (6) and (7)) is compensated by NO production via reaction



Reactions (6-8) all involve NO, they are crucial not only for N production and losses, but they also influence the density of NO. Reaction (8) is actually the most important for NO production (Figure 6). The other two important reactions are



In the steady state, the production of NO is compensated by its removal (Figure 7) via reactions (6), (7), (11) and (12)



NO_2 is produced almost exclusively by reaction (13) and removed by reaction (14)



Steady state NO_2 concentration $[\text{NO}_2]$ can be therefore easily calculated as $[\text{NO}_2] = k_{13} \cdot [\text{NO}] / k_{14}$, assuming balance between NO_2 production and destruction by reactions (13) and (14). In this formula, $[\text{NO}]$ is steady state concentration of NO, k_{13} and k_{14} are rate coefficients of reactions (13) and (14), respectively. At 2000 K, the ratio of these two rate coefficients is 7.3×10^{-4} and it explains why the steady state concentration of NO_2 is much lower than the concentration of NO (Figure 4).

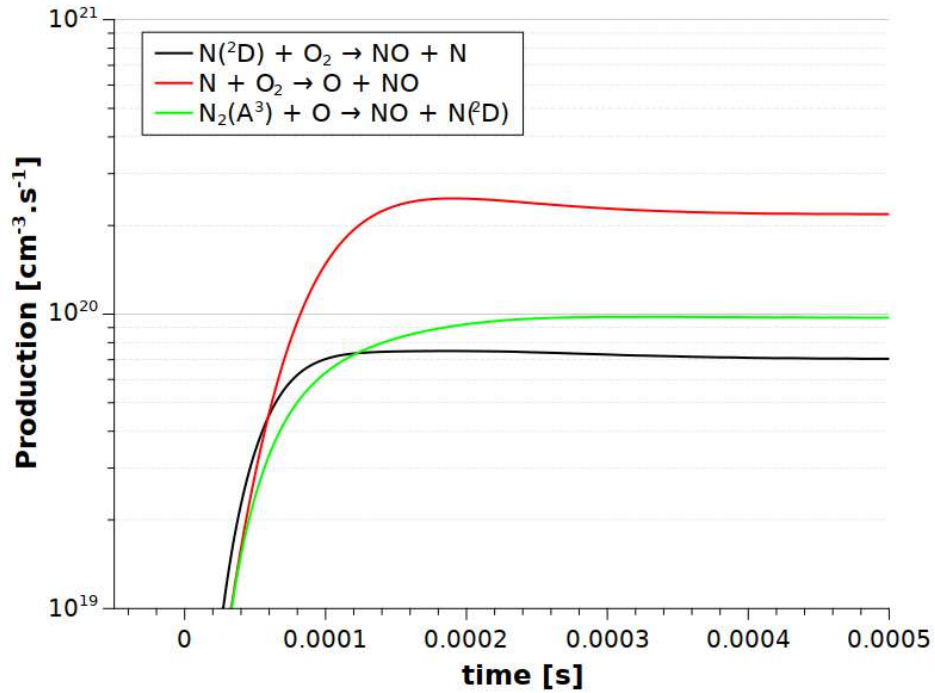


Fig. 6. Main reactions responsible for production of NO molecules; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

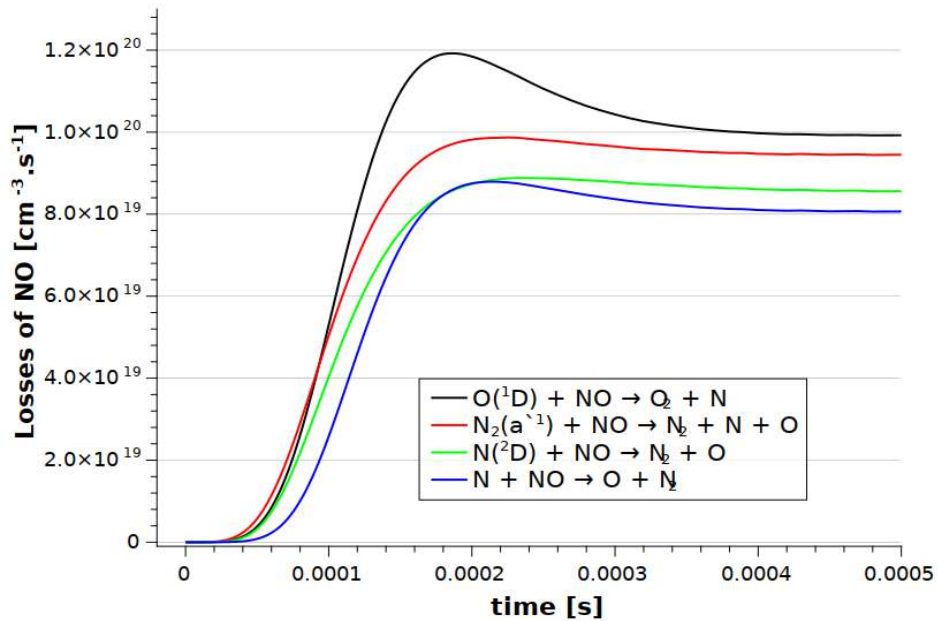


Fig. 7. Main reactions responsible for losses of NO molecules; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

Based on *ex-situ* measurements of the gas after treatment by glow discharge, the concentration of NO is higher than the concentration of NO₂, but the difference is not so significant [3]. Final NO and NO₂ concentration is mainly determined by reactions in the gas after leaving the discharge zone (afterglow). Figure 8 shows time evolution of gas temperature and N, O, NO and NO₂ species concentrations in the discharge afterglow, calculated with $\alpha_{diff} = 10 \text{ s}^{-1}$. The concentration of atomic species (N and O) decreases rapidly. While the NO concentration decreases slowly, the NO₂ concentration increases, despite mixing with the ambient air. Consequently, the difference between NO and NO₂ concentrations decreases in the discharge afterglow.

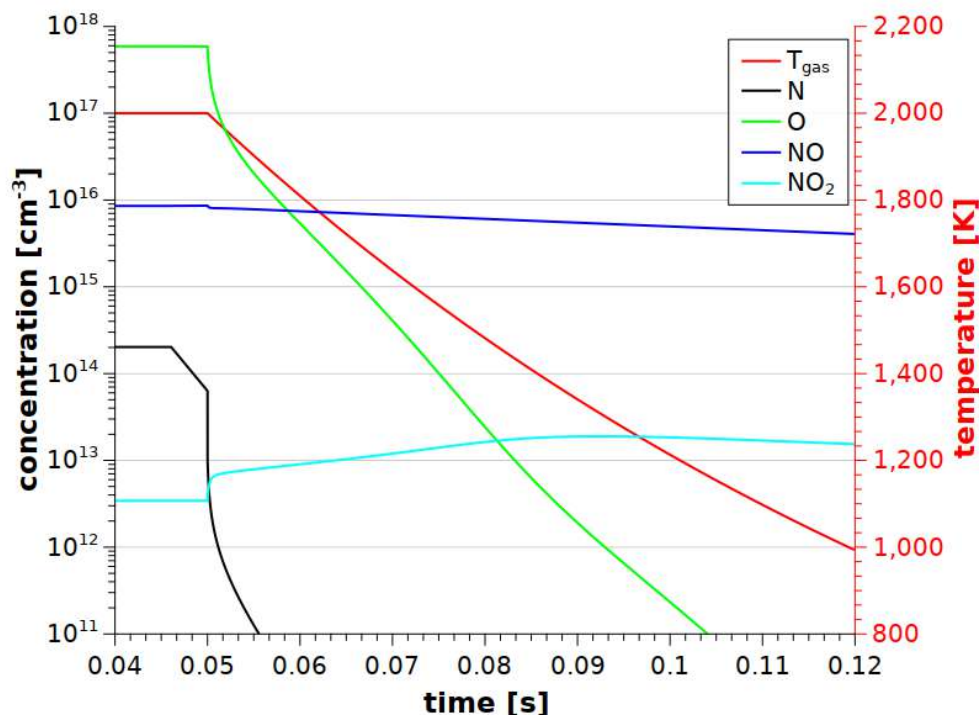


Fig. 8. Time evolution the gas temperature and N, O, NO and NO₂ species concentrations; calculated with $\alpha_{diff} = 10 \text{ s}^{-1}$.

A stable signal from ions directly generated by the HPGD was not detected by mass spectrometry. This can be attributed to two main factors. Firstly, the HPGD migrated on the surface of the orifice plate that served as anode, making its position relative to the spectrometer's gas entrance (a 100 μm orifice) highly variable. A stable ion signal could only be observed if the HPGD was positioned directly above this orifice, which occurred only randomly and occasionally. Secondly, even with the discharge positioned stably above the orifice, ion detection remains challenging. Positive ions are repelled from the anode, and the concentration of negative ions within the HPGD is relatively low, according to the kinetic model. The dominant negative ion, O⁻, has a concentration of approximately 10^{10} cm^{-3} , two orders of magnitude lower than the electron concentration. This low negative ion concentration may be due to the elevated gas temperature in the HPGD plasma channel.

5. Conclusions

Fixation of nitrogen from the air, converting it into reactive compounds, remains a significant challenge and a hot topic within the low-temperature plasma community. High-pressure glow discharges offer a stable and efficient means to generate nitrogen oxides, key precursors for nitrogen fixation. This study investigates the mechanisms of nitrogen oxide generation in an HPGD using a combined approach of mass spectrometry and chemical kinetic modeling.

Mass spectrometry provided insights into the types of reactive oxygen and nitrogen species produced, while the kinetic model simulated the complex chemical reactions within the plasma. Our findings shed light on the dominant reaction pathways on nitrogen oxide formation. However, further research is crucial to refine our understanding. Future experimental work should focus on improving the detection and quantification of reactive species, particularly ions. Model enhancements are also necessary, including a more accurate representation of diffusion processes and the discharge afterglow phase. Furthermore, incorporating additional chemical reactions involving water molecules will enable us to explore the crucial role of humidity in the generation of nitrogen oxides, nitric acid, and nitrous acid. This comprehensive approach promises to advance our knowledge of nitrogen fixation using HPGD

and pave the way for the development of efficient and sustainable plasma-based technologies for fertilizer production and other applications.

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MASS SPECTROMETRY OF DIELECTRIC BARRIER DISCHARGE WITH WATER ELECTRODE

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This paper presents the results of an investigation into the generation of reactive species in two setups of a dielectric barrier discharge (DBD) plasma source, using a water target with different vessels. By analyzing both neutral mass spectra and MID-scan spectra, we explore the concentrations of reactive oxygen and nitrogen species (NO, NO₂ and O₃) under varying plasma conditions and mass spectrometer configurations.

1. Introduction

In the past decade, a lot of interest has been drawn to atmospheric pressure plasmas (APPs) because of their unique properties and wide range of applications in fields such as material processing, agriculture, food industry and biomedicine [1, 2, 3, 4]. Since they do not require costly vacuum systems and operate at atmospheric pressure, APPs have the advantage of being accessible and versatile. Lately, atmospheric pressure plasma in contact with water has attracted significant interest due to its potential to generate reactive species and drive advanced chemical processes for various applications.

The behaviour and chemistry of APPs are greatly affected when they come into contact with water, either as an electrode or as a target. Reactive oxygen, nitrogen and hydrogen species are found in water and are essential for a variety of processes, such as biomedical treatments, sterilization, and water purification. Water forms a dynamic interface where plasma-induced reactions take place, producing reactive species like ozone (O₃), hydrogen peroxide (H₂O₂), hydroxyl radicals (OH), nitrates (NO₃⁻), nitrites (NO₂⁻) etc. These species play an essential role in enhancing the efficacy of plasma-based processes [5].

Also, higher humidity introduced in feeding gas of APPs has been demonstrated to increase the generation of reactive species, such as OH radicals, which are necessary for surface modification and decontamination [6]. The interaction, however, is complex and depends upon a number of variables, including ambient circumstances, water composition, and plasma characteristics. To optimize plasma processes and customize them for particular applications, it is essential to comprehend these interactions.

Dielectric barrier discharges (DBDs) are widely used for surface activation in atmospheric-pressure plasma applications. However, treating sensitive materials like polymers can be challenging because high-density plasma may cause damage, such as pin-holing. This issue often occurs in volume barrier discharges or coronas, where the plasma moves perpendicular to the treated surface. A practical solution is to generate plasma that travels parallel to the surface. This approach minimizes the risk of damage while maintaining effective treatment. One promising method is the surface dielectric barrier discharge (SDBD), where the plasma spreads along the surface of a dielectric plate. This setup not only protects the material but also improves the efficiency of the process.

In our earlier work [7, 8, 9] we introduced a novel plasma discharge reactor for efficiently activating polymers at the gas/liquid interface. This design uses liquid electrodes to ignite the SDBD directly from the liquid surface. Although the plasma-water interaction is limited to the edge of the dielectric tube, the system is both scalable and flexible, making it suitable for a wide range of applications.

Mass spectrometry is an analytical technique that measures the mass-to-charge ratio of ions to identify and quantify molecules in a sample. Primary advantage of atmospheric pressure mass spectrometry lies in its ability to rapidly and accurately analyse a wide range of chemical species [10]. These instruments are equipped with specialized pumping systems that create a pressure gradient, enabling the effective intake of gases from atmospheric plasmas. For neutral species, the mass spectrometer incorporates an ionization chamber that converts neutrals into ions, enabling their detection and analysis. The mass analyser which filters and detects neutral species or positive and negative ions, generating detailed mass spectra for all components. The technique provides real-time measurements of reactive species, ions, and neutral molecules, which are essential for understanding plasma processes and optimizing plasma-based applications.

When it comes to plasma in contact with water, due to technical challenges, mass spectrometric analysis of the plasma has so far been conducted by introducing water vapour into the working gas [11]. In this paper, we tackled the technical challenge and developed a setup where the mass spectrometer inlet was positioned in close proximity to the plasma-water interface, allowing us to successfully record mass spectra.

In this paper, we will present the results of our investigation into the reactive species generated by the dielectric barrier discharge (DBD) setup, but with two different water vessel configurations. The analysis includes both neutral mass spectra and MID-scan spectra, offering insights into the concentrations of key reactive oxygen and nitrogen species, such as NO, NO₂, CO₂ and O₃, as well as the detailed composition of the plasma obtained under these conditions.

2. Experimental set up

The schematic of the DBD at atmospheric pressure and HIDEN HPR60 mass-energy spectrometer is given in Figure 1. The DBD device is in the triple-phase interface (plasma-liquid-solid) plasma system consisting of a thin glass test tube with a 10 mm diameter and a 0.5 mm wall thickness was used. The liquid inside the test tube served as the high-voltage electrode and was connected to a power supply generating a sinusoidal voltage waveform. The Petri dish bath, which grounded the system, completed the circuit. Tap water with an electrical conductivity of approximately 0.3 mS/cm was used as the liquid electrode. The water was electrically insulated both inside and outside the test tube to ensure stability. The discharge operated in ambient air at atmospheric pressure. The high-voltage sine waveform had an amplitude range of 0 to 20 kV and could be adjusted to frequencies between 23 and 30 kHz. Power was delivered to the liquid electrodes through a high-voltage resonance generator (Lifetech-300W) paired with a function generator (FY3200S-24M).

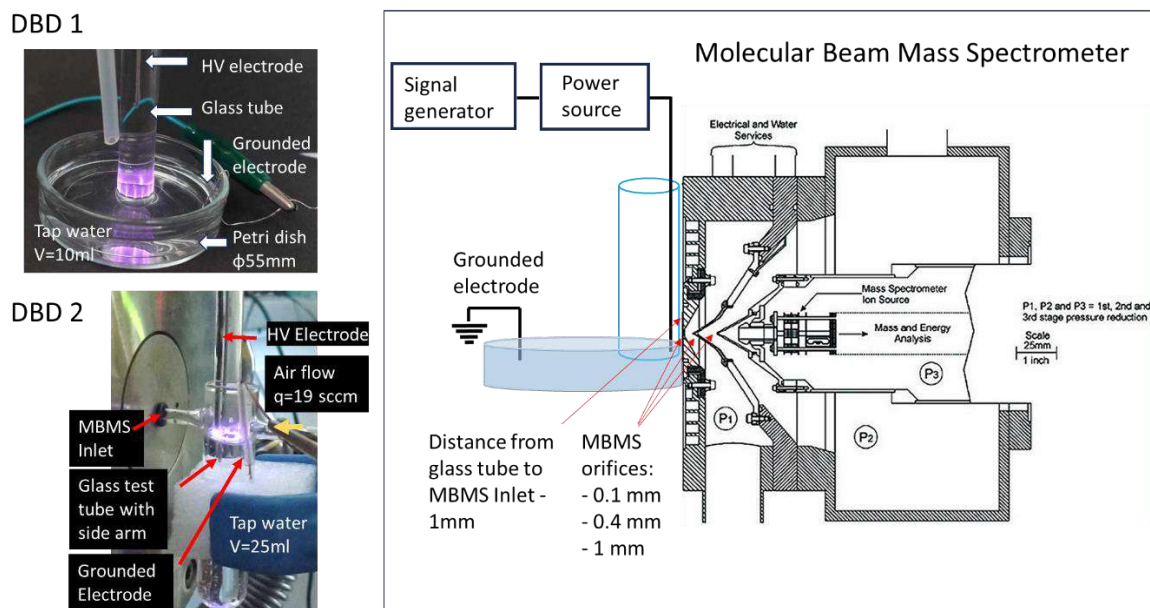


Fig. 1. DBD device in two configurations (DBD1 and DBD2) with a schematic representation of mass spectrometry measurements. In DBD1 setting, the device was immersed in 10ml of tap water placed in a Petri dish ($\phi 55\text{mm}$). In DBD2 setting, the device was placed in a glass test tube filled with 25ml of tap water, equipped with a side arm for gas sampling and a hole for synthetic air intake ($q = 19 \text{ sccm}$).

Mass spectrometry measurements were performed by using MBMS (Molecular Beam Mass Spectrometer) Hiden HPR60. To ensure the formation of a molecular beam, the geometry of the MBMS HPR60 system consists of a centralized combination of the orifice, cone1, and cone2 (P1 vacuum section is formed between the orifice and cone1, P2 vacuum section is formed between cone1 and cone2, and P3 vacuum section is formed after cone2 in the region of the mass analyzer). The orifice had an opening diameter of $\phi 0.1 \text{ mm}$, cone1 $\phi 0.4 \text{ mm}$, and cone2 $\phi 1 \text{ mm}$, accompanied by the following voltages for DBD setups (the orifice was grounded, $V_{\text{cone1}} = 0 \text{ V}$ and $V_{\text{cone2}} = 0 \text{ V}$).

During all experiments for DBD setups, the regions within the vacuum section of the mass spectrometer responsible for generating the pressure gradient had the following pressure values: $P_1 = 3.3 \cdot 10^{-1} \text{ Torr}$, $P_2 = 7.5 \cdot 10^{-6} \text{ Torr}$, and $P_3 = 2.4 \cdot 10^{-7} \text{ Torr}$. To identify the species of interest, we first recorded the mass spectra of neutrals (0–100 amu) using the RGA (Residual Gas Analyzer) mode, during which the ionization chamber was active. Within the ionization chamber, the electron emission current from the filament was for DBD1 = 5 μA and for DBD2 = 10 μA . In both cases, the electron energy was 70 eV.

After that, we used MID-scan to monitor the temporal changes of selected species for different formation conditions: without plasma, with plasma at specific applied powers for DBD1 (5W and 15W) and DBD2 (15W), with Swagelok open, and with Swagelok closed. Swagelok open represents the sum of foreground and background species, while Swagelok closed corresponds to background species only.

3. Results and discussion

In this study, we have measured the neutrals mass spectra by using a mass spectrometer for two different configurations of Dielectric Barrier Discharge (DBD) system. In both configurations, titled DBD1 and DBD2, discharge was in contact with water during mass spectrometry measurements. The analysis of neutral species was performed in two different measurement modes of HPR60: RGA mode for neutral mass spectra and MID-scan mode for track in time changes of specific neutral species. These modes provide a comprehensive overview of the ionization processes and chemical compositions

present in the plasma generated by each DBD system. The neutral mass spectra revealed the types and relative concentrations of neutral species, the MID-scan measurement provided insight into the temporal evolution of these species.

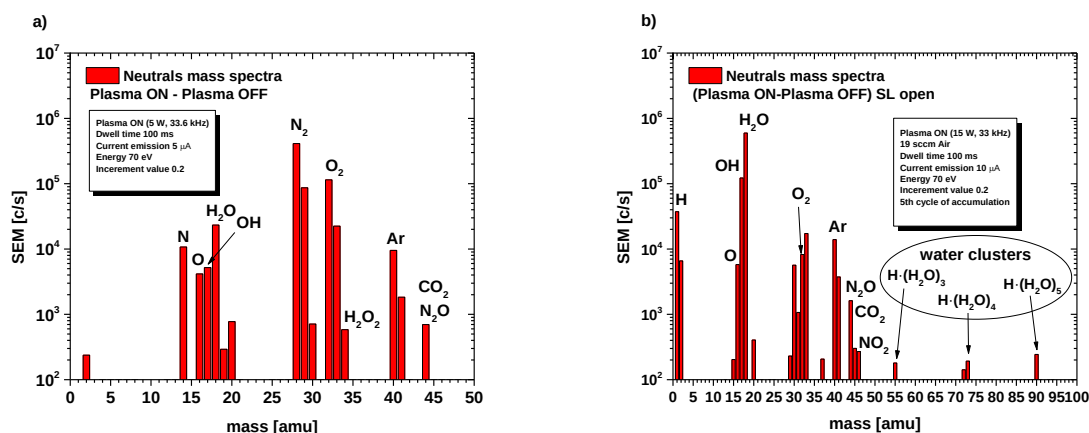


Fig. 2. Neutral mass spectra for a) DBD1 setting and b) DBD2 setting at an operating frequency of 33.6kHz. In the case of the DBD2, an airflow of 19sccm was added. The graphs were obtained by passing the raw results through a MatLab script to integrate the obtained lines for specific mass number.

Figure 2(a) and (b) present Neutral mass spectra for configurations DBD1 and DBD2, respectively. The presented spectra show the difference of Plasma ON mass spectra and Plasma OFF mass spectra. Plasma ON/OFF mass spectra represent the foreground signal i.e. the difference of total signal (swagelok open) and background signal (swagelok closed). The spectra in Figure 2 clearly show that the dominant species in the discharge are nitrogen and oxygen compounds (N, O₂, H₂O₂, N₂O, H, H₂O) which is to be expected because it is a discharge at atmospheric pressure where the target is water. Atomic nitrogen and atomic oxygen are present as a result of plasma reactions, along with the NO radical. The OH radical is also present, resulting from both plasma reactions and water dissociation in the MBMS. In Figure 2(b), water clusters can also be observed at mass numbers 53, 73 and 91, which appear in neutral mass spectra due to water vapor from the bottle containing the discharge. The high humidity in the DBD2 configuration and the plasma conditions promote cluster formation unlike in the case of DBD1 configuration where we did not detect any water clusters of mass above 50 amu.

While the mass spectra results provided an overall view of the main species present in the discharge chamber, nitrogen oxides and ozone can impact the industrial environment even at much lower concentrations (below the detection limit of the mass spectra used). Therefore, more sensitive measurements were conducted specifically for the important species NO, NO₂, N₂O, and O₃, as represented here. It was recorded for different conditions, Plasma ON and OFF with background only (BG), as well as Plasma ON and OFF with foreground and background (FG+BG). Unlike the case of BG, where only the inner part of the MS is considered in the measurement, in the case of FG+BG, the mass spectrometer is open so outside ambient air is also evaluated.

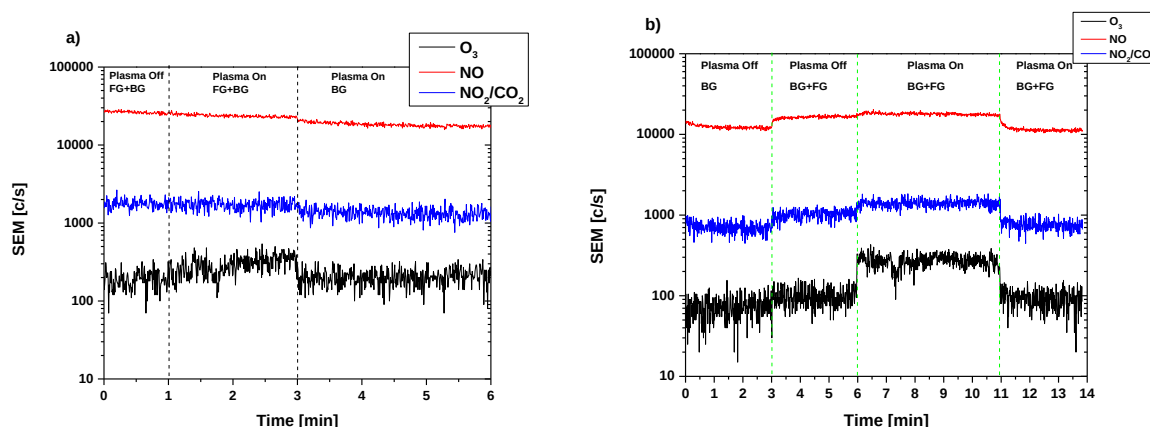


Fig. 3. The mid-scan provides constant tracking of selected species, with different stages of the experiment marked by labels: plasma on and off, foreground (FG) and background (BG); (a) DBD1 Setup, ionization filament current set to 5 μA at 70 eV, and (b) DBD2 Setup, ionization filament current set to 50 μA at 70 eV.

The MID-scan for DBD1 setup (Figure 3(a)) starts with Plasma OFF and swagelok open (1st minute), both the foreground (outside the spectrometer) and background are measured. The discharge is ignited in minute 1 and swagelok is open. Here we can see that NO continues to decrease slightly, NO₂ remains constant, and O₃ increases, indicating plasma-driven production of O₃. After the 3rd minute, with the plasma still on but swagelok closed, all species (NO, NO₂, and O₃) decrease as expected (only background inside device is measured).

The Figure 3(b) shows MID-scan spectra for DBD2 configuration. In the first three minutes, with the plasma off and the swagelok closed, only the background signal is measured. After opening of the swagelok (3-6 minutes), the increase in NO and NO₂ suggests the influence of ambient air, while O₃ remains unchanged. Between 6 and 11 minutes, with the plasma on and the swagelok open, NO and NO₂ rise slightly, but O₃ increases significantly, indicating plasma-driven O₃ production. Finally, from 11th minute, with the plasma on and the swagelok closed, all species decrease due to limited external interaction (only background is measured).

4. Conclusion

Mass spectrometry analysis of two setups of a DBD source with a water electrode, generated at atmospheric pressure was performed. Despite the difference in the water vessel used in each setup, in both setups similar trends in the behavior of reactive species were shown, indicating strong influence of plasma on water target. In both cases, plasma activation leads to the generation of reactive oxygen and nitrogen species, such as NO, NO₂, CO₂, and O₃, with notable increases in O₃ concentration when the plasma is on. Differences in the water vessel may affect the plasma's efficiency in producing reactive species, but both setups demonstrate that the plasma's interaction with the water is crucial for modulating the levels of reactive species. These findings highlight the importance of the plasma-water system in applications such as water treatment and agriculture, where reactive species generated in plasma-activated water could have significant biological and chemical effects.

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Book of Contributed Papers

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31 Jan - 5 Feb, 2025

Edited by G. D. Megersa, E. Maťaš, J. Országh, P. Papp, Š. Matejčík

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ON MECHANISM OF REACTIVE NITROGEN SPECIES FORMATION IN NEGATIVE POLARITY HIGH PRESSURE GLOW DISCHARGE

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Mass spectroscopy (MS) was used for detection of oxygen and nitrogen species produced by negative polarity high pressure glow discharge (HPGD). Atomic O and N species as well as nitrogen oxides NO and NO₂ were detected. It was not possible to detect ions generated in the discharge directly. As shown by chemical kinetic model, concentration of negative ions is negligible in HPGD because of elevated temperature. Chemical kinetic model was also used to study formation pathways of species detected by MS.

1. Introduction

Electrical discharges can generate chemically active non-equilibrium plasmas, where electrons have significantly higher energy than ions and neutral particles. Among the many types of electrical discharges, low-pressure glow discharges are one of the most common and fundamental, often used for illumination [1]. While less common and well-known than their low-pressure counterparts, high pressure glow discharges (HPGD) offer the possibility of removing organic pollutants from exhaust gases [2]. Furthermore, their stability and efficiency in generating nitric oxide from air make them useful for nitrogen fixation [3].

HPGD can be ignited between a high-voltage metal electrode and a water surface, with the second electrode submerged [4]. This configuration, in combination with the generation of nitrogen oxides in the gas phase, makes HPGD suitable for generating plasma-activated water [5].

Plasma-activated water (PAW) is water that has been exposed to plasma. This exposure infuses the water with reactive oxygen and nitrogen species (RONS), such as hydrogen peroxide, nitrates, and nitrites, which temporarily alter the water's chemical properties and make it useful in many applications in food, agriculture, and biomedicine [6, 7]. These possibilities have made PAW a hot topic in the low-temperature plasma community in recent years.

Despite many studies and obtained results, further research is crucial for a better understanding of the formation mechanisms of reactive species, such as nitrogen oxides, and for assessing the role of different gas-phase species in the formation of aqueous RONS in PAW. From a practical point of view, this knowledge will allow for increased energy efficiency and selectivity with respect to the desired products when generating PAW. For this reason, the formation of RONS by HPGD is studied in this paper, using mass spectrometry (MS) and chemical kinetic modelling. For MS measurements we used molecular beam mass spectrometer (MBMS) that can sample from atmospheric pressure enabling to directly access chemical species created in the plasma. In principle the MBMS can detect both neutral and ionic species but in this study we focused to neutrals.

2. Experimental setup

A schematic diagram of the experimental apparatus is shown in Figure 1. The high pressure glow discharge was generated by a DC high voltage (HV) power supply (Glassmann PSIWH 20R25) with negative polarity output, capable of delivering up to 20 kV. The power supply was connected to the cathode via a 1 MΩ series resistor (R) to limit current. This power supply can also operate as a stabilized current source, providing up to 30 mA.

The cathode consisted of a stainless steel needle with a flat tip and an outer diameter of 0.7 mm. The discharges were generated in dry air from the pressure cylinder (purity 5.0), flowing along the cathode

towards the anode with the gas flow rate of 0.3-0.45 slm, controlled by mass controller (Bronkhorst F201-EV).

For diagnostics of neutral species, a grounded steel ring with an inner diameter of about 4 mm and an outer diameter of about 8 mm served as the anode. This ring was positioned 5 mm in front of the entrance to the mass spectrometer, which had an orifice diameter of 100 μm . For the measurements the mass spectrometer front plate with the orifice was grounded. The cathode-anode gap distance was maintained at 3 mm.

The electrical potential between the cathode and ground was measured using a high voltage probe (Tektronix P6015A) connected to a digital oscilloscope (Keysight MSOX 3024T).

The MBMS used for measurements (Hiden Analytical HPR 60) was operating in two modes- RGA (Residual Gas Analyzer) mode, that provides data on the mass spectra of neutrals in the range 0–100 amu; and MID scan, when the device is set to monitor the temporal changes of selected species. In both cases, for the detection of neutral species a ionization chamber was active with the electron energy set to 70 eV. The MBMS has an internal shutter – Swagelok that allows recording of the background signal coming from the gas phase inside the device.

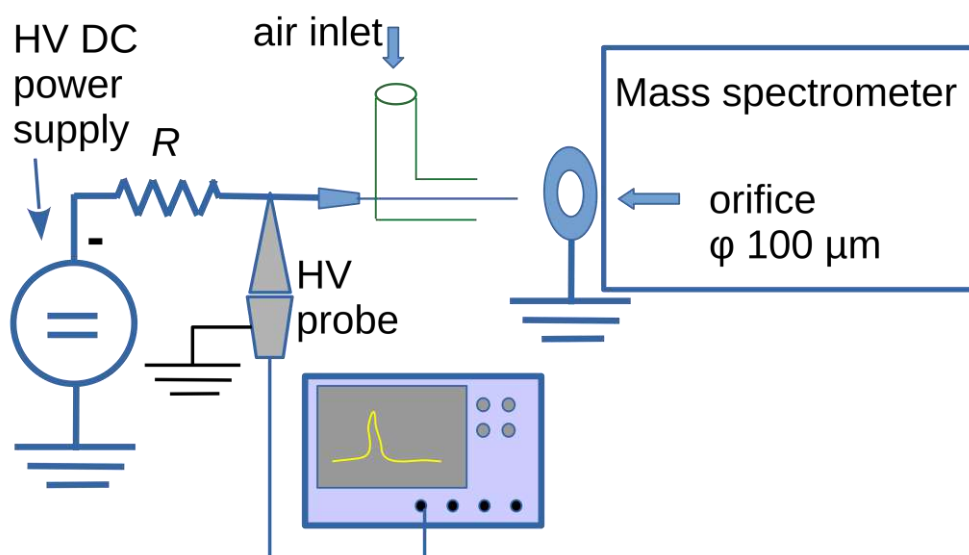


Fig. 1. A schematic diagram of the experimental apparatus.

3. Chemical kinetic model

The aim of the chemical kinetic model is to calculate density evolution of studied species interacting via defined set of chemical reactions. For this purpose we used ZDPlasKin module [8] that includes a Fortran 90 version of the VODE solver for numerical solution of system of ordinary differential equations [9]. Authors of ZDPlasKin also provide a ready-to-use list of plasma chemical processes in nitrogen-oxygen mixtures with all necessary rate coefficients [10]. This set of reactions (version 1.03) includes ~650 chemical reactions among 53 species.

The rate constants of reactions between heavy species from this list are calculated from the thermodynamic gas temperature T_g . The rate constant for electron impact reactions must be calculated from electron energy distribution function (EEDF) obtained by solving the Boltzmann equation for free electrons. The ZDPlasKin package includes a Bolsig+ solver [11] for this purpose. A set of required electron scattering cross sections was taken from the LXCat project database [12]. Finally, ZDPlasKin module requires use of additional subroutines written by user for comprehensive control of simulation conditions, e.g., changes in the gas temperature, pressure and reduced electric field. Our physical model has two parts, glow discharge and afterglow. The afterglow part has to be included because in some experiments, the gas from the discharge did not enter the mass spectrometer directly, but there was a 5 mm gap between the grounded ring electrode and orifice.

For modeling of GD we used constant temperature $T_g = 2000$ K, constant pressure of 1 atmosphere and constant reduced electric field strength $E_n = 60$ Td. We also used constant electron density $n_e = 10^{12}$ cm⁻³. These values were estimated based on previous experimental observations of HPGD [13]. In order to take into account diffusion of species out of the discharge plasma channel and mixing with the surrounding ambient air, we included a primitive diffusion model in our code. After each calculation step with duration Δt , concentration of each heavy particle n_i is decreased by Δn_i calculated as

$$\Delta n_i = -\alpha_{diff} n_i \Delta t, \quad (1)$$

where α_{diff} is coefficient representing diffusion of particles out of the plasma channel. To keep constant pressure (total density of particles), the removed particles were replaced by N₂ and O₂ molecules (ratio 4:1). This simulates mixing with the ambient air.

In the second part of the model, an afterglow period of 0.2 s, the reduced electric field strength decreased exponentially to 3 Td with a time constant of 20 ns. Electrons concentration was calculated dynamically along with the densities of all other species, rather than being held constant. Mixing with the surrounding air continued during the afterglow phase, leading to a calculated decrease in gas temperature. Electron diffusion was also incorporated, with a diffusion coefficient ten times higher than that of the heavy particles.

4. Results and Discussion

HPGD was generated with discharge current of either 2.1 mA, or 3 mA. The applied voltage was -3 kV and -4 kV, respectively. The discharge voltage (across the gap) decreased from ~1.3 kV to ~1.15 kV when the HPGD current increased from 2.1 mA to 3 mA. Mass spectra of produced neutral species were measured for both discharge currents. The obtained spectra had the same species visible for both currents so in Figure 2 we present the data recorded for 3 mA.

The data shows only species created in the plasma as the background signal, recorded in plasma off conditions, was subtracted. The most abundant species created in the discharge were atoms of H, N and O and reactive species OH, H₃O, NO, H₂O₂ and N₂O.

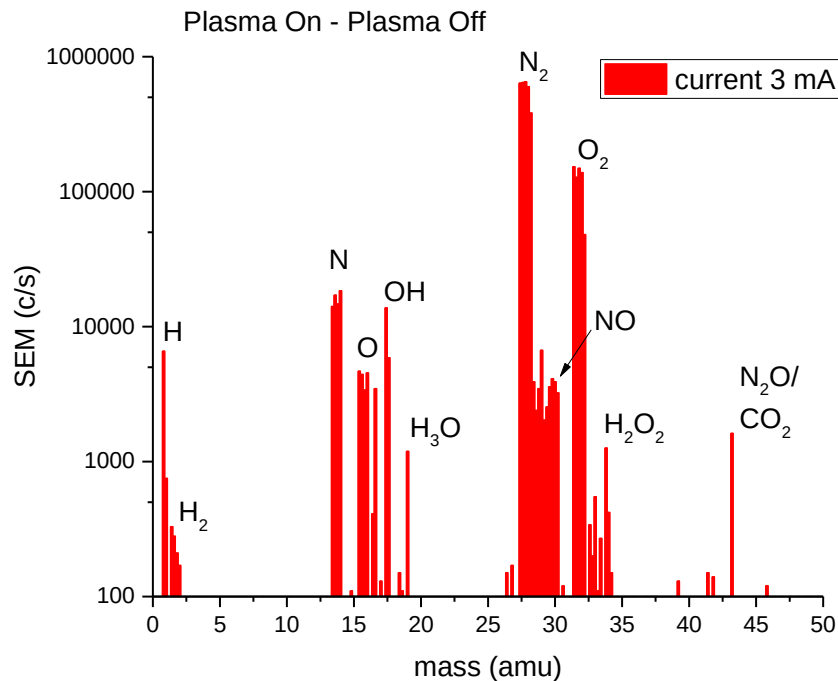


Fig. 2. Mass spectra of neutral species measured for current 3 mA.

In the MID scan mode, we monitored temporal changes in the nitrogen species NO, N₂O and NO₂ important for PAW generation. In Figure 3 we show recorded signals for these species with respect to different measurements conditions. Measurements were conducted with Swagelok open and with Swagelok closed. Swagelok open represents the sum of foreground and background species, while Swagelok closed corresponds to background species only. For the first 6 minutes of recording the discharge was off so variations in the signal is only due to processes inside the device. Once the discharge is ignited, increase in NO and NO₂ signals was due to the species created in the discharge. Obviously, creation of N₂O species was not large so the signal did not change after discharge inception.

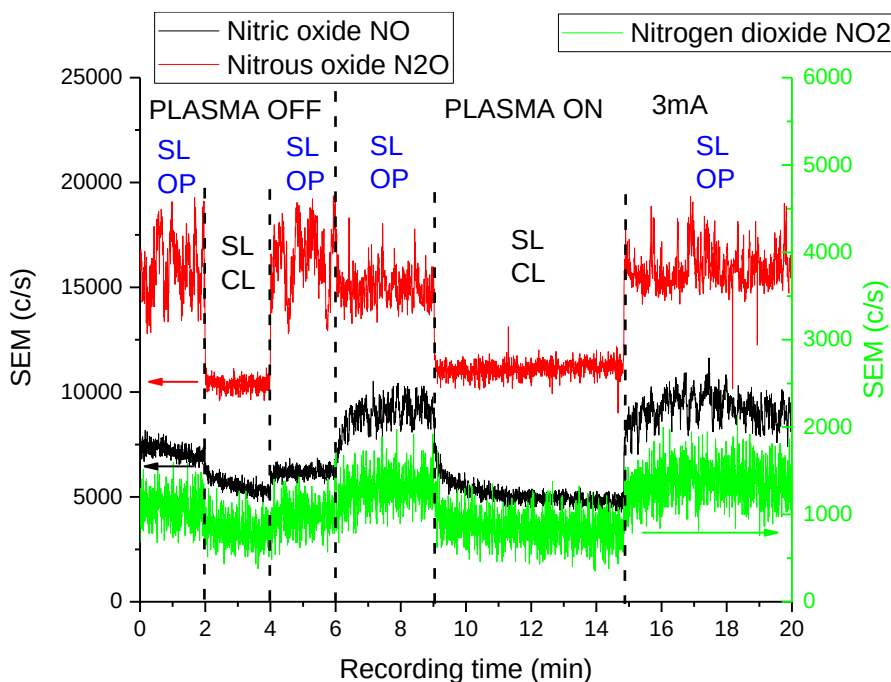


Fig. 3. MID-scan of NO, NO₂ and N₂O species without and with the discharge operating in front of the MS orifice with addition of synthetic air. Discharge current was 3 mA. Measurements were performed with Swagelok closed (SL CL) and open (SW OP).

In summary, the experimental data confirmed generation of N, O, NO and NO₂ by HPGD. Chemical kinetic model using ZDPlasKin module was used to explain their formation. As for example, Figure 4 shows time evolution of N, O, NO, NO₂ and O₃ species concentrations in the glow discharge. This calculation was performed with $\alpha_{diff} = 2 \text{ s}^{-1}$.

Our model incorporates diffusion, but the employed calculation approach is simplified, and the exact value of the diffusion coefficient remains unknown. Instead, we utilize a parameter, α_{diff} , to represent the diffusion rate. We performed several calculations with α_{diff} ranging from 0.1 to 10 s⁻¹. Since α_{diff} influences the calculated steady-state concentrations of species in the plasma, we cannot definitively determine the actual concentrations of the studied species in the HPGD. However, we observed that α_{diff} in studied range does not significantly affect the ratio of concentrations of various RONS or their production pathways.

Figure 4 demonstrates that the concentrations of all studied RONS reach a steady state after approximately 0.2 ms, remaining constant thereafter. This steady state arises from a balance between production (through chemical reactions) and removal (via chemical reactions and diffusion). Consequently, we analyzed the reaction pathways separately for the initial phase of the simulation and for the subsequent steady state.

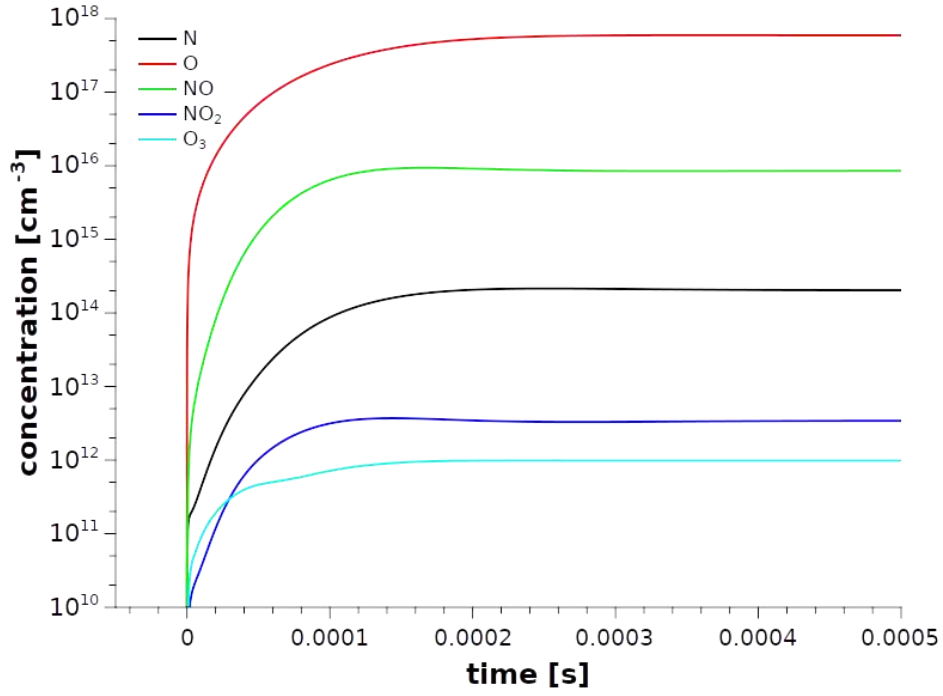


Fig. 4. Time evolution of N, O, NO, NO₂ and O₃ species concentration; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

In the initial phase (Figure 5), O atoms are mainly produced by reactions of O₂ with electronically excited molecular nitrogen species N₂* (the most important being N₂(B³Σ), and by electron impact dissociation of O₂ molecules:



Contribution of other reactions, such as



on O atoms production is already quite small.

In the later steady state phase, the production reactions (2-4) are compensated by O atoms recombination reactions



In this simplified O production/removal mechanism we omitted reactions between O, O(¹D) and O(¹S) species. An equilibrium between these species is achieved quickly, with O representing more than 99.9% of them.

N atoms are produced mostly by these two reactions:



There is also a third important reaction producing N atoms, electron impact dissociation of N₂, but it plays an important role at the very beginning of the simulation ($t < 50 \text{ } \mu\text{s}$), when there is still not enough NO molecules for N generation by equations (6) and (7).

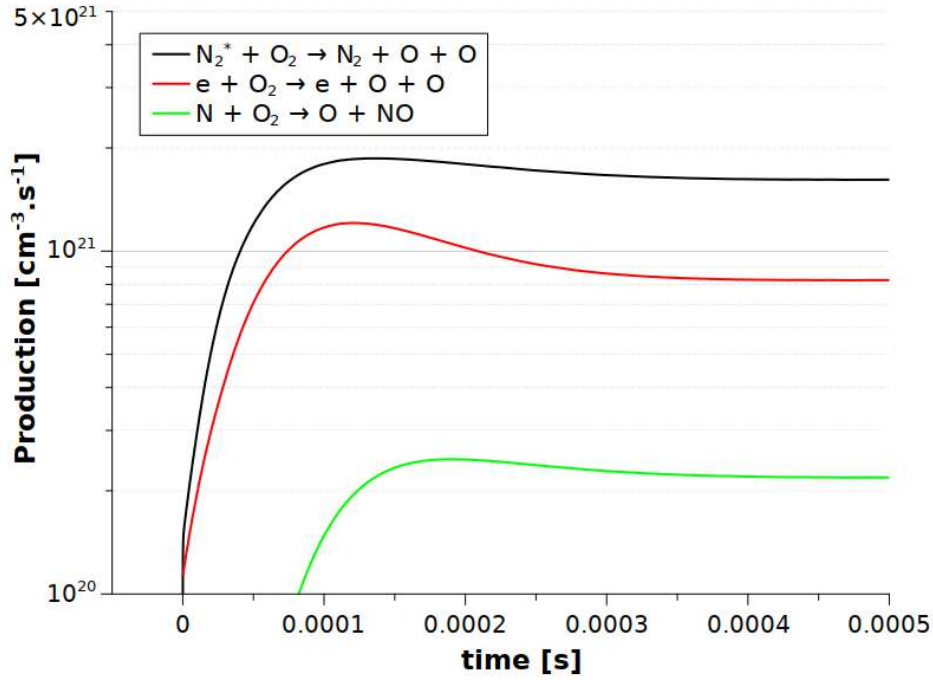


Fig. 5. Production of O atoms during the initial phase of the simulation; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

In steady state, the removal of NO molecules by N (equations (6) and (7)) is compensated by NO production via reaction



Reactions (6-8) all involve NO, they are crucial not only for N production and losses, but they also influence the density of NO. Reaction (8) is actually the most important for NO production (Figure 6). The other two important reactions are



In the steady state, the production of NO is compensated by its removal (Figure 7) via reactions (6), (7), (11) and (12)



NO_2 is produced almost exclusively by reaction (13) and removed by reaction (14)



Steady state NO_2 concentration $[\text{NO}_2]$ can be therefore easily calculated as $[\text{NO}_2] = k_{13} \cdot [\text{NO}] / k_{14}$, assuming balance between NO_2 production and destruction by reactions (13) and (14). In this formula, $[\text{NO}]$ is steady state concentration of NO, k_{13} and k_{14} are rate coefficients of reactions (13) and (14), respectively. At 2000 K, the ratio of these two rate coefficients is 7.3×10^{-4} and it explains why the steady state concentration of NO_2 is much lower than the concentration of NO (Figure 4).

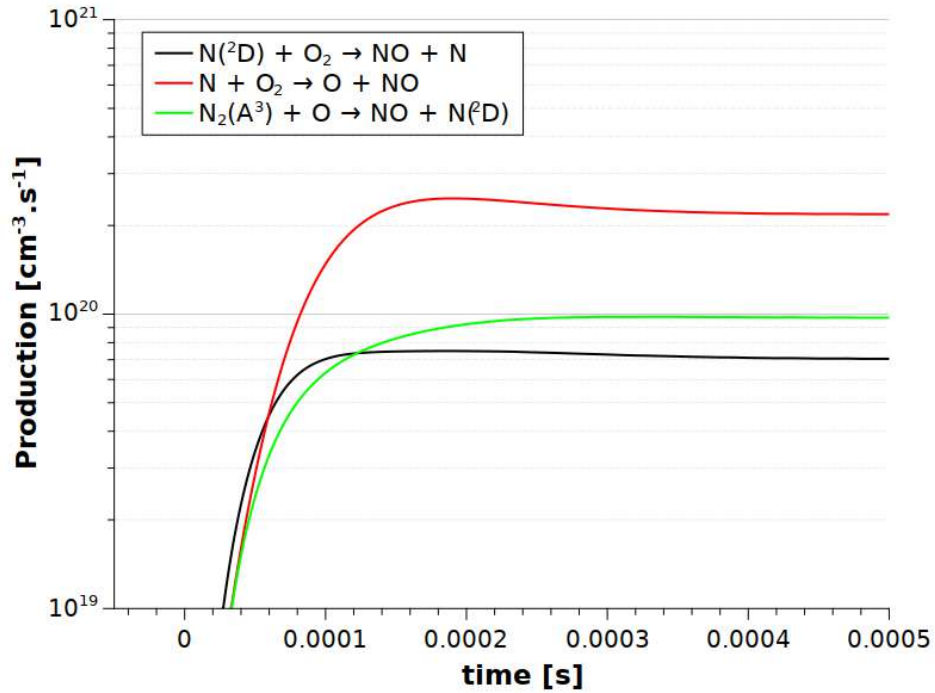


Fig. 6. Main reactions responsible for production of NO molecules; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

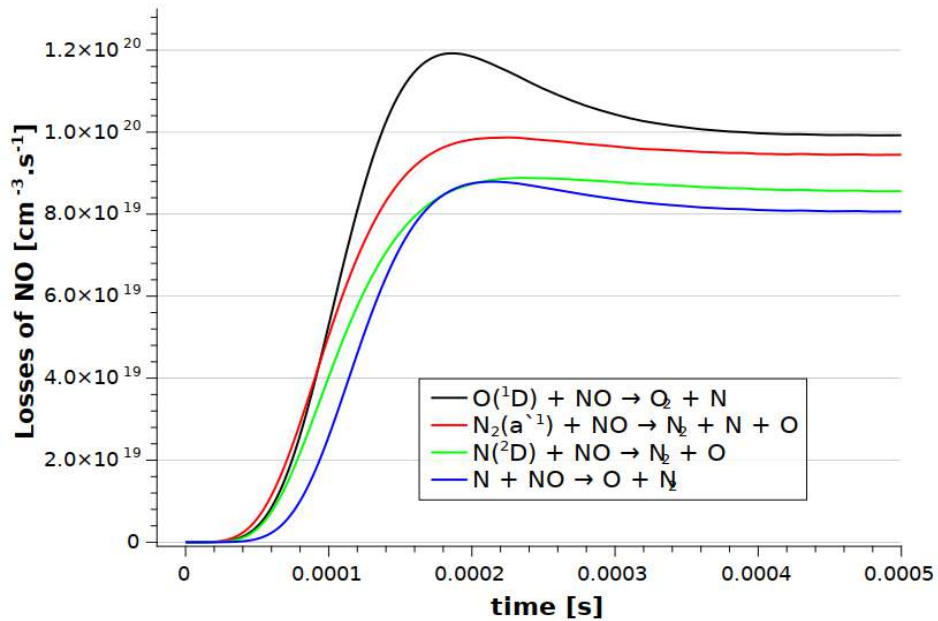


Fig. 7. Main reactions responsible for losses of NO molecules; calculated with $\alpha_{diff} = 2 \text{ s}^{-1}$.

Based on *ex-situ* measurements of the gas after treatment by glow discharge, the concentration of NO is higher than the concentration of NO₂, but the difference is not so significant [3]. Final NO and NO₂ concentration is mainly determined by reactions in the gas after leaving the discharge zone (afterglow). Figure 8 shows time evolution of gas temperature and N, O, NO and NO₂ species concentrations in the discharge afterglow, calculated with $\alpha_{diff} = 10 \text{ s}^{-1}$. The concentration of atomic species (N and O) decreases rapidly. While the NO concentration decreases slowly, the NO₂ concentration increases, despite mixing with the ambient air. Consequently, the difference between NO and NO₂ concentrations decreases in the discharge afterglow.

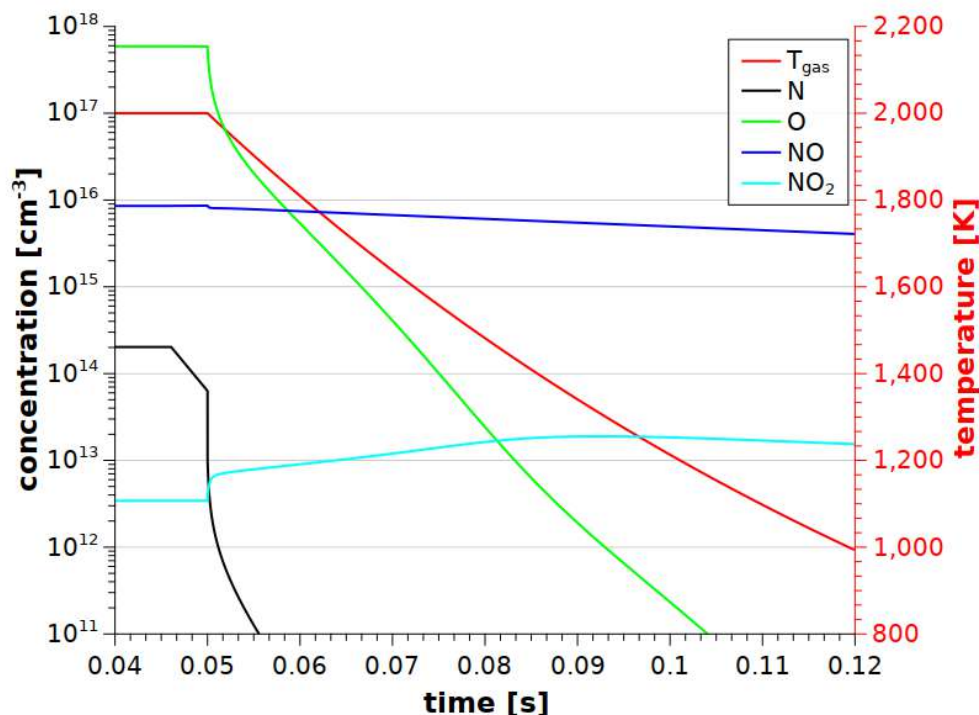


Fig. 8. Time evolution the gas temperature and N, O, NO and NO₂ species concentrations; calculated with $\alpha_{diff} = 10 \text{ s}^{-1}$.

A stable signal from ions directly generated by the HPGD was not detected by mass spectrometry. This can be attributed to two main factors. Firstly, the HPGD migrated on the surface of the orifice plate that served as anode, making its position relative to the spectrometer's gas entrance (a 100 μm orifice) highly variable. A stable ion signal could only be observed if the HPGD was positioned directly above this orifice, which occurred only randomly and occasionally. Secondly, even with the discharge positioned stably above the orifice, ion detection remains challenging. Positive ions are repelled from the anode, and the concentration of negative ions within the HPGD is relatively low, according to the kinetic model. The dominant negative ion, O⁻, has a concentration of approximately 10^{10} cm^{-3} , two orders of magnitude lower than the electron concentration. This low negative ion concentration may be due to the elevated gas temperature in the HPGD plasma channel.

5. Conclusions

Fixation of nitrogen from the air, converting it into reactive compounds, remains a significant challenge and a hot topic within the low-temperature plasma community. High-pressure glow discharges offer a stable and efficient means to generate nitrogen oxides, key precursors for nitrogen fixation. This study investigates the mechanisms of nitrogen oxide generation in an HPGD using a combined approach of mass spectrometry and chemical kinetic modeling.

Mass spectrometry provided insights into the types of reactive oxygen and nitrogen species produced, while the kinetic model simulated the complex chemical reactions within the plasma. Our findings shed light on the dominant reaction pathways on nitrogen oxide formation. However, further research is crucial to refine our understanding. Future experimental work should focus on improving the detection and quantification of reactive species, particularly ions. Model enhancements are also necessary, including a more accurate representation of diffusion processes and the discharge afterglow phase. Furthermore, incorporating additional chemical reactions involving water molecules will enable us to explore the crucial role of humidity in the generation of nitrogen oxides, nitric acid, and nitrous acid. This comprehensive approach promises to advance our knowledge of nitrogen fixation using HPGD

and pave the way for the development of efficient and sustainable plasma-based technologies for fertilizer production and other applications.

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MASS SPECTROMETRY OF DIELECTRIC BARRIER DISCHARGE WITH WATER ELECTRODE

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This paper presents the results of an investigation into the generation of reactive species in two setups of a dielectric barrier discharge (DBD) plasma source, using a water target with different vessels. By analyzing both neutral mass spectra and MID-scan spectra, we explore the concentrations of reactive oxygen and nitrogen species (NO, NO₂ and O₃) under varying plasma conditions and mass spectrometer configurations.

1. Introduction

In the past decade, a lot of interest has been drawn to atmospheric pressure plasmas (APPs) because of their unique properties and wide range of applications in fields such as material processing, agriculture, food industry and biomedicine [1, 2, 3, 4]. Since they do not require costly vacuum systems and operate at atmospheric pressure, APPs have the advantage of being accessible and versatile. Lately, atmospheric pressure plasma in contact with water has attracted significant interest due to its potential to generate reactive species and drive advanced chemical processes for various applications.

The behaviour and chemistry of APPs are greatly affected when they come into contact with water, either as an electrode or as a target. Reactive oxygen, nitrogen and hydrogen species are found in water and are essential for a variety of processes, such as biomedical treatments, sterilization, and water purification. Water forms a dynamic interface where plasma-induced reactions take place, producing reactive species like ozone (O₃), hydrogen peroxide (H₂O₂), hydroxyl radicals (OH), nitrates (NO₃⁻), nitrites (NO₂⁻) etc. These species play an essential role in enhancing the efficacy of plasma-based processes [5].

Also, higher humidity introduced in feeding gas of APPs has been demonstrated to increase the generation of reactive species, such as OH radicals, which are necessary for surface modification and decontamination [6]. The interaction, however, is complex and depends upon a number of variables, including ambient circumstances, water composition, and plasma characteristics. To optimize plasma processes and customize them for particular applications, it is essential to comprehend these interactions.

Dielectric barrier discharges (DBDs) are widely used for surface activation in atmospheric-pressure plasma applications. However, treating sensitive materials like polymers can be challenging because high-density plasma may cause damage, such as pin-holing. This issue often occurs in volume barrier discharges or coronas, where the plasma moves perpendicular to the treated surface. A practical solution is to generate plasma that travels parallel to the surface. This approach minimizes the risk of damage while maintaining effective treatment. One promising method is the surface dielectric barrier discharge (SDBD), where the plasma spreads along the surface of a dielectric plate. This setup not only protects the material but also improves the efficiency of the process.

In our earlier work [7, 8, 9] we introduced a novel plasma discharge reactor for efficiently activating polymers at the gas/liquid interface. This design uses liquid electrodes to ignite the SDBD directly from the liquid surface. Although the plasma-water interaction is limited to the edge of the dielectric tube, the system is both scalable and flexible, making it suitable for a wide range of applications.

Mass spectrometry is an analytical technique that measures the mass-to-charge ratio of ions to identify and quantify molecules in a sample. Primary advantage of atmospheric pressure mass spectrometry lies in its ability to rapidly and accurately analyse a wide range of chemical species [10]. These instruments are equipped with specialized pumping systems that create a pressure gradient, enabling the effective intake of gases from atmospheric plasmas. For neutral species, the mass spectrometer incorporates an ionization chamber that converts neutrals into ions, enabling their detection and analysis. The mass analyser which filters and detects neutral species or positive and negative ions, generating detailed mass spectra for all components. The technique provides real-time measurements of reactive species, ions, and neutral molecules, which are essential for understanding plasma processes and optimizing plasma-based applications.

When it comes to plasma in contact with water, due to technical challenges, mass spectrometric analysis of the plasma has so far been conducted by introducing water vapour into the working gas [11]. In this paper, we tackled the technical challenge and developed a setup where the mass spectrometer inlet was positioned in close proximity to the plasma-water interface, allowing us to successfully record mass spectra.

In this paper, we will present the results of our investigation into the reactive species generated by the dielectric barrier discharge (DBD) setup, but with two different water vessel configurations. The analysis includes both neutral mass spectra and MID-scan spectra, offering insights into the concentrations of key reactive oxygen and nitrogen species, such as NO, NO₂, CO₂ and O₃, as well as the detailed composition of the plasma obtained under these conditions.

2. Experimental set up

The schematic of the DBD at atmospheric pressure and HIDEN HPR60 mass-energy spectrometer is given in Figure 1. The DBD device is in the triple-phase interface (plasma-liquid-solid) plasma system consisting of a thin glass test tube with a 10 mm diameter and a 0.5 mm wall thickness was used. The liquid inside the test tube served as the high-voltage electrode and was connected to a power supply generating a sinusoidal voltage waveform. The Petri dish bath, which grounded the system, completed the circuit. Tap water with an electrical conductivity of approximately 0.3 mS/cm was used as the liquid electrode. The water was electrically insulated both inside and outside the test tube to ensure stability. The discharge operated in ambient air at atmospheric pressure. The high-voltage sine waveform had an amplitude range of 0 to 20 kV and could be adjusted to frequencies between 23 and 30 kHz. Power was delivered to the liquid electrodes through a high-voltage resonance generator (Lifetech-300W) paired with a function generator (FY3200S-24M).

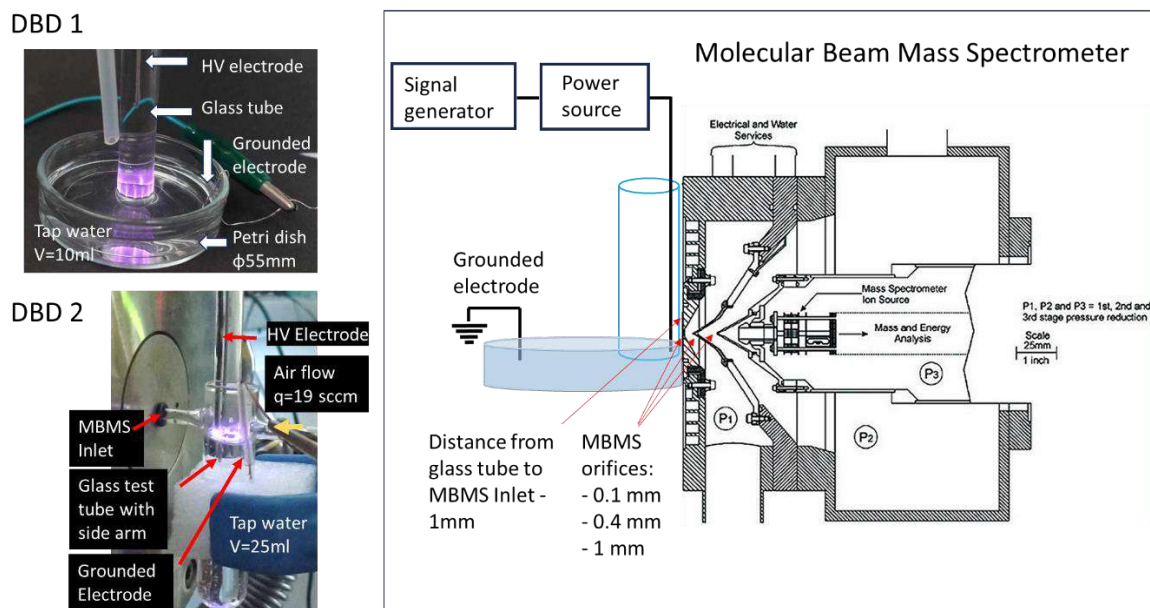


Fig. 1. DBD device in two configurations (DBD1 and DBD2) with a schematic representation of mass spectrometry measurements. In DBD1 setting, the device was immersed in 10ml of tap water placed in a Petri dish ($\phi 55\text{mm}$). In DBD2 setting, the device was placed in a glass test tube filled with 25ml of tap water, equipped with a side arm for gas sampling and a hole for synthetic air intake ($q = 19\text{ sccm}$).

Mass spectrometry measurements were performed by using MBMS (Molecular Beam Mass Spectrometer) HIDEN HPR60. To ensure the formation of a molecular beam, the geometry of the MBMS HPR60 system consists of a centralized combination of the orifice, cone1, and cone2 (P1 vacuum section is formed between the orifice and cone1, P2 vacuum section is formed between cone1 and cone2, and P3 vacuum section is formed after cone2 in the region of the mass analyzer). The orifice had an opening diameter of $\phi 0.1\text{ mm}$, cone1 $\phi 0.4\text{ mm}$, and cone2 $\phi 1\text{ mm}$, accompanied by the following voltages for DBD setups (the orifice was grounded, $V_{\text{cone1}} = 0\text{ V}$ and $V_{\text{cone2}} = 0\text{ V}$).

During all experiments for DBD setups, the regions within the vacuum section of the mass spectrometer responsible for generating the pressure gradient had the following pressure values: $P_1 = 3.3 \cdot 10^{-1}\text{ Torr}$, $P_2 = 7.5 \cdot 10^{-6}\text{ Torr}$, and $P_3 = 2.4 \cdot 10^{-7}\text{ Torr}$. To identify the species of interest, we first recorded the mass spectra of neutrals (0–100 amu) using the RGA (Residual Gas Analyzer) mode, during which the ionization chamber was active. Within the ionization chamber, the electron emission current from the filament was for DBD1 = $5\text{ }\mu\text{A}$ and for DBD2 = $10\text{ }\mu\text{A}$. In both cases, the electron energy was 70 eV.

After that, we used MID-scan to monitor the temporal changes of selected species for different formation conditions: without plasma, with plasma at specific applied powers for DBD1 (5W and 15W) and DBD2 (15W), with Swagelok open, and with swagelok closed. Swagelok open represents the sum of foreground and background species, while swagelok closed corresponds to background species only.

3. Results and discussion

In this study, we have measured the neutrals mass spectra by using a mass spectrometer for two different configurations of Dielectric Barrier Discharge (DBD) system. In both configurations, titled DBD1 and DBD2, discharge was in contact with water during mass spectrometry measurements. The analysis of neutral species was performed in two different measurement modes of HPR60: RGA mode for neutral mass spectra and MID-scan mode for track in time changes of specific neutral species. These modes provide a comprehensive overview of the ionization processes and chemical compositions

present in the plasma generated by each DBD system. The neutral mass spectra revealed the types and relative concentrations of neutral species, the MID-scan measurement provided insight into the temporal evolution of these species.

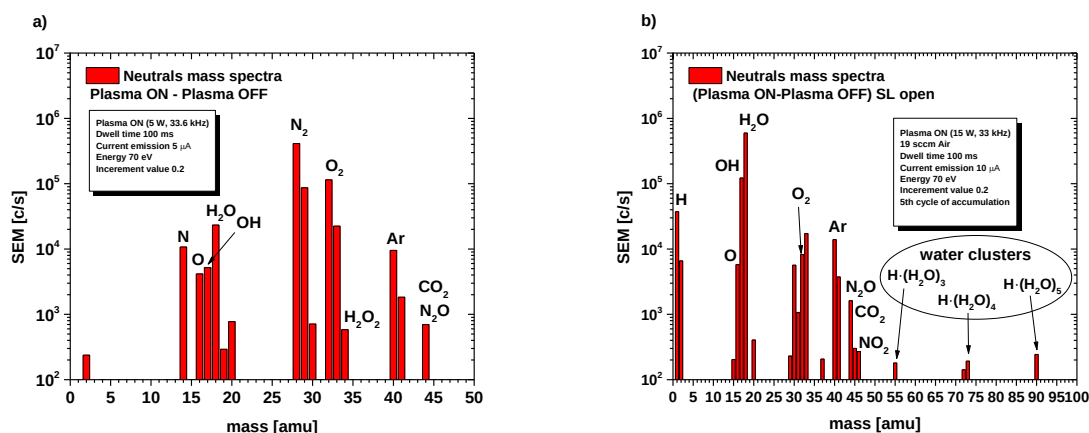


Fig. 2. Neutral mass spectra for a) DBD1 setting and b) DBD2 setting at an operating frequency of 33.6kHz. In the case of the DBD2, an airflow of 19sccm was added. The graphs were obtained by passing the raw results through a MatLab script to integrate the obtained lines for specific mass number.

Figure 2(a) and (b) present Neutral mass spectra for configurations DBD1 and DBD2, respectively. The presented spectra show the difference of Plasma ON mass spectra and Plasma OFF mass spectra. Plasma ON/OFF mass spectra represent the foreground signal i.e. the difference of total signal (swagelok open) and background signal (swagelok closed). The spectra in Figure 2 clearly show that the dominant species in the discharge are nitrogen and oxygen compounds (N, O₂, H₂O₂, N₂O, H, H₂O) which is to be expected because it is a discharge at atmospheric pressure where the target is water. Atomic nitrogen and atomic oxygen are present as a result of plasma reactions, along with the NO radical. The OH radical is also present, resulting from both plasma reactions and water dissociation in the MBMS. In Figure 2(b), water clusters can also be observed at mass numbers 53, 73 and 91, which appear in neutral mass spectra due to water vapor from the bottle containing the discharge. The high humidity in the DBD2 configuration and the plasma conditions promote cluster formation unlike in the case of DBD1 configuration where we did not detect any water clusters of mass above 50 amu.

While the mass spectra results provided an overall view of the main species present in the discharge chamber, nitrogen oxides and ozone can impact the industrial environment even at much lower concentrations (below the detection limit of the mass spectra used). Therefore, more sensitive measurements were conducted specifically for the important species NO, NO₂, N₂O, and O₃, as represented here. It was recorded for different conditions, Plasma ON and OFF with background only (BG), as well as Plasma ON and OFF with foreground and background (FG+BG). Unlike the case of BG, where only the inner part of the MS is considered in the measurement, in the case of FG+BG, the mass spectrometer is open so outside ambient air is also evaluated.

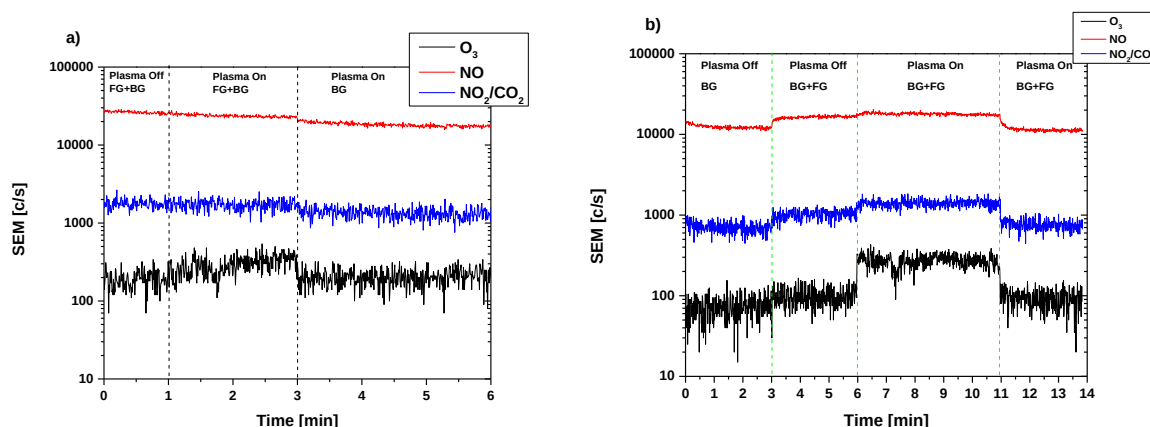


Fig. 3. The mid-scan provides constant tracking of selected species, with different stages of the experiment marked by labels: plasma on and off, foreground (FG) and background (BG); (a) DBD1 Setup, ionization filament current set to 5 μA at 70 eV, and (b) DBD2 Setup, ionization filament current set to 50 μA at 70 eV.

The MID-scan for DBD1 setup (Figure 3(a)) starts with Plasma OFF and swagelok open (1st minute), both the foreground (outside the spectrometer) and background are measured. The discharge is ignited in minute 1 and swagelok is open. Here we can see that NO continues to decrease slightly, NO₂ remains constant, and O₃ increases, indicating plasma-driven production of O₃. After the 3rd minute, with the plasma still on but swagelok closed, all species (NO, NO₂, and O₃) decrease as expected (only background inside device is measured).

The Figure 3(b) shows MID-scan spectra for DBD2 configuration. In the first three minutes, with the plasma off and the swagelok closed, only the background signal is measured. After opening of the swagelok (3-6 minutes), the increase in NO and NO₂ suggests the influence of ambient air, while O₃ remains unchanged. Between 6 and 11 minutes, with the plasma on and the swagelok open, NO and NO₂ rise slightly, but O₃ increases significantly, indicating plasma-driven O₃ production. Finally, from 11th minute, with the plasma on and the swagelok closed, all species decrease due to limited external interaction (only background is measured).

4. Conclusion

Mass spectrometry analysis of two setups of a DBD source with a water electrode, generated at atmospheric pressure was performed. Despite the difference in the water vessel used in each setup, in both setups similar trends in the behavior of reactive species were shown, indicating strong influence of plasma on water target. In both cases, plasma activation leads to the generation of reactive oxygen and nitrogen species, such as NO, NO₂, CO₂, and O₃, with notable increases in O₃ concentration when the plasma is on. Differences in the water vessel may affect the plasma's efficiency in producing reactive species, but both setups demonstrate that the plasma's interaction with the water is crucial for modulating the levels of reactive species. These findings highlight the importance of the plasma-water system in applications such as water treatment and agriculture, where reactive species generated in plasma-activated water could have significant biological and chemical effects.

Acknowledgments: The research was supported by the project of bilateral cooperation between Republic of Serbia and Republic of Slovakia 2024-2025 (project no. 337-00-3/2024-05/07), grant of the Ministry of Science, Technological Development and Innovations no. 451-03-68/2024-14/200024 and by the EU NextGenerationEU through the Recovery and Resilience Plan for Slovakia under the project No. 09I03-03-V04-00094.

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ISPC 24

24TH INTERNATIONAL SYMPOSIUM ON PLASMA CHEMISTRY
NAPLES (ITALY) JUNE 9-14, 2019

Final Program



ALMA MATER STUDIORUM
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TUESDAY June 11, 2019 (afternoon)			
	ROOM GALATEA	ROOM DIONE	ROOM PERSEIDE
14:00	Chairs Pietro Favia - Rony Snyders <i>Marc Böke</i> Separated effects of plasma species and post-treatment on the properties of barrier layers on polymers O-52 (4)	Chairs Anton Nikiforov - Alessandro Patelli <i>Bram Wolf</i> Elucidating the role of gas dynamics in the vortex-confined microwave plasma on CO₂ dissociation efficiency O-56 (2)	Chairs: Javad Mostaghimi - Gervais Soucy <i>Masasya Shigeta</i> To simulate turbulent thermal plasma flows for nanopowder fabrication I-12 (5)
14:15	<i>Annaëlle Demaude</i> Easy synthesis of hybrid hydrophilic-hydrophobic patterned surfaces by atmospheric plasmas O-53 (4)	<i>Georgi Trenchev</i> Atmospheric pressure glow discharge: design improvement based on modelling and experiments O-57 (2)	
14:30	<i>Huidong Hou</i> Effect of Precursor Chemistry on the microstructure of Ba(Mg₁/3Ta₂/3)O₃ during Hybrid Suspension/Solution Precursor Plasma Spraying O-54 (4)	<i>Françoise Massines</i> Dual frequency DBDs or how to design an atmospheric pressure plasma for surface treatment I-11 (2)	<i>Vittorio Colombo</i> Design-oriented modelling for the synthesis of Cu nanoparticles by a RF thermal plasma: impact of quenching solutions, radiative losses and thermophoresis O-60 (5)
14:45	<i>Shota Nunomura</i> Defect generation and annihilation in hydrogenated amorphous silicon during plasma treatment O-55 (4)		<i>Yasunori Tanaka</i> Effect of Alternating Gas Injection on Temperature Fields in Reaction Chamber using Inductively Coupled Thermal Plasmas for Nanoparticle Synthesis O-61 (5)
15:00	<i>Maria Adriana Creatore</i> Plasma-assisted atomic layer deposition of highly conductive HfNx layers I-10 (4)	<i>Javad Mostaghimi</i> Advantages of the New Conical Torch for ICP Spectrometry O-58 (2)	<i>Alexander Ustimenko</i> Plasma Treatment of Biomedical waste O-62 (5)
15:15		<i>Efe Kemaneci</i> A zero-dimensional modelling of a coaxial surface wave discharge in oxygen diluted with hexamethyldisiloxane for the deposition of SiOx films O-59 (2)	<i>Tae-Hee Kim</i> Reproduction of cosmic dust analogues by non-equilibrium condensation in triple DC thermal plasma jet system O-63 (5)
15:30	Refreshment break (30 min)		
	ROOM GALATEA		
16:00	Chair: Romolo Laurita		
	3 min poster pitches - Session II		
	POSTER AREA		
16:45	Parallel poster sessions (1-2-4-7-8)		
19:00			

Plasmas for plant bio-engineering and in agriculture for resource recovery

N. Puač¹, N. Škoro¹, S. Živković², M. Milutinović², S. Jevremović², O. Jovanović¹, G. Malović¹ and Z.Lj. Petrović^{1,3}

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Abstract: We have used different low-temperature atmospheric pressure plasmas for direct or indirect treatment of plant tissue. The plasma sources were characterised in detail by using optical emission spectroscopy, mass spectroscopy and electrical probes. The influence of the plasma treatments on the plant cells was investigated through enzyme response, development of calli and process of somatic embryogenesis. Also, the effect of plasma treated water and plasma decontaminated water was followed through the process of seed germination.

Keywords: plasma agriculture, plant calli, plasma decontamination.

1. Introduction

In the last decade the atmospheric pressure plasma systems have been extensively used in biomedical application [1-3]. Their construction and development was governed by the type of application and by the type of biological system that is treated. In parallel another field of plasma applications was growing – plasma agriculture. The first experiments were done with the low pressure nonequilibrium plasmas and primarily in treatments of seeds [4, 5]. Due to the fact that most seeds, plants, plants cells etc. cannot withstand vacuum, the devices that are nowadays mostly used are operating at atmospheric pressure. In such system, for example, the rich plasma chemistry changes the coat of the treated seed resulting in changes in wettability, better water uptake, increased percentage and speed of germination [6-8]. At the same time the pathogens that can be found on the seed coat and can be responsible for low germination percentage or contaminated plant are also destroyed during the plasma treatments. Therefore, unlike the classical methods involving different chemicals, plasma treatments can have a double or even multiple impact on the treated seeds.

As with the mammal cells, the interaction of the chemical species created in plasma and the triggered mechanisms are of the outmost importance. In the world of plants this can be done through investigation of the plasma treatments of the plant calli. In biological research and biotechnology the plant callus (pl. calli) is induced from plant tissue and it forms growing mass of plant parenchyma cells. The influence of the plasma created reactive oxygen and nitrogen species (RONS) and their interaction with calli can be investigated through the response of the cell enzymes. Puač et al. have shown that the interactions of the plasma created RONS with the plant cells can have even a long term influence [9].

Apart from the plasma treatments where plasma is in direct contact with seeds or plant cells like calli, there is a large group of experiments that investigate the influence of plasma treated water (plasma activated water – PAW) on these specimens. It was shown that PAW used for

imbibition of the different types of seeds increases germination percentage and on the molecular level changes the enzyme activity level.

Another very important topic is the possibility of re-usage of the water contaminated by agricultural means (pesticides, fertilization, biological farm waste etc.). Here we will present the results of applications of atmospheric pressure plasmas in direct treatment of plant seeds and calli or treatment of clean or contaminated water.

2. Application of atmospheric pressure plasmas in treatments of plant tissue and water used in agriculture

We have used several types of nonequilibrium plasma systems that operate at atmospheric pressure for direct treatments of plant calli, for treatment of water in order to produce PAW and for treatment of water contaminated with pesticides.



Fig. 1. *Daucus carota* callus

Pin geometry plasma sources used for these applications operate in the range of frequencies from kHz to MHz. These devices were used with helium as working gas and He gas flow was kept constant at 1 slm and 2 slm depending on the application. All plasma systems were characterised in detail by optical emission spectroscopy, mass spectroscopy and by using electrical probes.

The direct plasma treatment of plant calli was used to investigate the plasma cell interactions and to follow the response of the plant tissue several hours and days after the

treatment. Some of the samples that were used were taken from the model plants (like carrot cells – see Fig. 1.) and others from the plants that have an issue that needs to be overcome. For example callus of the yellow irises fall in the second group with its inability to enter the process of somatic embryogenesis (SE).

The direct plasma treatments of calli of *Daucus carota* were performed in order to investigate if the process of SE can be triggered even in the conditions where it is disabled. This means that the ratio of the growth hormones (in particular Auxin) in the culture medium is set to keep out the plant cells from going into SE. The results of such treatments are shown in Fig. 2. We can see that even in the conditions where SE cannot be initiated, plasma treatment triggered this process and the number of formed somatic embryos is higher with longer treatment time.

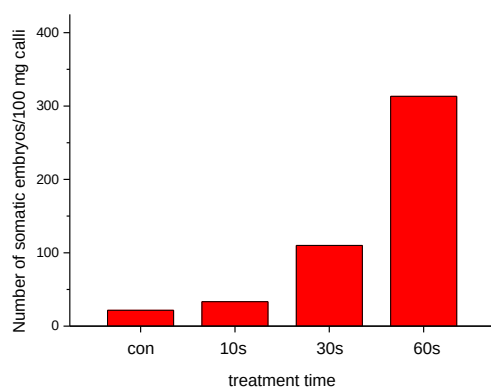


Fig. 2. Number of somatic embryos in *Daucus carota* callus for untreated and treated samples.

In case of water treatment, we have used clean distilled water and water contaminated by pesticides for creation of PAW. Both types of treated samples were then used for imbibition of seeds and investigation of effects on germination percentage and enzyme response. In order to determine the amount of active RONS species the PAW and decontaminated water samples were also characterised in detail by using spectrophotometry, total organic compound, dissolved oxygen, pH and conductivity measurements.

3. Conclusion

Nonequilibrium plasmas have shown a lot of potential in applications in biotechnology and agriculture. We have used several types of atmospheric plasma sources for treatments of plant calli or clean/contaminated water that was used for imbibition of seeds or watering of plants. All the plasma devices were characterised in great detail and optimised depending on the type of the sample and application. In the direct plasma treatment of plant calli it was determined that plasma species interacting with plant tissue can serve as a trigger for process of SE, even when this process is blocked or conditions unfavourable.

When used in treatments of water, RONS created in plasma and deposited in PAW increased the germination percentage of the imbibed seeds. Also, in the case when this water was contaminated by pesticides it was found that plasma treatments reduced harmful effect.

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Treatment of pesticide polluted water by atmospheric pressure plasma sources

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Abstract. In this work we present a method for purifying water contaminated with pesticides for further application in agriculture. For this purpose, a needle-type atmospheric pressure plasma jet (APPJ) was used, with electrode powered by a high voltage signal at frequency 330 kHz. We have used He as working gas with flow rate 2 slm. The solution of pesticide Malathion in distilled water was treated as contaminated sample. Optical emission spectroscopy was performed during liquid sample treatment. Pollutant concentrations in the samples were analysed and parameters of the chemical kinetics were obtained. Malathion destruction is described with the first order reaction. Then, the samples were used for radish seed imbibition. The results have shown that the plasma decontaminated water treatments have positive effect on germination of radish seeds.

1. Introduction

For more than a decade cold atmospheric pressure plasma have been successfully used in various types of biomedical treatments [1-3]. Studies performed within this field proved that non-equilibrium plasma is very versatile and efficient medium with complex dependence between its properties and controlling parameters as well as the interaction between plasma and treated surfaces. Research in the new field of plasma agriculture, which brings novel applications for APP, in the first place should study the key mechanisms underlying particular application in order to understand and control new processes [4]. One of the most important issues of modern agriculture is water pollution due to immense use of pesticides.

Thus, in our study we investigate application of cold atmospheric pressure plasma source for decontamination of water containing dissolved pesticide. Here, as an initial step, we present results of employment of atmospheric pressure plasma jet (APPJ) for the decontamination of aqueous solutions of pesticide Malathion.

2. Experimental

In Fig. 1 we show a schematic diagram of our experimental setup. Plasma source used for treatments is an atmosphere pressure plasma jet (APPJ) which consists of a metal cylindrical case, glass tube, with the inner and outer diameters of 4 mm and 6 mm, respectively, with

concentrically placed powered electrode. The electrode was made of stainless steel wire with the diameter of 1 mm. The end of the wire is sharpened and it is extending for 3 mm from the ceramic insulation into the glass tube. A commercial high voltage RF generator working at frequency of 330 kHz is used as a power source. In all treatments He was used as a working gas with flow rate of 2 slm. The experimental setup is similar to the one described in [5].

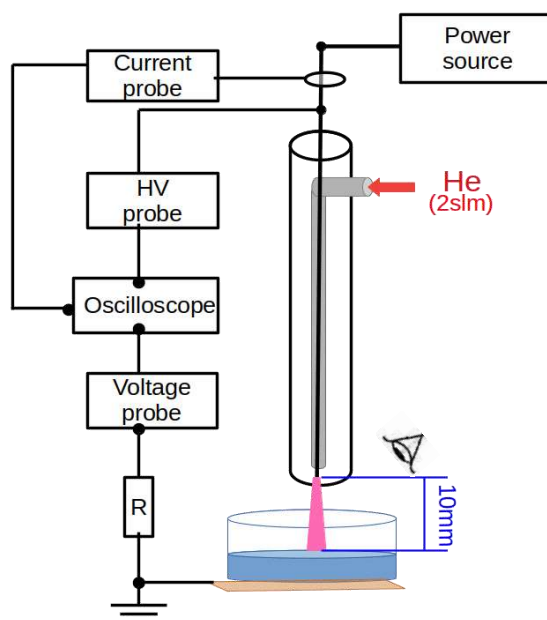


Fig. 1 Schematic overview of the experimental set up.

The samples containing Malathion were treated in the wells of 6-well microtiter plate. The volume of treated solution of Malathion in the well was 4 ml and initial concentration was 500 ppm in distilled water. Treatment times were 5 and 10 minutes. At the bottom of the micro titter plate copper tape is glued and connected to the ground through a 1 k Ω resistance for monitoring the current passing through the plasma. The distance between the surface of the liquid in the plate and wire was 10 mm. Optical emission spectroscopy is performed by using a UV-VIS lens positioned above the microtiter plate (as shown in Fig. 1), a fiber and spectrometer (Shamrock 750). Analysis of the liquid samples are accomplished in HPLC measurements, determining the area below the peak corresponding to Malathion.

3. Results and Discussion

In order to study the pollutant degradation process induced by plasma we performed characterization of plasma and liquid samples that are treated with the APPJ. Monitoring of the current through the plasma allowed us to continuously check the treatment conditions. Since we had the values of $I_{RMS} = 5 \mu A$ in all treatments, we had stable plasma conditions. To reveal what kind of excited species we produce in plasma, we employed optical emission spectroscopy measurements. The lens was positioned in such way that its viewing angle contained the plasma plume and the interface volume between plasma and the liquid surface.

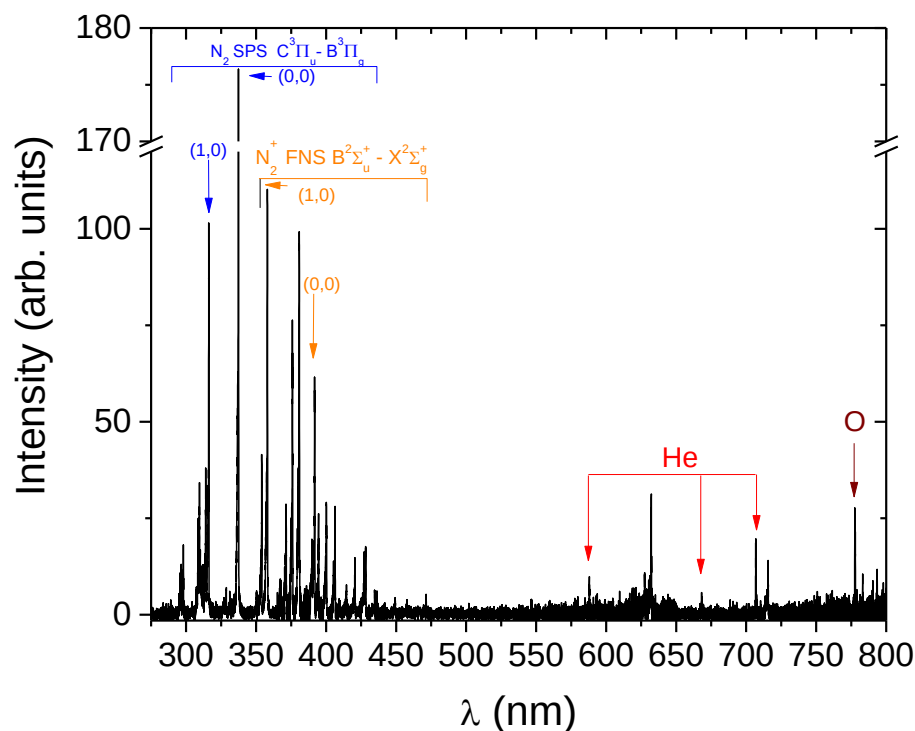


Fig. 2 Optical emission spectrum of APPJ with 2 slm of He as working gas recorded while operating against water sample contaminated with 500 ppm of Malathion.

In Fig. 2 we show emission spectrum taken at treatment conditions in wide range of wavelengths – from 290 nm to 800 nm. The spectrum is recorded in time-integrated manner with exposition time of 500 ms. Lines with the highest intensities are designated in the plot. Overall, the highest intensity lines belong to excited Nitrogen molecules, particularly the Second Positive System (SPS). Then, the lines of the First Negative System (FNS) of Nitrogen ion follow in the intensity scale. Strong Oxygen atom line at 777 nm is also visible suggesting the existence of high concentration of Oxygen atoms. These atoms are produced in the plasma is several dissociation channels [6]. Therefore, both atomic Oxygen species and probably some Nitrogen species are present in the plasma above the liquid [7]. Three He lines are also visible although their intensity is much lower in comparison to Nitrogen and Oxygen lines. Low-intensity OH (A-X) band (306-309 nm) can be also found in the spectrum. This band originates mostly from excited OH radicals formed after molecules of water vapour dissociation [8].

Fig. 3 shows data of measured Malathion decomposition in liquid samples treated by the APPJ. The initial concentration in these measurements were $C_0=10\text{ppm}$ and the evaluation of concentrations C after treatments of 1 min, 5 mins and 10 mins was performed from the chromatographic peak area of Malathion peak. In Fig.3b) the relative concentration values are plotted in semi-logarithmic scale and fitted with linear fit. Therefore, Malathion destruction process can be defined with the first order reaction with $R^2=0.989$. From the fit we obtained parameters of the chemical kinetics $k=0.5\text{ min}^{-1}$ and $T_{1/2}=1.4\text{ min}$.

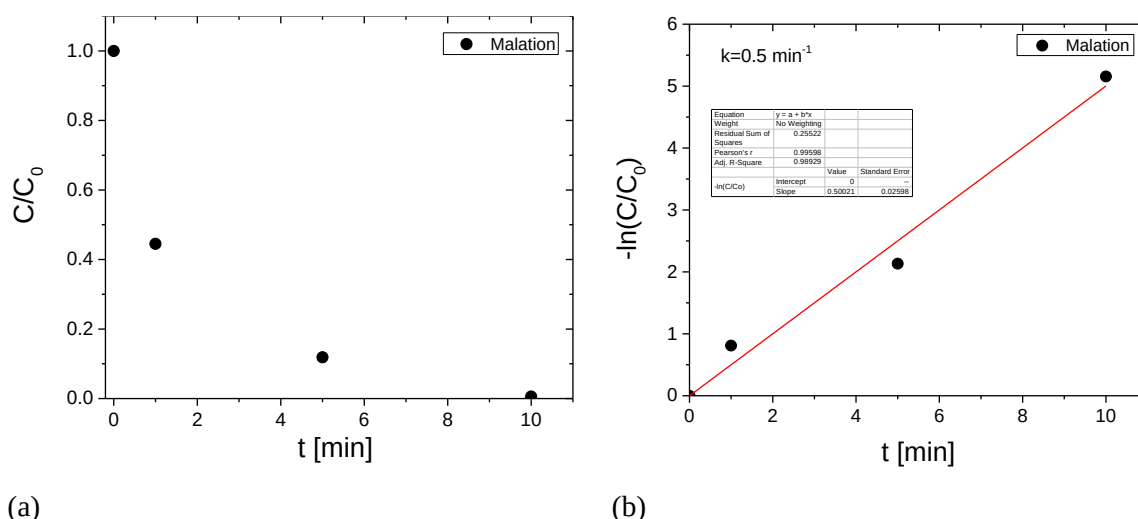


Fig. 3 Change in the relative concentration of Malathion after different treatment times: a) in linear scale of concentrations, b) in semi-logarithmic scale with linear fit (red line).

Treated contaminated water samples were used for imbibition of radish seeds. The 40 seeds were placed in Petri dishes and they were imbibed with 2 ml of liquid per Petri dish (5 cm in diameter). The dishes with the seeds were left in a room with constant temperature and with day-night cycle. For the control samples, we used seeds imbibed with distilled water and solution of Malathion (500ppm). All experiments were done in triplicate.

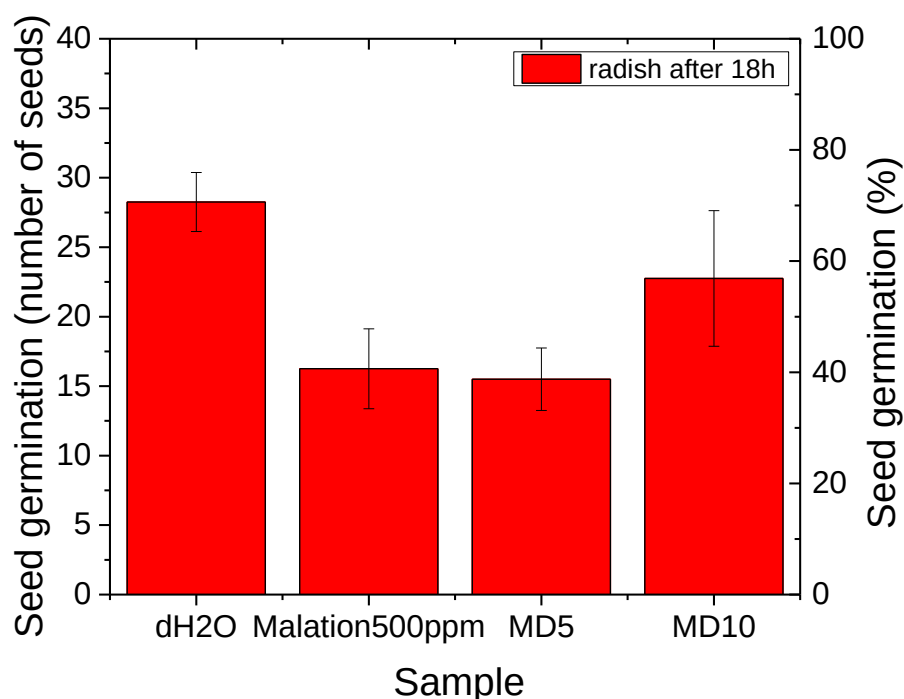


Fig. 4 Mean values of radish seeds germinated after 18 hours of imbibition with different water samples. dH2O – distilled water, distilled water contaminated with 500 ppm Malathion, MD5 – contaminated water treated 5 minutes, MD10 – contaminated water treated 10 minutes.

In Fig. 4 we show histograms of mean value of germinated radish seeds (left hand-side axis) and germination percent (right hand-side axis) 18h after seed imbibition. Comparison between control groups showed that the number of germinated seeds imbibed with distilled water is almost twice as higher than the number of seeds germinated after imbibition by Malathion solution. As for the seeds imbibed with plasma decontaminated water, we can see the positive effect of longer plasma treatment of water polluted by Malathion. More precise, the results on plasma treated samples show that in this case duration of treatment of water polluted by Malathion significantly affects germination. While the set imbibed with solution treated for 5 mins is almost the same as the Malathion sample with the germination below 40%, the sample treated for 10 mins has the germination of almost 60%, i.e. the value which is 1.5 times higher. However, even after 10 mins of plasma treatment these seeds still have germination lower than 70% which is the value obtained with seeds imbibed with distilled water. Nevertheless, it is clear that seeds imbibed with plasma treated samples have higher germination percent than the seeds imbibed with contaminated water.

4. Conclusion

We conducted a research aiming to explore the possibility of utilization of atmospheric pressure plasma for decontamination of water polluted with organic pollutants. As the first step, we choose to target pesticide Malathion dissolved in distilled water. The liquid samples were treated by Helium APPJ while performing electrical and optical diagnostics. The optical emission spectrum showed existence of nitrogen and oxygen reactive species and small concentrations of OH radicals. Measurements of pollutant concentrations before and after treatments showed that destruction process can be fitted with first order reaction. Finally, radish seeds were imbibed with plasma treated samples. Results of germination of seeds imbibed with plasma decontaminated water showed increase in number of germinated seeds compared to the case of seed imbibition with Malathion polluted water. This proves that plasma treatments can purify water and cause decomposition of pollutant molecules to the extent suitable for the plasma treated water to be reused for plant cultivation. However, in order to achieve efficient process, one need to investigate decomposition mechanisms triggered by plasma treatment and the influence of treated solution on seed physiological processes.

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Aug. 28 - Sep. 1, 2018, Belgrade, Serbia

CONTRIBUTED PAPERS &

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Serbian Academy
of Sciences and Arts

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2. EXPERIMENTAL SETUP

The Fig. 1 shows a schematic diagram of the experimental setup. Plasma source was an atmosphere pressure plasma jet which consists of a metal cylindrical body, glass tube, with the inner and outer diameters of 4 mm and 6 mm, respectively, and concentrically placed electrode with ceramic insulation. The electrode was made of stainless steel wire with the diameter of 1 mm. The end of the wire (powered electrode) is sharpened and it is protruding for 3 mm from the ceramic insulation into the glass tube. As power source we have used a commercial high voltage RF generator working at frequency of 330 kHz. In all experiments He was used as a working gas with flow rate of 2 slm.

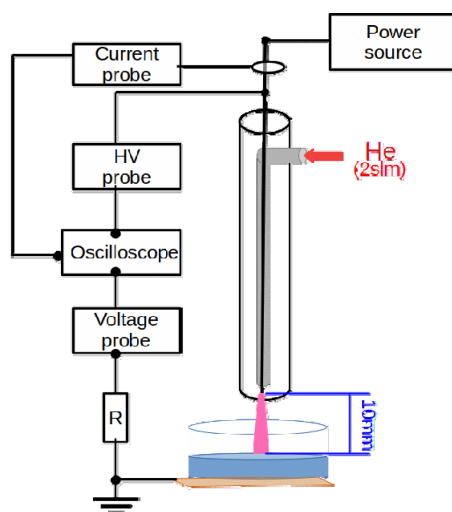


Figure 1. Schematic overview of the experimental set up.

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3. RESULTS AND DISCUSSION

Treated solutions as well as distilled water and Malathion contaminated solution were used for the imbibition of maize and radish seeds in Petri dishes, with 12 and 40 seeds, respectively in each dish. The seeds were imbibed with 2 ml of liquid per Petri dish (5 cm in diameter). The dishes with the seeds were

left in a room with constant temperature and with day-night cycle. All experiments were done in triplicate.

For the control samples, we used seeds imbibed with distilled water and solution of Malathion (500ppm). Histogram shown in Fig. 2 presents mean value of germinated maize seeds four days after imbibition. Comparison between control groups showed that number of germinated seeds imbibed with distilled water is more than twice higher than number of seeds germinated after imbibition by Malathion solution. As for the seeds imbibed with plasma decontaminated water, we can see the positive effect of plasma treatment of water polluted by Malathion. The number of germinated seeds watered with the solutions treated for 5 minutes and 10 minutes is higher 1.5 times than in the untreated Malathion solution. Mean values in both sets of seeds imbibed with decontaminated water are the same (around the value 5) within the error bar. Nevertheless, it is still lower than the control group imbibed with distilled water where the mean value is 7.3.

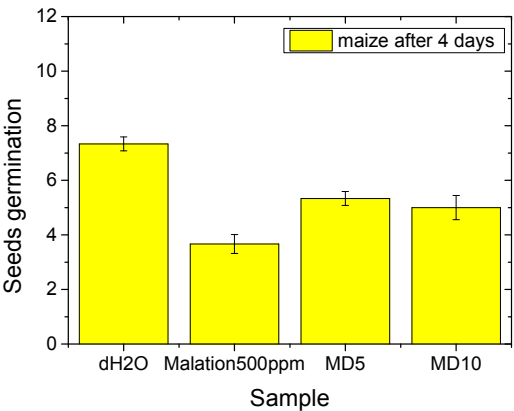


Figure 2. Mean values of maize seeds germinated after 4 days of imbibition with different water samples. dH₂O – distilled water, distilled water contaminated with 500 ppm Malathion, MD5 – contaminated water treated 5 minutes, MD10 – contaminated water treated 10 minutes.

Similar results are obtained in the case of germination of radish seeds. Fig. 3 shows mean values of germinated radish seeds 18 hours after imbibition. In this case, values in control groups with H₂O and 500ppm Malathion have difference of 1.75 times. However, the results on plasma treated samples show that in this case duration of treatment of water polluted by Malathion significantly affects germination. While the set imbibed with solution treated for 5 mins is almost the same as the Malathion sample (mean value around 15.5), the sample treated for 10 mins has the value which is 1.5 times higher.

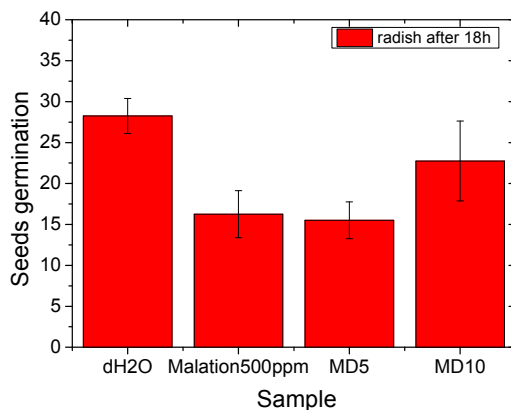


Figure 3. Mean values of radish seeds germinated after 18 hours of imbibition with different water samples. dH₂O – distilled water, distilled water contaminated with 500 ppm Malathion, MD5 – contaminated water treated 5 minutes, MD10 – contaminated water treated 10 minutes.

4. CONCLUSION

Results of germination of seeds imbibed with plasma decontaminated water showed increase in number of germinated seeds compared to the case of seed imbibition with Malathion polluted water. This proves that plasma treatments can purify water and cause decomposition of pollutant molecules to the extent suitable for the plasma treated water to be reused for plant cultivation. However, in order to achieve efficient process, one need to investigate decomposition mechanisms triggered by plasma treatment and the influence of treated solution on seed physiological processes.

Acknowledgements

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TUESDAY June 11, 2019 (afternoon)			
	ROOM GALATEA	ROOM DIONE	ROOM PERSEIDE
14:00	Chairs Pietro Favia - Rony Snyders <i>Marc Böke</i> Separated effects of plasma species and post-treatment on the properties of barrier layers on polymers O-52 (4)	Chairs Anton Nikiforov - Alessandro Patelli <i>Bram Wolf</i> Elucidating the role of gas dynamics in the vortex-confined microwave plasma on CO₂ dissociation efficiency O-56 (2)	Chairs: Javad Mostaghimi - Gervais Soucy <i>Masasya Shigeta</i> To simulate turbulent thermal plasma flows for nanopowder fabrication I-12 (5)
14:15	<i>Annaëlle Demaude</i> Easy synthesis of hybrid hydrophilic-hydrophobic patterned surfaces by atmospheric plasmas O-53 (4)	<i>Georgi Trenchev</i> Atmospheric pressure glow discharge: design improvement based on modelling and experiments O-57 (2)	
14:30	<i>Huidong Hou</i> Effect of Precursor Chemistry on the microstructure of Ba(Mg_{1/3}Ta_{2/3})O₃ during Hybrid Suspension/Solution Precursor Plasma Spraying O-54 (4)	<i>Françoise Massines</i> Dual frequency DBDs or how to design an atmospheric pressure plasma for surface treatment I-11 (2)	<i>Vittorio Colombo</i> Design-oriented modelling for the synthesis of Cu nanoparticles by a RF thermal plasma: impact of quenching solutions, radiative losses and thermophoresis O-60 (5)
14:45	<i>Shota Nunomura</i> Defect generation and annihilation in hydrogenated amorphous silicon during plasma treatment O-55 (4)		<i>Yasunori Tanaka</i> Effect of Alternating Gas Injection on Temperature Fields in Reaction Chamber using Inductively Coupled Thermal Plasmas for Nanoparticle Synthesis O-61 (5)
15:00	<i>Maria Adriana Creatore</i> Plasma-assisted atomic layer deposition of highly conductive HfNx layers I-10 (4)	<i>Javad Mostaghimi</i> Advantages of the New Conical Torch for ICP Spectrometry O-58 (2)	<i>Alexander Ustimenko</i> Plasma Treatment of Biomedical waste O-62 (5)
15:15		<i>Efe Kemaneci</i> A zero-dimensional modelling of a coaxial surface wave discharge in oxygen diluted with hexamethyldisiloxane for the deposition of SiOx films O-59 (2)	<i>Tae-Hee Kim</i> Reproduction of cosmic dust analogues by non-equilibrium condensation in triple DC thermal plasma jet system O-63 (5)
15:30	Refreshment break (30 min)		
	ROOM GALATEA		
16:00	Chair: Romolo Laurita		
	3 min poster pitches - Session II		
	POSTER AREA		
16:45	Parallel poster sessions (1-2-4-7-8)		
19:00			

Plasmas for plant bio-engineering and in agriculture for resource recovery

N. Puač¹, N. Škoro¹, S. Živković², M. Milutinović², S. Jevremović², O. Jovanović¹, G. Malović¹ and Z.Lj. Petrović^{1,3}

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Abstract: We have used different low-temperature atmospheric pressure plasmas for direct or indirect treatment of plant tissue. The plasma sources were characterised in detail by using optical emission spectroscopy, mass spectroscopy and electrical probes. The influence of the plasma treatments on the plant cells was investigated through enzyme response, development of calli and process of somatic embryogenesis. Also, the effect of plasma treated water and plasma decontaminated water was followed through the process of seed germination.

Keywords: plasma agriculture, plant calli, plasma decontamination.

1. Introduction

In the last decade the atmospheric pressure plasma systems have been extensively used in biomedical application [1-3]. Their construction and development was governed by the type of application and by the type of biological system that is treated. In parallel another field of plasma applications was growing – plasma agriculture. The first experiments were done with the low pressure nonequilibrium plasmas and primarily in treatments of seeds [4, 5]. Due to the fact that most seeds, plants, plants cells etc. cannot withstand vacuum, the devices that are nowadays mostly used are operating at atmospheric pressure. In such system, for example, the rich plasma chemistry changes the coat of the treated seed resulting in changes in wettability, better water uptake, increased percentage and speed of germination [6-8]. At the same time the pathogens that can be found on the seed coat and can be responsible for low germination percentage or contaminated plant are also destroyed during the plasma treatments. Therefore, unlike the classical methods involving different chemicals, plasma treatments can have a double or even multiple impact on the treated seeds.

As with the mammal cells, the interaction of the chemical species created in plasma and the triggered mechanisms are of the outmost importance. In the world of plants this can be done through investigation of the plasma treatments of the plant calli. In biological research and biotechnology the plant callus (pl. calli) is induced from plant tissue and it forms growing mass of plant parenchyma cells. The influence of the plasma created reactive oxygen and nitrogen species (RONS) and their interaction with calli can be investigated through the response of the cell enzymes. Puač et al. have shown that the interactions of the plasma created RONS with the plant cells can have even a long term influence [9].

Apart from the plasma treatments where plasma is in direct contact with seeds or plant cells like calli, there is a large group of experiments that investigate the influence of plasma treated water (plasma activated water – PAW) on these specimens. It was shown that PAW used for

imbibition of the different types of seeds increases germination percentage and on the molecular level changes the enzyme activity level.

Another very important topic is the possibility of re-usage of the water contaminated by agricultural means (pesticides, fertilization, biological farm waste etc.). Here we will present the results of applications of atmospheric pressure plasmas in direct treatment of plant seeds and calli or treatment of clean or contaminated water.

2. Application of atmospheric pressure plasmas in treatments of plant tissue and water used in agriculture

We have used several types of nonequilibrium plasma systems that operate at atmospheric pressure for direct treatments of plant calli, for treatment of water in order to produce PAW and for treatment of water contaminated with pesticides.



Fig. 1. *Daucus carota* callus

Pin geometry plasma sources used for these applications operate in the range of frequencies from kHz to MHz. These devices were used with helium as working gas and He gas flow was kept constant at 1 slm and 2 slm depending on the application. All plasma systems were characterised in detail by optical emission spectroscopy, mass spectroscopy and by using electrical probes.

The direct plasma treatment of plant calli was used to investigate the plasma cell interactions and to follow the response of the plant tissue several hours and days after the

treatment. Some of the samples that were used were taken from the model plants (like carrot cells – see Fig. 1.) and others from the plants that have an issue that needs to be overcome. For example callus of the yellow irises fall in the second group with its inability to enter the process of somatic embryogenesis (SE).

The direct plasma treatments of calli of *Daucus carota* were performed in order to investigate if the process of SE can be triggered even in the conditions where it is disabled. This means that the ratio of the growth hormones (in particular Auxin) in the culture medium is set to keep out the plant cells from going into SE. The results of such treatments are shown in Fig. 2. We can see that even in the conditions where SE cannot be initiated, plasma treatment triggered this process and the number of formed somatic embryos is higher with longer treatment time.

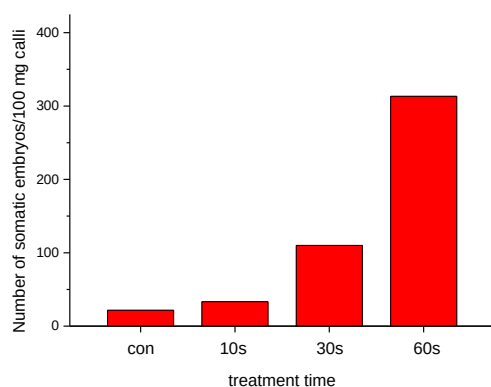


Fig. 2. Number of somatic embryos in *Daucus carota* callus for untreated and treated samples.

In case of water treatment, we have used clean distilled water and water contaminated by pesticides for creation of PAW. Both types of treated samples were then used for imbibition of seeds and investigation of effects on germination percentage and enzyme response. In order to determine the amount of active RONS species the PAW and decontaminated water samples were also characterised in detail by using spectrophotometry, total organic compound, dissolved oxygen, pH and conductivity measurements.

3. Conclusion

Nonequilibrium plasmas have shown a lot of potential in applications in biotechnology and agriculture. We have used several types of atmospheric plasma sources for treatments of plant calli or clean/contaminated water that was used for imbibition of seeds or watering of plants. All the plasma devices were characterised in great detail and optimised depending on the type of the sample and application. In the direct plasma treatment of plant calli it was determined that plasma species interacting with plant tissue can serve as a trigger for process of SE, even when this process is blocked or conditions unfavourable.

When used in treatments of water, RONS created in plasma and deposited in PAW increased the germination percentage of the imbibed seeds. Also, in the case when this water was contaminated by pesticides it was found that plasma treatments reduced harmful effect.

Acknowledgments: Supported by MESTDRS III41011 and ON171037.

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Treatment of pesticide polluted water by atmospheric pressure plasma sources

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Abstract. In this work we present a method for purifying water contaminated with pesticides for further application in agriculture. For this purpose, a needle-type atmospheric pressure plasma jet (APPJ) was used, with electrode powered by a high voltage signal at frequency 330 kHz. We have used He as working gas with flow rate 2 slm. The solution of pesticide Malathion in distilled water was treated as contaminated sample. Optical emission spectroscopy was performed during liquid sample treatment. Pollutant concentrations in the samples were analysed and parameters of the chemical kinetics were obtained. Malathion destruction is described with the first order reaction. Then, the samples were used for radish seed imbibition. The results have shown that the plasma decontaminated water treatments have positive effect on germination of radish seeds.

1. Introduction

For more than a decade cold atmospheric pressure plasma have been successfully used in various types of biomedical treatments [1-3]. Studies performed within this field proved that non-equilibrium plasma is very versatile and efficient medium with complex dependence between its properties and controlling parameters as well as the interaction between plasma and treated surfaces. Research in the new field of plasma agriculture, which brings novel applications for APP, in the first place should study the key mechanisms underlying particular application in order to understand and control new processes [4]. One of the most important issues of modern agriculture is water pollution due to immense use of pesticides.

Thus, in our study we investigate application of cold atmospheric pressure plasma source for decontamination of water containing dissolved pesticide. Here, as an initial step, we present results of employment of atmospheric pressure plasma jet (APPJ) for the decontamination of aqueous solutions of pesticide Malathion.

2. Experimental

In Fig. 1 we show a schematic diagram of our experimental setup. Plasma source used for treatments is an atmosphere pressure plasma jet (APPJ) which consists of a metal cylindrical case, glass tube, with the inner and outer diameters of 4 mm and 6 mm, respectively, with

concentrically placed powered electrode. The electrode was made of stainless steel wire with the diameter of 1 mm. The end of the wire is sharpened and it is extending for 3 mm from the ceramic insulation into the glass tube. A commercial high voltage RF generator working at frequency of 330 kHz is used as a power source. In all treatments He was used as a working gas with flow rate of 2 slm. The experimental setup is similar to the one described in [5].

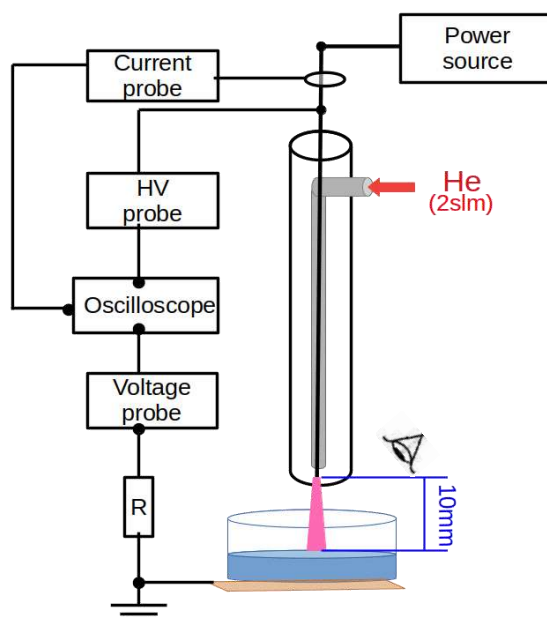


Fig. 1 Schematic overview of the experimental set up.

The samples containing Malathion were treated in the wells of 6-well microtiter plate. The volume of treated solution of Malathion in the well was 4 ml and initial concentration was 500 ppm in distilled water. Treatment times were 5 and 10 minutes. At the bottom of the micro titter plate copper tape is glued and connected to the ground through a 1 k Ω resistance for monitoring the current passing through the plasma. The distance between the surface of the liquid in the plate and wire was 10 mm. Optical emission spectroscopy is performed by using a UV-VIS lens positioned above the microtiter plate (as shown in Fig. 1), a fiber and spectrometer (Shamrock 750). Analysis of the liquid samples are accomplished in HPLC measurements, determining the area below the peak corresponding to Malathion.

3. Results and Discussion

In order to study the pollutant degradation process induced by plasma we performed characterization of plasma and liquid samples that are treated with the APPJ. Monitoring of the current through the plasma allowed us to continuously check the treatment conditions. Since we had the values of $I_{RMS} = 5 \mu A$ in all treatments, we had stable plasma conditions. To reveal what kind of excited species we produce in plasma, we employed optical emission spectroscopy measurements. The lens was positioned in such way that its viewing angle contained the plasma plume and the interface volume between plasma and the liquid surface.

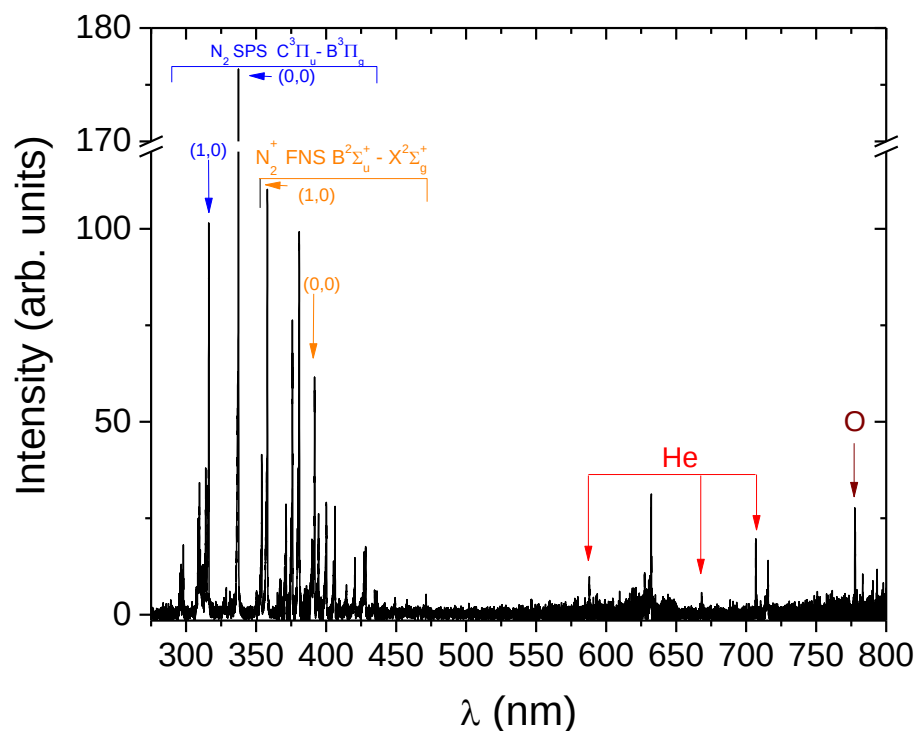


Fig. 2 Optical emission spectrum of APPJ with 2 slm of He as working gas recorded while operating against water sample contaminated with 500 ppm of Malathion.

In Fig. 2 we show emission spectrum taken at treatment conditions in wide range of wavelengths – from 290 nm to 800 nm. The spectrum is recorded in time-integrated manner with exposition time of 500 ms. Lines with the highest intensities are designated in the plot. Overall, the highest intensity lines belong to excited Nitrogen molecules, particularly the Second Positive System (SPS). Then, the lines of the First Negative System (FNS) of Nitrogen ion follow in the intensity scale. Strong Oxygen atom line at 777 nm is also visible suggesting the existence of high concentration of Oxygen atoms. These atoms are produced in the plasma is several dissociation channels [6]. Therefore, both atomic Oxygen species and probably some Nitrogen species are present in the plasma above the liquid [7]. Three He lines are also visible although their intensity is much lower in comparison to Nitrogen and Oxygen lines. Low-intensity OH (A-X) band (306-309 nm) can be also found in the spectrum. This band originates mostly from excited OH radicals formed after molecules of water vapour dissociation [8].

Fig. 3 shows data of measured Malathion decomposition in liquid samples treated by the APPJ. The initial concentration in these measurements were $C_0=10\text{ppm}$ and the evaluation of concentrations C after treatments of 1 min, 5 mins and 10 mins was performed from the chromatographic peak area of Malathion peak. In Fig.3b) the relative concentration values are plotted in semi-logarithmic scale and fitted with linear fit. Therefore, Malathion destruction process can be defined with the first order reaction with $R^2=0.989$. From the fit we obtained parameters of the chemical kinetics $k=0.5\text{ min}^{-1}$ and $T_{1/2}=1.4\text{ min}$.

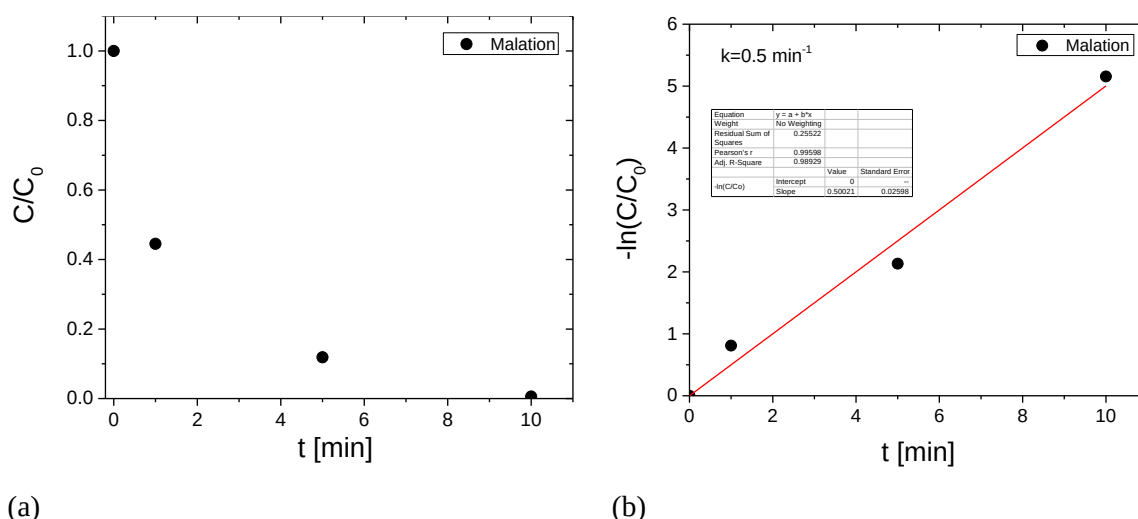


Fig. 3 Change in the relative concentration of Malathion after different treatment times: a) in linear scale of concentrations, b) in semi-logarithmic scale with linear fit (red line).

Treated contaminated water samples were used for imbibition of radish seeds. The 40 seeds were placed in Petri dishes and they were imbibed with 2 ml of liquid per Petri dish (5 cm in diameter). The dishes with the seeds were left in a room with constant temperature and with day-night cycle. For the control samples, we used seeds imbibed with distilled water and solution of Malathion (500ppm). All experiments were done in triplicate.

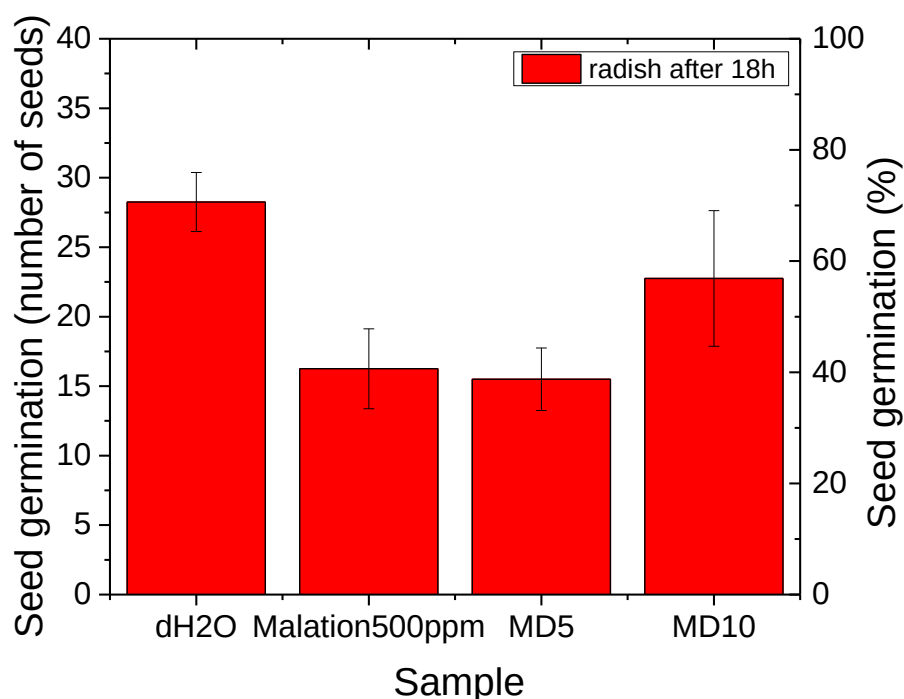


Fig. 4 Mean values of radish seeds germinated after 18 hours of imbibition with different water samples. dH2O – distilled water, distilled water contaminated with 500 ppm Malathion, MD5 – contaminated water treated 5 minutes, MD10 – contaminated water treated 10 minutes.

In Fig. 4 we show histograms of mean value of germinated radish seeds (left hand-side axis) and germination percent (right hand-side axis) 18h after seed imbibition. Comparison between control groups showed that the number of germinated seeds imbibed with distilled water is almost twice as higher than the number of seeds germinated after imbibition by Malathion solution. As for the seeds imbibed with plasma decontaminated water, we can see the positive effect of longer plasma treatment of water polluted by Malathion. More precise, the results on plasma treated samples show that in this case duration of treatment of water polluted by Malathion significantly affects germination. While the set imbibed with solution treated for 5 mins is almost the same as the Malathion sample with the germination below 40%, the sample treated for 10 mins has the germination of almost 60%, i.e. the value which is 1.5 times higher. However, even after 10 mins of plasma treatment these seeds still have germination lower than 70% which is the value obtained with seeds imbibed with distilled water. Nevertheless, it is clear that seeds imbibed with plasma treated samples have higher germination percent than the seeds imbibed with contaminated water.

4. Conclusion

We conducted a research aiming to explore the possibility of utilization of atmospheric pressure plasma for decontamination of water polluted with organic pollutants. As the first step, we choose to target pesticide Malathion dissolved in distilled water. The liquid samples were treated by Helium APPJ while performing electrical and optical diagnostics. The optical emission spectrum showed existence of nitrogen and oxygen reactive species and small concentrations of OH radicals. Measurements of pollutant concentrations before and after treatments showed that destruction process can be fitted with first order reaction. Finally, radish seeds were imbibed with plasma treated samples. Results of germination of seeds imbibed with plasma decontaminated water showed increase in number of germinated seeds compared to the case of seed imbibition with Malathion polluted water. This proves that plasma treatments can purify water and cause decomposition of pollutant molecules to the extent suitable for the plasma treated water to be reused for plant cultivation. However, in order to achieve efficient process, one need to investigate decomposition mechanisms triggered by plasma treatment and the influence of treated solution on seed physiological processes.

Acknowledgments

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Vinča Institute of
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Serbian Academy
of Sciences and Arts

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2. EXPERIMENTAL SETUP

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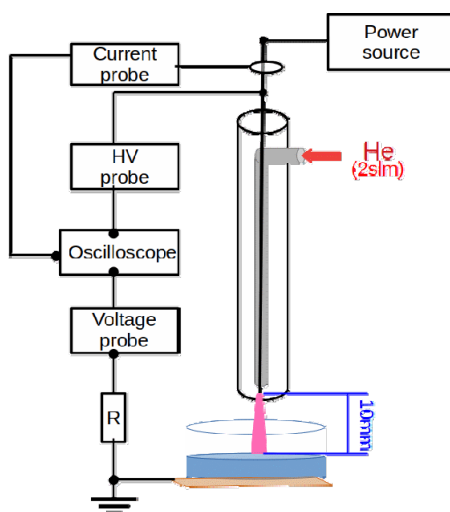


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3. RESULTS AND DISCUSSION

Treated solutions as well as distilled water and Malathion contaminated solution were used for the imbibition of maize and radish seeds in Petri dishes, with 12 and 40 seeds, respectively in each dish. The seeds were imbibed with 2 ml of liquid per Petri dish (5 cm in diameter). The dishes with the seeds were

left in a room with constant temperature and with day-night cycle. All experiments were done in triplicate.

For the control samples, we used seeds imbibed with distilled water and solution of Malathion (500ppm). Histogram shown in Fig. 2 presents mean value of germinated maize seeds four days after imbibition. Comparison between control groups showed that number of germinated seeds imbibed with distilled water is more than twice higher than number of seeds germinated after imbibition by Malathion solution. As for the seeds imbibed with plasma decontaminated water, we can see the positive effect of plasma treatment of water polluted by Malathion. The number of germinated seeds watered with the solutions treated for 5 minutes and 10 minutes is higher 1.5 times than in the untreated Malathion solution. Mean values in both sets of seeds imbibed with decontaminated water are the same (around the value 5) within the error bar. Nevertheless, it is still lower than the control group imbibed with distilled water where the mean value is 7.3.

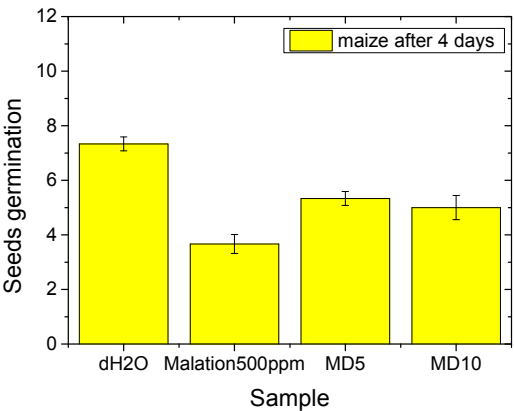


Figure 2. Mean values of maize seeds germinated after 4 days of imbibition with different water samples. dH₂O – distilled water, distilled water contaminated with 500 ppm Malathion, MD5 – contaminated water treated 5 minutes, MD10 – contaminated water treated 10 minutes.

Similar results are obtained in the case of germination of radish seeds. Fig. 3 shows mean values of germinated radish seeds 18 hours after imbibition. In this case, values in control groups with H₂O and 500ppm Malathion have difference of 1.75 times. However, the results on plasma treated samples show that in this case duration of treatment of water polluted by Malathion significantly affects germination. While the set imbibed with solution treated for 5 mins is almost the same as the Malathion sample (mean value around 15.5), the sample treated for 10 mins has the value which is 1.5 times higher.

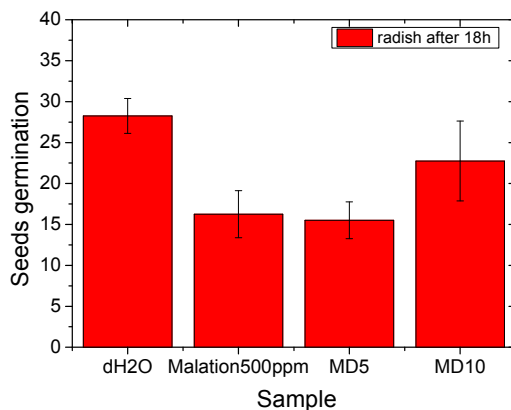


Figure 3. Mean values of radish seeds germinated after 18 hours of imbibition with different water samples. dH₂O – distilled water, distilled water contaminated with 500 ppm Malathion, MD5 – contaminated water treated 5 minutes, MD10 – contaminated water treated 10 minutes.

4. CONCLUSION

Results of germination of seeds imbibed with plasma decontaminated water showed increase in number of germinated seeds compared to the case of seed imbibition with Malathion polluted water. This proves that plasma treatments can purify water and cause decomposition of pollutant molecules to the extent suitable for the plasma treated water to be reused for plant cultivation. However, in order to achieve efficient process, one need to investigate decomposition mechanisms triggered by plasma treatment and the influence of treated solution on seed physiological processes.

Acknowledgements

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Atmospheric Pressure Plasma Treatment and Decontamination of Water Samples (invited)

N. Skoro, N. Puac, A. Kumar, O. Jovanovic, A. Petrovic (Institute of Physics Belgrade), U. Cvelbar (Jozef Stefan Institute), and Z. L. Petrovic (Serbian Academy for Sciences and Arts, School of Engineering, Ulster University)

Abstract Text:

As water scarcity becomes widespread concern in many countries, new approaches for water processing are being considered. In order to have the potential to be applied in the industry, these new methods should be primarily environmentally friendly and with low-energy consumption. Employment of non-equilibrium plasma for water treatment entirely satisfies these two points and therefore makes plasma a good candidate. Non-equilibrium (or cold) plasma at atmospheric pressure has so far been successfully used in various applications related to biology and medicine. Plasma rich-chemistry environment produced at room atmosphere with ambient temperature enabled treatment of cells, plants, bacteria, tissues etc. with aims ranging from destruction of pathogens to healing or enhancing the growth in biological systems. Therefore, these applications clearly point out that plasma sources can deliver broad spectrum of treatment conditions.

The latest research direction related to plasma and liquid treatment is plasma agriculture where different approaches for application of plasma in agricultural processes are investigated. Within these novel research area we investigate two possible applications: for the treatment of clean water samples for the production of plasma activated water (PAW); and for decontamination of polluted water containing organic micro pollutants (OMPs). PAW in comparison to clean water is rich in reactive oxygen and nitrogen species (RONS) and this proves to be an important asset when applied to seeds and plants. We showed that germination and plant growth can be enhanced when PAW is applied for seed imbibition and plant watering. On the other hand, the presence of reactive oxygen species in plasma and inside the treated water samples (especially droplets or aerosols) induces the decomposition of different molecules of OMPs. We demonstrated ability of plasma decontamination for several different organic molecules, such as decomposition of Malathion in atmospheric pressure plasma jet with high efficiency, and also for some chemicals used as chemical warfare surrogates. Apart from direct treatment of polluted samples by plasma, we have investigated a possibility of using PAW as a decontamination chemical to decompose organic dye Acid Blue 25 dissolved in water. Along the line of plasma agriculture research we combined the effects of two plasma applications in the plasma processing of agricultural waste water for reuse in irrigation. The experimental results showed that pesticide-containing polluted water can be decontaminated and at the same time enriched with RONS that positively influence germination of maize and radish.

These successful applications unfold many questions related to the plasma properties, plasma gas and liquid phase chemical reactions, an entangled connection between plasma produced reactive species and their chemical interactions with organic molecules in water. In order to make a detailed insight into the treatment process, we performed comprehensive diagnostics of the plasma and of the liquid samples. Our results provide information about electrical and optical plasma properties and make connection with physical parameters of the liquid sample (pH, electrical conductivity). We used colorimetric methods to determine RONS in the treated liquids. To acquire information about plasma-induced decomposition and obtain by-products in the treated samples we performed liquid chromatography coupled with mass spectrometry. All these results provide important knowledge on plasma water treatments and represent another step towards understanding which are the best parameters for treatment monitoring and techniques for up-scaling plasma devices to treat large amounts of water.

Acknowledgement: This study was partially supported by NATO SPS 984555, projects ON141037 and III 41011 of MESTD Republic of Serbia and H2020-MSCA-ITN 812880 Nowelties.

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- 08P-17 Initial Growth Kinetics of Hydrogenated Amorphous Carbon Films Observed by Real-Time Ellipsometry**
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The effects of plasma discharge regime on production of reactive species in water

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1. Introduction

Knowledge of the composition of active species generated in low temperature plasmas is of crucial importance for new biomedical and chemical applications [1]. Plasma activated water (PAW) is a major area of interest within the field of plasma agriculture. A number of researchers have reported that PAW, containing mainly reactive species, have wide variety of applications such as increasing the germination percentage of treated seeds, decontamination and sterilization of food products etc. PAW can also be an ecological alternative method for fertilization, decontamination and modification of soil [2].

Here we will present the electrical characterization and optical emission spectroscopy (OES) of an atmospheric pressure plasma jet (APPJ) that can operate in two different discharge regimes and it is employed for treatments of water samples and PAW production.

2. Experimental Setup

Plasma treatments of distilled water were carried out using an APPJ in a pin-electrode configuration with different flow rates of He as working gas.

The water samples were placed below APPJ powered by a continuous kHz sine signal and treated for 5, 10 and 20 min. OES of the plasma above the sample surface was performed during water treatment. To assess concentrations of long lived reactive species formed in the liquid phase we used a colorimetric method.

3. Results and Discussion

Fig. 1. presents concentrations of long lived reactive species existing in PAW after 20 min

treatment. In case of helium flow of 1 slm plasma plume was formed in air above the water sample (histogram left side). The right side of histogram shows results for regime when plasma was in contact with water (He 2slm). As can be seen from the Fig. 1. there was a significant difference in hydrogen peroxide concentrations, but almost same amount of nitrogen reactive species was acquired.

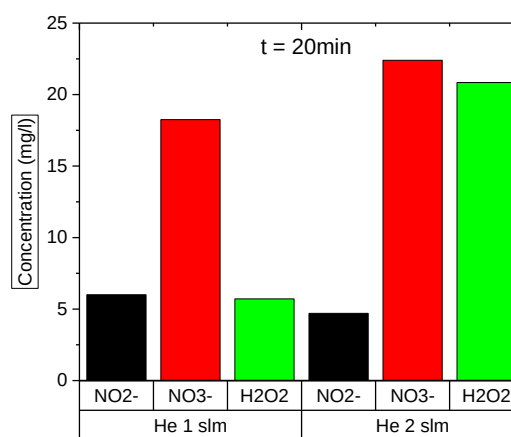


Fig. 1 Concentrations of hydrogen peroxide, nitrate and nitrite in PAW samples treated for 20 minutes for two different He flow rates.

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About MD-GAS

Emerging highly advanced ion-beam traps and storage rings combined with synchrotrons, X-ray facilities, and high performance computers offer completely new ways to study Molecular Dynamics in the GAS phase (MD-GAS). Cryogenic traps and rings will allow studies of decay and reaction processes involving molecular ions in well-defined conformations and in single or narrow ranges of quantum states.

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09:40-10:00	<i>High radiative cooling rates of small clusters</i>	P. Ferrari
10:00-10:20	<i>The stability of the smallest carbon cluster dianion: C_7^{2-}</i>	P. Najeeb
10:20-11:00	Coffee break	
11:00-11:30	Working Group 3 Kick off Meeting - M. Alcamí	
11:30-12:00	<i>Ion-collision induced reactivity in molecular clusters</i>	P. Rousseau
12:00-12:30	<i>Gas-phase molecules through the lens of time-resolved photoelectron spectroscopy</i>	A. Ponzi
12:30-13:00	<i>Interaction of low energy electrons with biomolecules and clusters of biomolecules</i>	J. Kocisek
13:00-14:30	Lunch at the GANIL restaurant	
14:30-15:00	<i>Highly charged helium nanodroplets</i>	M. Gatchell
15:00-15:30	<i>Creation and destruction of chemical species in liquids treated by atmospheric pressure plasmas - from gas phase chemistry to bulk liquid</i>	N. Skoro
15:30-16:00	<i>Resonant Inelastic X-ray scattering of chloromethanes</i>	M. Zitnik
16:00-16:30	<i>Elastic electron scattering on molecules in the gas phase in the middle energy range</i>	J. Maljković
16:30-17:30	Coffee break	
16:30-17:30	Laboratory visit	
19:00-21:30	Conference diner - Café Mancel, le Château Ducal, Caen	

CREATION AND DESTRUCTION OF CHEMICAL SPECIES IN LIQUIDS TREATED BY ATMOSPHERIC PRESSURE PLASMAS - FROM GAS PHASE CHEMISTRY TO BULK LIQUID

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Large number of recent studies are investigating operation of cold atmospheric pressure (CAP) plasmas in contact with liquids. This is a continuation of the research related to biomedical applications where CAP has proved its excellent potential for sterilization and cleaning of both living tissues and inorganic materials from pathogenic microorganisms [1]. Applications of CAP use chemically reactive gaseous environment that contain reactive oxygen and nitrogen species such as $\bullet\text{OH}$, $\bullet\text{NO}$, H_2O_2 , NO_2^- , NO_3^- , HNO_3 etc. These species produced in CAP, which is in contact with the liquid, can penetrate and react with molecules in a bulk liquid modifying its physical and chemical properties [2]. This interaction goes through the interfacial region located between the gaseous plasma and a bulk liquid where many important processes involving short-lived species occur. As a result, the treated liquid is activated or, in case of polluted water, decontaminated by plasma [3]. However, in many cases specific plasma-liquid interactions behind the achieved positive results are elusive due to unknown processes in the interfacial region.

Here we will present results of laboratory-scale studies using different plasma source configurations that aim to induce decontamination of polluted water and/or activation of clean water. In all experiments we used plasma jets powered by a continuous kHz signals with the liquid samples placed below the jets. We will show results of detailed plasma diagnostics as well as measurements of basic physico-chemical properties of treated samples in order to reveal the influence of the plasma treatment. Special attention will be devoted to the possibility of assessing reactions in the interfacial region in order to clarify important reactions which exist in the particular treatments.

Acknowledgement: This work is supported by projects ON171037 and III41011 of the MESTD, Republic of Serbia.

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Plasma treatment of liquids and applications in agriculture

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As recent investigations in non-equilibrium atmospheric pressure plasma field are directed towards different applications in biology, agriculture and food industry, many new challenges emerge. In this work we aim to examine properties of a plasma treated water and its further influence on biological samples within the scope of plasma agriculture applications of atmospheric pressure plasma jets. We used two configurations of plasma jets for treatments of pure distilled water and water contaminated with dissolved pesticides. The jet parameters were varied and we measured physico-chemical properties of the treated liquid samples. Moreover, we used the treated samples for imbibition of commercially used plant seeds and then investigated their germination.

1. Introduction

With expansion of the field of non-equilibrium low temperature plasmas to atmospheric pressure sources, their application domain has grown immensely. In the last 20 years, development of new plasma sources operating at atmospheric pressure has been mostly related to the new biomedical applications such as wound healing, cancer treatment as well as sterilization and treatment/production of biocompatible materials [1,2]. Since low temperature plasmas are environmentally friendly and the active gaseous environment is at room temperature, recent investigations are directed towards different aspects of plant biology, agriculture and food industry [3].

Although investigations in these fields so far proved that there is a great potential for application of atmospheric pressure plasma (APP) sources, what also became known is that complexity of the processes in the whole plasma-treated target system is immense thus making a large obstacle in studying the mechanisms of interaction between plasma and target of interest.

2. Results and Discussion

In this work we aim to examine results of a plasma treatment of liquid and its further influence to biological samples within the scope of plasma agriculture applications. Treatments of pure distilled water and water contaminated with dissolved pesticides were performed by using a plasma jet. In the study we used two configurations of APP jets powered by a continuous kHz signal and operated with He and Ar as working gases. After the procedure, we established physico-chemical properties of the liquid sample, e.g. by measuring concentrations of nitrite ions, nitrate ions and hydrogen peroxide by using a colorimetric method.

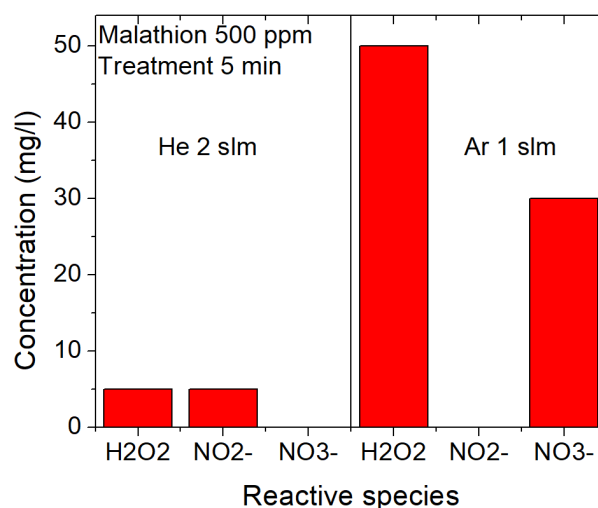


Fig. 1. Reactive species concentration in water sample contaminated with 500 ppm of Malathion after 5 mins treatment by pin-electrode jet.

In Fig. 1 we show concentrations of reactive species obtained after 5 min treatment of Malathion spiked water sample by a pin-electrode jet. Depending on the working gas, H₂O₂ concentrations differ by an order of magnitude while nitrites and nitrates show completely different behaviour. Therefore, it is obvious that changing the working gas produced different reactive species in the liquid. This proved to be of great importance since the treated samples were used for imbibition of commercially used plant seeds and a follow-up of their germination process. Within the framework of plasma agriculture, changes in the properties of treated liquid samples could play essential role when successful and efficient treatment of pure or contaminated water is expected. Moreover, as reactive species produced in the plasma treatment are also biologically significant molecules important for functioning of plant cells their mutual balance is

also important. Therefore, the influences of plasma parameters, properties of plasma treated sample and of the biological target are all intertwined in particular plasma treatment. Only a detailed knowledge of all parameters and mechanisms could provide a prosperous result for the particular application in plasma agriculture field.

This research has been supported by MESTD Republic of Serbia, under projects III41011 and ON171037.

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Part 1 of 2



I1.304 Influence of the atmospheric pressure plasma source configurations on the properties of treated liquid samples

Monday, July 8, 2019 6:00 PM (30 minutes)

See the full abstract here <http://ocs.ciemat.es/EPS2019ABS/pdf/I1.304.pdf>

In recent years, expansion of non-equilibrium plasma applications is directed towards plasma sources operating at atmospheric pressure that are used for treatment of liquids. Usually, different applications involving liquid targets have different aims and, as precondition for all these applications, it is important to make the plasma processes as efficient as possible in every specific case. Therefore, these tasks are demanding due to the complexity of plasma chemistry, which depends on the type of plasma source and is further tangled by the presence of liquid target. Here we will present results of a laboratory-scale study aiming to make comparison between different plasma source configurations and to reveal their influence on treated pure and contaminated water samples. Two-level approach is necessary: on one side one should characterize the plasma used for treatments while, on the other side, properties of the liquid samples should be obtained. Detailed discharge diagnostics involving optical emission spectroscopy and electrical characterisation will provide information on plasma conditions for particular source. Emission spectrum provide information on excited species produced in the gas phase. Measurement of electrical signals allow to calculate power input provided to the system and thus establish a parameter describing the plasma. Additionally, certain physicochemical properties of the treated liquids (pH, dissolved Oxygen content, conductivity, total organic carbon content in contaminated samples etc.) will be obtained allowing to cross reference data with plasma characterization and give an insight into interaction chemistry of the specific plasma source used for liquid treatment. Results regarding the influence of different plasma source configurations to the treated liquid will be presented in the context of possibility of applications in the field of plasma agriculture (PAW, water decontamination etc.).

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HZS P 1

Identifikacija degradacionih produkata malationa nastalih primenom plazme na atmosferskom pritisku

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U ovom radu opisan je razvoj instrumentalne metode za određivanje proizvoda degradacije organofosfatnog pesticida malationa. Za razgradnju malationa u cilju dekontaminacije poljoprivrednih otpadnih voda koje se potom mogu ponovo koristiti za zalivanje, primenjen je izvor plazme na atmosferskom pritisku - plazma mlaz. Polazni rastvor malationa i uzorci tretirani plazma mlazom su analizirani korišćenjem LTQ XL linearnog jonskog trapa, kao masenog spektrometra. Na osnovu rezultata MSⁿ analize odabrane su reakcije fragmentacije dobijenih degradacionih produkata malationa. Uz tačno-hromatografsku analizu, ove reakcije su zatim korišćene za identifikaciju i praćenje malationa i odabranih produkata degradacije u tretiranim uzorcima. Kao dominantni produkti razgradnje identifikovani su dietil-2-merkaptosukcinat (m/z 229), kao i malaokson (m/z 315) koji se dalje degraduje u dimetilfosfonat (m/z 111). Takođe je utvrđeno postojanje degradacionih proizvoda (m/z 127, 273 i 293) koji su prisutni u manjem obimu. Na osnovu dobijenih rezultata predložen je put razgradnje malationa i nastanak degradacionih proizvoda.

Zahvalnica: Ovaj rad je finansijski podržan od strane Ministarstva prosvete, nauke i tehnološkog razvoja Republike Srbije (projekti: ON 172007, ON 171037 i III 41011).

Identification of malathion degradation products produced by atmospheric pressure plasma

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This paper describes the development of an instrumental method for determination of degradation products of organophosphate pesticide malathion. For degradation of malathion, an atmospheric pressure plasma jet was applied, for the purpose of decontamination of agricultural wastewater, which can then be re-used for watering. Initial malathion solution and samples treated with atmospheric pressure plasma were analyzed using the LTQ XL linear ion trap as a mass spectrometer. Based on the results of MSⁿ analysis, fragmentation reactions of obtained malathion degradation products were selected. Using liquid chromatography, these reactions were then used to identify and track malathion and selected degradation products in the treated samples. The dominant degradation products were identified as diethyl-2-mercaptopuccinate (m/z 229), and malaoxon (m/z 315), which further degrades to dimethyl phosphonate (m/z 111). Additional degradation products (m/z 127, 273 and 293), present to a lesser extent, were also determined. Based on the obtained results, the pathway of malathion degradation and formation of degradation products was proposed.

Bulletin of the American Physical Society

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Monday–Friday, May 27–31, 2019; Milwaukee, Wisconsin

Session S01: Poster Session III (4:00pm-6:00pm)
4:00 PM, Thursday, May 30, 2019
Wisconsin Center Room: Hall A

Abstract: S01.00027 : Tracing Plasma Produced Atomic and Molecular species from Plasma into the Liquid and Living tissue for various applications

[Preview Abstract](#)

⬅ Abstract ➡

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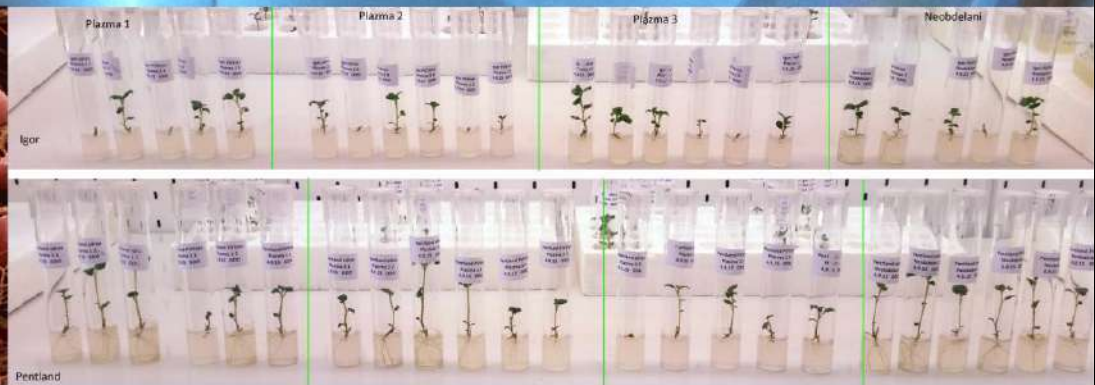
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(Institute of Physics, University of Belgrade Serbia)

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(Institute of Physics, University of Belgrade Serbia)

Significant effects observed in applications of atmospheric pressure non-equilibrium plasmas have been shown to be due to the effect of plasma produced atomic and molecular reactive species. Some of those species are the ones acting as signaling agents initiating response of the living cells. At the same time, albeit in larger numbers, they may be chemical agents that can damage or dissociate unwanted living organisms, human cells or chemical components. We try to follow the trail of atomic and molecular physics starting from their formation, their passage into liquids and then passage into living cells or the reaction of cells that they invoke. Two examples will be the long term changes in enzymes regulating hydrogen peroxide in plant cells and destruction of malathion, a pesticide used in agriculture that may be a model of more lethal weapons of mass destruction. Finally we shall illustrate how presence of those active species in plasma treated water affects the germination of seeds. -abstract- Authors Zoran LJ Petrovic, Nikola Skoro, Suzana Zivkovic, Milica Milutinovic, Olivera Jovanovic, Nenad Selakovic,



8th Central European Symposium on Plasma Chemistry



26th to 30th May 2019, Gozd Martuljek, Slovenia

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Production of reactive species in pure and polluted water treated by atmospheric pressure plasma

O. Jovanović (1), N. Škoro (1), N. Puač (1) and Z. Lj. Petrović (1, 2)

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Recent studies deliver wider knowledge on the role and significance of reactive oxygen and nitrogen species (RONS) that lead to their new chemical and biological applications and, at the same time, make a demand for development of new methods to generate them. Numerous research show that atmospheric pressure plasma can be used to efficiently produce numerous aqueous reactive species in plasma treated water. In the studies involving the influence of plasma treated water on biological samples researchers demonstrate that hydrogen peroxide as well as nitrite and nitrate play an important role in biological effects. Therefore, it is suggested that quantitative analysis of the above species should be related to the plasma source characterization in terms of the evaluation of the biological activity of liquids treated with atmospheric pressure plasma source.

In this work we have used an atmospheric pressure plasma jet to produce nitrate and nitrite ions and hydrogen peroxide in liquid samples. The plasma source used in the experiments was a pin-electrode jet with He as working gas with different flow rates. The treatments of pure distilled water and water contaminated with dissolved pesticides were performed. The liquid samples were placed below the plasma source powered by a continuous kHz signal and treated for different treatment times. After treatments, the physicochemical properties of the treated samples were measured. Additionally, we measured concentrations of nitrite ions, nitrate ions and hydrogen peroxide generated by the discharge plasma in exposed water solutions. This was done by using a colorimetric method with test strips. The chemical analysis of aqueous solutions after treatments show that creation of RONS in pure and polluted water is different, i.e. that the treated liquid composition is affecting the RONS production. The existence of hydrogen peroxide and nitrite ions was observed in all samples, but nitrate ions were detected only in clear treated water. Additionally, results show that increase in concentrations of nitrite ions and nitrate ions depends on the duration of the treatment.

Acknowledgements. This work was supported by projects ON171037 and III41011, MESTD, Serbia.

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Atmospheric pressure plasma decontamination of water polluted by organophosphates used in agriculture

N. Škoro¹, N. Puač¹, O. Jovanović¹, G. Malović¹, Z. Lj. Petrović^{1,2}

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In biomedicine, atmospheric pressure plasmas (APP) have proved their excellent potential for sterilization and cleaning of both living tissues and inorganic materials from pathogenic microorganisms [1]. Moreover, large number of recent studies are investigating operation of APP in contact with liquids. Results show that chemically reactive gaseous environment produced by plasma sources can influence and modify physical and chemical properties of liquids [2]. As a result of the knowledge acquired in previous studies the investigation of APP now widens to a new emerging research field - plasmas in agriculture, where novel applications for APP appear.

Nowadays, one of the biggest environmental problems is water pollution with contemporary agriculture techniques as one of the main sources causing the pollution of surface waters. Organic micropollutants, originating from immense use of pesticides in agriculture production, require special chemical or biological treatments for water purification, again using environmentally hazardous substances. Our idea is to employ APP for decontamination of water polluted by pesticides, which is by now successfully used in warfare applications [3]. As a first step, we conducted a study on decontamination of water samples polluted with different pesticides, i.e. organophosphate compounds, by using an atmospheric pressure plasma jet (APPJ) operating with He as working gas. The plasma jet was powered by a continuous kHz signal source. Liquid samples placed below the APPJ were treated for different duration times, different sample volumes and different water contamination levels. Optical and electrical characterization of the APPJ was performed in order to obtain information about stability of the treatment conditions and the plasma properties. Before and after the treatment liquid samples were analyzed by spectrophotometric techniques, high performance liquid chromatography (HPLC) and liquid chromatography coupled with mass spectrometry (LC-MS) in order to follow degradation of organophosphates. Significant and efficient degradation of pesticides is noticed in all cases and appearance of degradation products is observed in the liquid sample. Thus, we could also evaluate toxicity of produced by-products. From measurements of parent molecule degradation we established dependence of the decontamination efficiency on treatment time. Having in mind the possibility of reuse the decontaminated water in agriculture, we also investigated the influence of treated water on seed germination.

This work was supported by projects ON171037 and III41011, MESTD, Serbia.

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GEC 2022

75th Annual Gaseous Electronics Conference



ICRP-11

11th International Conference on Reactive Plasmas

PROGRAM BOOK

Date : October 3 - 7, 2022

Sendai International Center Conference Building

Joint Conference in Sendai, Japan

Supported by
American Physical Society
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The Japan Society of Applied Physics



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Day 5 *Friday, October 7th, 2022*

Room	Tachibana	Hagi	
8:00 AM - 9:30 AM	DF1 Plasmas and Nanotechnology III [Wei-Hung Chiang] - 9:00 AM	EF1 Plasma Medical & Agricultural Application I [Dingxin Liu]	
9:30 AM - 10:00 AM	--- Coffee Break ---		
10:00 AM - 12:00 PM	DF2 Laser Diagnostics II [Holger Kersten]	EF2 Plasma Medical & Agricultural Application II [Kenji Ishikawa] - 11:45 AM	
12:00 PM - 1:30 PM	--- Lunch ---		
1:30 PM - 3:30 PM	DF3 Plasma Propulsion III [Andrei Smolyakov]	EF3 Plasma Medical & Agricultural Application III [Nevena Puac] - 3:15 PM	
3:30 PM - 4:00 PM	--- Coffee Break ---		
4:00 PM - 5:30 PM	DF4 Fundamental Processes - 5:15 PM	EF4 Plasma Medical & Agricultural Application IV [Mounir Laroussi]	
5:30 PM - 6:00 PM	DF5 Closing Ceremony - Toshiro Kaneko - Julian Schulze - Shahid Rauf		

Bulletin of the American Physical Society

75th Annual Gaseous Electronics Conference

Monday–Friday, October 3–7, 2022; Sendai International Center, Sendai, Japan

The session times in this program are intended for Japan Standard Time zone in Tokyo, Japan (GMT+9)

Session EF3: Plasma Medical & Agricultural Application III

1:30 PM–3:15 PM, Friday, October 7, 2022

Sendai International Center Room: Hagi

Chair: Jun-Seok Oh, Osaka Metropolitan University

Abstract: EF3.00001 : Role of atmospheric pressure plasma in triggering of cell mechanisms in plant cells*

1:30 PM–2:00 PM

[Abstract](#) ➔

Presenter:

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The atmospheric pressure plasmas (APPs) are known to be rich in Reactive Oxygen and Nitrogen Species (RONS) and this rich chemistry is responsible for triggering of cell mechanisms in case of plant or human/animal cells. We can divide this influence in two groups: (1) RONS in gas phase; (2) RONS in liquid phase. Therefore, in order to better understand the reasons for triggered mechanisms and outcomes (better germination percentage and speed, breakout of dormancy, creation of embryos etc.) we need to know and be able to tailor the plasma chemistry both in gas and liquid phase. We have used several APPs for gas phase treatments of plant cells and also for production of Plasma Activated Water (PAW) in order to investigate the influence of liquid RONS chemistry on plant cells. Here we will present different APP sources that are used for production of PAW used for seed imbibition in germination process and direct treatment of meristematic plant cells. Dielectric Barrier Discharge type of APPJ was used for production of PAW, while the plasma needle type was used for direct treatment of meristematic plant cells. The main idea was to check if plasma treatments can be used as for breaking of dormancy and trigger mechanisms in cells even in a normally non-permissive conditions.

*This work was supported by MESTD RS, grant numbers 451-03-9/2021-14/200007 and 451-03-68/2022-14/200024.

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GEC 2022

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IR4 Plasma Liquid Interaction III**Chair:** Naoki Shirai (*Hokkaido University*)

- IR4.00001**
1:30PM - 2:00PM
Generating enhanced chemical reactions inside highly charged microscale droplets for remote delivery of reactive radicals and high purity nanomaterials
Invited Speaker Paul Maguire
- IR4.00002**
2:00PM - 2:15PM
Analyses of chemical reactions in plasma generated within humid oxygen bubbles with highly concentrated ozone
Nozomi Takeuchi, Ryota Kazama, Taichi Watanabe, Shungo Zen
- IR4.00003**
2:15PM - 2:30PM
Measurement of Radicals Generated by Plasma in Contact with Dilute Sulfuric Acid by Using Electron Spin Resonance (ESR) Method
Kosuke Tachibana, Nao Murata, Kaede Saito, Seiji Kanazawa, Katsuyuki Takahashi, Junko Hieda, Nozomi Takeuchi, Oi Lun Li
- IR4.00004**
2:30PM - 2:45PM
Creation of reaction species by an atmospheric pressure plasma jet when treating liquids
Nikola Skoro, Olivera Jovanović, Anđelija Petrović, Gordana Malović, Nevena Puac
- IR4.00005**
2:45PM - 3:00PM
Numerical simulation of chemical reactions in PBS-like solution exposed to atmospheric-pressure plasmas
Enggar Alfianto, Kazumasa Ikuse, Zoltan Donko, Satoshi Hamaguchi
- IR4.00006**
3:00PM - 3:15PM
Experimental study of the plasma chemistry in atmospheric pressure plasma contacts with dilute sulfuric acid
Siqi Deng, Nozomi Takeuchi, Junko Hieda, Katsuyuki Takahashi, Kosuke Tachibana, Oi Lun Li
- IR4.00007**
3:15PM - 3:30PM
Polymerization of EDOT on H₂O by DBD treatment
Tomohiro Okamoto, Tatsuru Shirafuji, Jun-Seok Oh

DR5 Optical Diagnostics**Chair:** Holger Kersten (*Kiel University, Germany*)

- DR5.00001**
4:00PM - 4:15PM
Coupled Electrical and Optical Characterization of Electrostatic Discharges
Claudia A Schrama, Sarah Hinnegan, Jonathan Barolak, Daniel Adams, Alex Wilhelm, Charles G Durfee
- DR5.00002**
4:15PM - 4:30PM
Locally-resolved temperature and electron number density measurements in the VKI inductively-coupled plasma wind tunnel
Andrea Fagnani, Diana Luis, Damien Le Quang, Alan Viladegut, Bernd Helber, Olivier Chazot
- DR5.00003**
4:30PM - 5:00PM
Optical Emission Spectroscopy Measurement for Plasma Parameter Identification — from Kinetic Modeling to Data Science
Invited Speaker Hiroshi Akatsuka

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Session IR4: Plasma Liquid Interaction III

1:30 PM–3:30 PM, Thursday, October 6, 2022

Sendai International Center Room: Sakura 2

Chair: Naoki Shirai, Hokkaido University

Abstract: IR4.00004 : Creation of reaction species by an atmospheric pressure plasma jet when treating liquids*

2:30 PM–2:45 PM

← Abstract →

Presenter:

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Versatility of effects induced in liquids after plasma treatments has facilitated development of various applications in the fields of medicine, biology, water treatment etc. Plasma treated liquids (PTL) may contain different long-lived chemical species, but their occurrence and concentrations depend on the plasma system used. Some of the fundamental and significant reactions have been specified, but due to complexity of the chemistry in both plasma and liquid and their interaction, obtaining a wider prospect is still remote. To investigate interaction between atmospheric pressure plasma and a liquid target, in this study we associated results of spectrally resolved imaging and optical emission spectroscopy with quantification of reactive oxygen and nitrogen species (RONS) formed in the treated sample. For creation of the plasma above the sample we used a plasma jet operating in kHz regime with He/Ar as working gases. Relation between plasma and liquid properties has been established for wide range of different plasma parameters and several liquid targets. Obtained results provided relation between creation of RONS in the gas phase and in PTL.

*This research was supported by the Science Fund of the Republic of Serbia, grant No. 3114/2021 - Project APPerTAin-BIOM and the MESTD of the Republic of Serbia grant No. 451-03-68/2022-14/200024.

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BOOK OF ABSTRACTS

including general information and program



Edited by Karol HENSEL, Richard CIMERMAN, Aleksandra LAVRIKOVA,
Mário JANDA, and Zdenko MACHALA

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		Non-thermal plasma for generation of antimicrobial aerosol	
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		Selective study of ion-substrate interactions using the VUV-photoionization chamber	
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Correlation between properties of plasma treated liquids with characteristics of atmospheric pressure plasma devices

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Non-equilibrium plasma at atmospheric pressure that operate in contact with liquids has proven to induce various chemical reactions in liquids. The most important chemically reactive species formed in the gas discharge above the liquid target are reactive oxygen and nitrogen species (RONS), electrons, UV radiation etc [1]. In complex interactions these species penetrate and/or react with liquid-phase molecules producing short and long-lived species in the target. Thus, adjusting of the plasma chemistry in the gas phase results in different outcomes of the liquid treatment which supports employment of the plasma treatments for different applications [2,3]. With respect to the purpose of the plasma treatment, it can be used for creation of new species, i.e. for liquid activation and creation of plasma activated water (PAW) and plasma activated medium (PAM), or for destruction of dissolved pollutant molecules – for decontamination processes. In this work we will present both kind of plasma treatments featuring comparison of properties of the treated liquids depending on different plasma setups, operating parameters etc. Treatments of pure and contaminated water (with organic dye AB25) were performed by using pin-type of plasma sources operating with He and Ar. These jets were operated with continuous sine signal at the frequency around 350 kHz. The setups were designed to enable precise determination of power delivered to the plasma and the sample. We investigated the influence of the working gas as well as sample volume to the RONS concentration in the activated water. For PAW treatments, we observed much higher production of H₂O₂ in case of Ar plasma than in He. We tested variation in the efficiency of the plasma decontamination by adding multi-pin source and for conditions of recirculation of the contaminated sample. The effective treatment surface parameter proved to be significant for regulation of the decontamination efficiency. With respect to the treatments of the cell media, we used a dielectric barrier discharge (DBD) jet configuration that operated with continuous signal at around 80 kHz. We studied the influence of the input power on production of RONS in the PAW and PAM. It was shown also that important operating parameter for this kind of plasma jet was gas feed gas humidity. Understanding and optimizing the production of RONS in the plasma system is important due to their roles in major biological processes, thus fine tuning of the produced species is of great importance.

This work was supported by NOWELTIES project - EU H2020 MSCA ITN No. 812880 and by Ministry of Education, Science and Technological Development of Republic of Serbia grant number 451-03-68/2022-14/200024.

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ABSTRACTS

PIAgri Workshop



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Molecular response to PAW in model plant species

**M. Milutinović¹, O. Jovanović², N. Devrnja¹, M. Todorović¹, S. Živković¹, J. Savić¹,
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Non-thermal temperature plasmas (NTPs) have rich chemistry of Reactive Oxygen and Nitrogen Species (RONS) that are formed in gas phase and, in case of water treatment, in gas/liquid interface in liquid [1]. NTPs can be applied in direct treatments of plant samples or indirectly when treated water, called Plasma Activated Water (PAW), is used in treatments. In both cases RONS (short or long-living) are responsible for triggering various mechanisms and effects in plant cells. RONS have a dual role and a dose-dependent effect, they can regulate the normal physiological activities of plants as signaling molecules at the range of physiological concentration, and can trigger damage to lipids, proteins and DNA at too high or too low concentration. Plants integrate RONS with genetic, epigenetic and external signals to regulate developmental processes. RONS signaling is highly integrated with hormonal signaling networks, thereby allowing plants to adjust to environmental cues.

All organisms have adaptive responses to oxidative stress, with antioxidant enzymes (i.e. catalase, peroxidase, superoxide dismutase) being induced by changes in the levels of H₂O₂ or O₂^{•-}, leading to the activation or silencing of genes encoding defensive enzymes and transcription factors [2]. Although various observations have led to the suggestion that cells have the means to sense RONS and to induce specific responses, the underlying mechanisms are still not fully understood. In addition, plants also have non-enzymatic systems known to remove RONS, which are important players in plant processes that use RONS-dependent signaling mechanisms [3]. In this work, we have shown, by using molecular approaches, how PAW treatment affect expression of genes coding for specialized metabolites and hormones, thus regulating development and stress responses in the model plant system *Arabidopsis thaliana*.

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Investigation of an atmospheric pressure pin-type plasma jet for water treatment - optical diagnostics and temperature measurements

O. Jovanović¹, N. Puač¹, A. Petrović¹, M. Gromov², A. Nikiforov², N. Škoro¹

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In the increasingly important field of plasma agriculture, atmospheric pressure plasmas have proved their excellent potential for the elimination or reduction of organic contaminants and sterilization of microorganisms [1,2]. The exposure of aqueous solutions to plasma induces many reactions occurred in the gaseous phase and introduces reactive species in aqueous phases. One of the most commonly detected chemical species in gas plasma is OH radical because it plays a key role in the reactions of degradation of organic pollutants from water [3]. Another important factor that should be controlled during the plasma wastewater treatment to avoid the evaporation of harmful substances is the gas temperature. From the point of view of plasma physics, it is essential for future applications that the plasma processes and the effects of treatment are correlated, which can only be done by accurate and thorough diagnostics of the plasma sources.

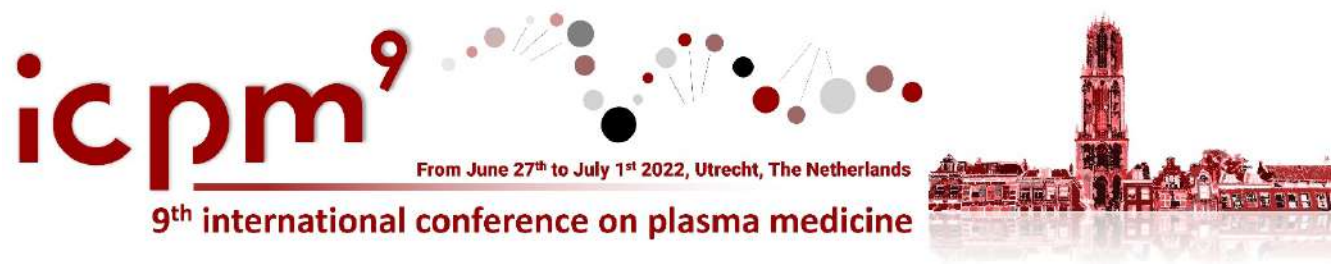
Atmospheric pressure plasma jet (APPJ) in pin-electrode configuration operating in contact with liquid sample has been investigated by two different measurement techniques for gas temperature measurement. Firstly, the gas temperature above the liquid was determined by using the optical emission spectroscopy (OES) method for measurement based on partially rotationally resolved emission from hydroxyl (OH) radical. Another goal was to employ an active method - Rayleigh scattering laser spectroscopy which allows spatially resolved gas temperature measurements and to compare obtained results with OES measurements. Since the plasma jet generates intense streamer discharge, such a detailed analysis of the emission spectrum of plasma radiation and precise measurement of gas temperature was necessary for the potential application in biotechnology and wastewater treatment. Finally, as earlier studies have shown that OH radicals formed in the plasma play an important role in the degradation of pollutants dissolved in water, the absolute values of OH radicals were recorded by laser-induced fluorescence (LIF) technique.

The recorded spectra showed intense emission of OH radicals at wavelengths around 306 nm for discharge generated in pure Ar and an admixture of Ar and synthetic air. Preliminary results showed that the temperature in the plasma core reached values of up to 300°C. Also, it was obtained that the intensity of the LIF signal increases with the increase in the power deposited in the plasma, whether the discharge was in contact with distilled water or tap water.

This work was supported by a STSM Grant from the COST Action CA19110 PLAGri, supported by the COST Association (European Cooperation in Science and Technology) and the Ministry of Education, Science and Technological Development, grant number 451-03-68/2022- 14/200024.

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Book of Abstracts

Editors: Gerrit Kroesen, Romolo Laurita,
Ana Sobota, Matteo Gherardi

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* 1st Annual Meeting of COST Action PlasTHER CA20114		

Plasma treatment of seeds and plant cells: role of reactive oxygen and nitrogen species in formation of plantlets and embryos in non-permissive conditions

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The population growth together with constant climate changes represent a serious challenge for humankind. Additionally, the usage of the pesticides have created adverse effect on environment, which in return impact even more agricultural production. In order to comply with the demands and to adapt to the new conditions the farmers need to change or upgrade existing practices by employing new technologies. As being a promising tools in application in medicine, non-thermal (cold) plasmas (NTPs) are seen as a green alternative to conventional fertilizers in agriculture to improve yields, increase size and robustness of plants and to reduce (or eliminate) the need for pesticides [1, 2].

NTPs have rich chemistry of Reactive Oxygen and Nitrogen Species (RONS) that are formed in gas phase and, in case of water treatment, in gas/liquid interface in liquid [2, 3]. We can use NTPs in direct treatments of seeds or plant cells where samples are in contact with plasma gas phase chemistry or indirectly when treated water is applied to the biological samples. In both cases RONS (short or long-living) are responsible for triggering various mechanisms and effects in plant cells. To better understand the reasons for triggered mechanisms and outcomes (better germination percentage and speed, breakout of dormancy, creation of embryos etc.) it is important to characterize the plasma chemistry both in gas and liquid phase. We have used several atmospheric pressure plasma systems in treatments of seeds and plant cells in order to investigate the mechanisms responsible for better germination in seeds, enzyme response and formation of somatic embryos in non-permissive conditions. The mechanisms investigated were linked with the chemistry of RONS created in gas phase and/or deposited in liquid phase. Thus, we were able to acquire the data that can be used in optimization of plasma treatment processes.

This work was supported by Ministry of Education, Science and Technological Development of Republic of Serbia, grant numbers 451-03-9/2021-14/200007 and 451-03-9/2021-14/200024.

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76th Annual Gaseous Electronics Conference
Monday–Friday, October 9–13, 2023; Michigan League, Ann Arbor, Michigan

Session FF2: Agricultural and Environmental Applications

10:00 AM–11:30 AM, Friday, October 13, 2023
Room: Michigan League, Koessler

Chair: Nils Hansen, Sandia National Laboratories

Abstract: FF2.00005 : Use of atmospheric pressure plasma reactivity in water treatment for agricultural applications*

11:00 AM–11:30 AM

← Abstract

Presenter:

Nikola Skoro
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Non-thermal Plasmas (NTP), formed at atmospheric pressure, produce rich gas phase chemistry of Reactive Oxygen and Nitrogen Species (RONS). In a contact with a liquid target, NTPs induce production of different long-lived RONS species inside the liquid. NTPs can be employed for creation of plasma activated water (PAW) or for destruction of pollutants dissolved in water. In recent years both aspects are being developed as parts of a new field of plasma agricultural applications. The way to tailor the RONS concentrations is by adjusting of the plasma chemistry in the gas phase, the type of water and sample vessel properties. We will present results of plasma treatments of distilled and tap water using an atmospheric pressure plasma jet with pin electrode operating with addition of noble gas (He, Ar). The idea was to feature comparison of PAW properties obtained with different plasma system parameters. Produced PAW was used for treatments of different seeds and plants with an attention on linking the plasma properties with effects on treated plant material. On the other hand, the multi-pin jet was used for decontamination of water polluted by organic dyes and pharmaceuticals. This plasma source was designed based on a single-pin unit and obtained results can provide an insight into scaling up the plasma reactivity which is a necessary step for any agricultural application. We also tested variation in the efficiency of the plasma decontamination by adding recirculation of the contaminated sample. The effective treatment surface parameter proved to be significant for regulation of the decontamination level.

*This work was supported by the Science Fund of the Republic of Serbia, 7739780, Atmospheric pressure plasmas operating in wide frequency range – a new tool for production of biologically relevant reactive species for applications in biomedicine - APPerTAin-BIOM. We acknowledge also MSTDI of Republic of Serbia grant no. 451-03-68/2023-01/200024, no. 451-03-47/2023-01/200007 and NOWELTIES project - EU H2020 MSCA ITN No. 812880.

Technical Meeting on Emerging Applications of Plasma Science and Technology

Report of Contributions

09:00	Plasma electrochemistry for organic synthesis: Pinacol coupling as proof-of-concept <i>CR-4 , IAEA Headquarters</i>	<i>R. Mohan Sankaran</i> 09:00 - 09:30
	Plasma nanotechnology for clean energy, green chemistry, and zero-carbon future <i>CR-4 , IAEA Headquarters</i>	<i>Kostya (Ken) Ostrikov</i> 09:30 - 10:00
10:00	Recent Developments in Atmospheric Pressure Plasma for Gas Treatment <i>CR-4 , IAEA Headquarters</i>	<i>Duc Ba Nguyen</i> 10:00 - 10:15
	Coffee Break <i>CR-4 , IAEA Headquarters</i>	10:15 - 10:35
11:00	CHEMISTRY ASSOCIATED WITH PLASMA-LIQUID INTERACTIONS: CHALLENGES AND OPPORTUNITIES <i>CR-4 , IAEA Headquarters</i>	<i>Petr Lukes</i> 10:35 - 11:05
	Methane as a feedstock in plasma-liquid processes <i>CR-4 , IAEA Headquarters</i>	<i>Necip Uner</i> 11:05 - 11:35
12:00	Microwave and RF Plasma Enhanced Synthesis of Nanomaterials <i>CR-4 , IAEA Headquarters</i>	<i>Mohan Jacob</i> 11:35 - 12:05
	Lunch	
13:00	<i>CR-4 , IAEA Headquarters</i>	12:05 - 13:15
	Atmospheric pressure plasma and its application in textile and polymers <i>CR-4 , IAEA Headquarters</i>	<i>Mr Vishal Jain</i> 13:15 - 13:45
14:00	Pros and Cons of Plasma Agriculture: A Current View <i>CR-4 , IAEA Headquarters</i>	<i>Masaharu Shiratani</i> 13:45 - 14:15
	Perspectives on atmospheric pressure air plasma for chemical and microbial decontamination across the food production sector. <i>James Walsh</i>	
15:00	Plant pathogens control using air atmospheric pressure plasmas <i>CR-4 , IAEA Headquarters</i>	<i>Toshiro Kaneko</i> 14:45 - 15:15
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	Plasma applications for smart and sustainable agriculture-possibilities and challenges <i>CR-4 , IAEA Headquarters</i>	<i>Dr Nevena Puač</i> 16:05 - 16:35
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Plasma applications for smart and sustainable agriculture-possibilities and challenges

Thursday, September 21, 2023 4:05 PM (30 minutes)

The population growth represents a serious challenge for humankind due to the continuous increase in demand for food. The climate change is also presenting a significant factor on food production due to the change in weather patterns, pest appearance, water availability etc. Therefore it is necessary to develop new and efficient technologies that can enhance productivity while maintaining food quality and safety. One of these technologies are non-equilibrium (non-thermal plasmas) plasmas that are considered as a green alternative to conventional fertilizers in agriculture to improve yields, increase size and robustness of plants, to reduce (or eliminate) the need for pesticides and in the case of final products treatments of food and its packaging. The reason for this is their rich plasma chemistry that is created in gas phase (at room temperature) and can be easily transferred to liquid samples. Plasma chemistry is rich in Reactive Oxygen Nitrogen Species (RONS) [1] and it was shown that low pressure and atmospheric pressure plasmas can be successfully used in stimulation of the seed growth, increase of germination percentage and decontamination, breaking of dormancy or lengthening of the seed sprout [2, 3]. In direct plasma treatments (gas phase plasma is in contact with sample surface) of seeds the surface is activated and different functional groups can be attached resulting in increased wettability, coating with desired compounds or just decontaminated from pathogens [4]. Another successful approach is in treatment of water for production of Plasma Activated Water (PAW) rich in long living RONS [5]. Here we will present some of the recent results in the field of Plasma Agriculture and address future challenges of plasma applications in this field.

Acknowledgment: This publication is based upon work from COST Action "Plasma applications for smart and sustainable agriculture (PLAgri)", supported by COST (European Cooperation in Science and Technology).

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Presenter: Dr PUAČ, Nevena (Institute of Physics Belgrade)

Session Classification: Agriculture

Track Classification: Atmospheric Pressure Plasma



PLATHINIUM

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Abstract book

11 - 15 September 2023 - Antibes, French Riviera

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HELIAG-P1-203 • Investigation of toxicity of Plasma Activated Water on *Lemna Minor*

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Cold plasmas with their highly reactive chemistry of Reactive Oxygen and Nitrogen Species (RONS) are of a key importance in biomedical applications [1, 2]. The production and composition of RONS (O, O₃, OH, NO, H₂O₂, O₂⁻ etc.) depend on plasma parameters and the success of creating adequate plasma chemical cocktail for certain treatment rely on the control of these parameters. In case of treatments of water RONS created in gas phase interact with liquid interface and are deposited in the water creating Plasma Activated Water (PAW). It is shown that PAW has a positive effect on germination percentage, its application as a watering agent results in a better yield of plants etc. [3]. But it is still unknown how the PAW released into land water bodies can influence a wildlife. Here we will present results of PAW effect on the *Lemna minor*, a model plant used for toxicity tests in environmental sciences. We have used an Atmospheric Pressure Plasma Jet (APPJ) configuration with sharpened-end powered electrode that generates a streamer discharge above the water sample (tap water). The plasma source was powered by a continuous kHz signal and as working gas we used Argon. After the treatment we have performed detailed physico-chemical analysis of PAW including RONS detection, pH, temperature and electrical conductivity. PAW was then used in 5 different dilutions (100%, 90%, 60%, 30% and 10%) in order to investigate its influence on *Lemna minor* samples. The samples were kept in controlled conditions (temperature, humidity) for three weeks after which we have measured lengths of the roots, surface of the leaves and percentage of chlorophyll. It was shown that small percentage of PAW can have positive effect, while for higher percentages it is completely toxic. For all samples the length of the roots reduced. On the other hand, PAW diluted to 10% and 30% resulted in the increase of the leaf surface and in percentage of chlorophyll when compared to control samples.

Thanks/Acknowledgement

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ICPIG XXXV Egmond aan Zee 2023



July 9th – 14th, 2023

Hotel Zuiderduin, Egmond aan Zee,
The Netherlands.

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Version: July 12, 2023

TU/e

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Institute of Physics Belgrade, Serbia.
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University of Michigan, United States of America.
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DIFFER, Netherlands.
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Indian Institute of Technology Kanpur, India.
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Superhydrophobic surfaces production by PECVD method

The influence of non-thermal Ar plasma jet on physicochemical properties of treated liquid

O. Jovanović¹, N. Puač¹ and N. Škoro²

¹ Institute of Physics, University of Belgrade, Pregrevica 118, 11080 Belgrade, Serbia

We investigated an atmospheric pressure plasma jet (APPJ) that generates a streamer discharge above the liquid sample. In order to create plasma-activated water (PAW), the device was used for treating both distilled and tap water. The working gases were Ar and an admixture of Ar and synthetic air. Optical characterization of the plasma gas phase showed that the streamer discharge, in addition to the excited Ar lines, produces an abundance of reactive oxygen and nitrogen species (RONS) above and in the treated water. The measurement of the physicochemical properties of PAWs showed that different types and concentrations of the measured RONS (H_2O_2 , NO_3^- , NO_2^-) were formed in different targets.

1 Introduction

The development of APPJs in recent years has been initiated by applications in various fields, among which are biomedicine [1-2] and plasma agriculture [3]. In these fields, plasma-treated liquids play an essential role [4]. In order to always provide the same treatment conditions, ensure the safety of the target and improve the properties of the treated liquid, a thorough study of these systems is necessary.

Here we will present the results of the influence of parameters such as feed gas, treatment time, sample properties and treated volume on the creation of reactive species in both the gas and liquid phase.

2 Experimental setup

In this work, we employed APPJ with pin electrode configuration that operated in kHz regime. As a working gas we used Ar and Ar+Air and plasma was generated above the liquid sample. Electrical characterization and discharge power measurement were conducted to acquire additional information about the treatments' stability, plasma properties and impact of plasma parameters on PAW. We have used spectrally resolved imaging and optical emission spectroscopy for determination and spatial distribution of excited species created in plasma-liquid interaction. We have treated distilled and tap water samples for different treatment times. After treatments, detailed liquid sample diagnostics including electrical conductivity, temperature, pH and RONS measurement were performed.

3 Results and conclusion

Figure 1 shows recorded optical emission spectra of Ar discharge (gas flow 1slm) from the gas gap between the powered electrode and water surface. The right hand side of Figure 1 represents the discharge structure captured by ICCD with addition of the optical filter (@310nm). A correlation between plasma and liquid properties has been established over a wide variety of plasma parameters and liquid

targets. The obtained results demonstrated a link between the formation of RONS in the gas phase and in PAW.

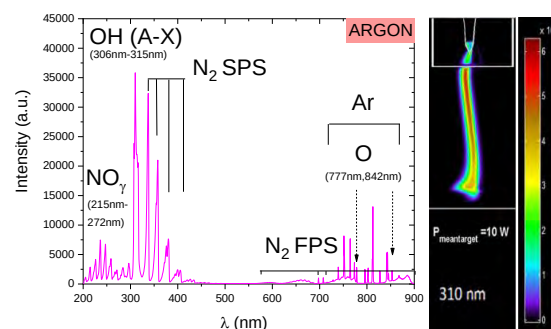


Fig. 1. The obtained optical spectra of Ar plasma above the water sample (left) and the ICCD image of discharge recorded by using 310nm optical filter (right).

Acknowledgment

This work was supported by: The Science Fund of the Republic of Serbia, 7739780, Atmospheric pressure plasmas operating in wide frequency range – a new tool for production of biologically relevant reactive species for applications in biomedicine - APPerTAin- BIOM and by MSTDI Republic of Serbia grant number 451-03-68/2022-14/200024.

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Welcome to the 14th International Conference of the French Society of Plant Biology



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the increasing concentration of the metals compared to the control group, as well as in the cell cycle phases, depending on the variations of the metals and concentration levels with a decrease in the distribution of the cells in the G1 phase and an increase in G2M. Our study shows that the maize plant can be used as a model to evaluate the effect of Ni, Cr and Pb, depending on their concentration in relation to the DNA content and cell cycle phases.

0145-B

ALTERATIONS IN SPECIALIZED METABOLITES' PROFILE OF DAUCUS CAROTA L. CALLI INDUCED BY LOW-TEMPERATURE PLASMA TREATMENT

Suzana ZIVKOVIC^{1*}; Milutinovic MILICA ¹; Danijela MISIC ¹; Nakarada DURA ²; Mojovic MILOS ²; Olivera JAVANOVIC ³; Nikola SKORO ³; Nevena PUAC ³

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Non-thermal plasma (NTP) technology offers a promising future in plant metabolic engineering, being energy efficient and Eco-friendly alternative to the conventional treatments [1]. Plasma environment is enriched with reactive oxygen and nitrogen species (RONS) that participate in various signaling pathways in plants by regulating their metabolic and developmental processes. In the present study calli of different carrot (*Daucus carota* L.) varieties was treated by using plasma needle device designed for biomedical applications [2]. Metabolite profiling revealed that plasma treatment could induce severe qualitative and quantitative changes of the major phenolic compounds detected in carrot calli. Current metabolic alteration was followed by the significant shift in the antioxidant capacity of the treated calli. Obtained results outline the potential application of plasma treatment as a novel elicitor for the production of bio-active compounds in plant in vitro culture systems.

0146-C

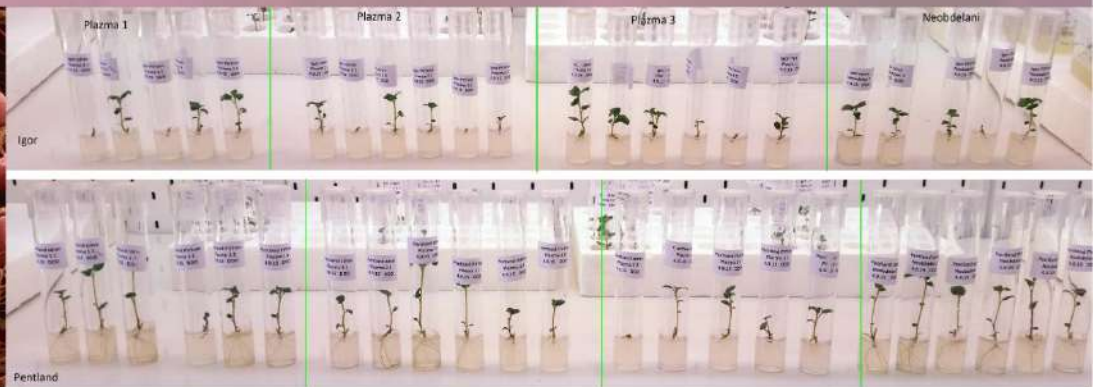
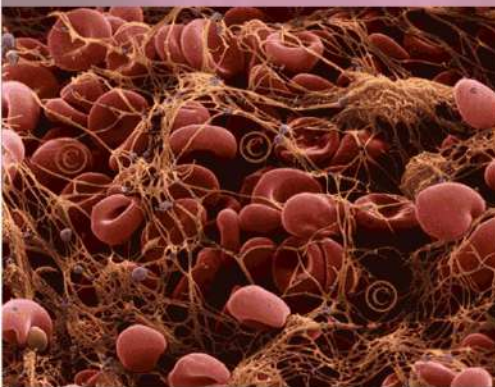
COLD STRESS TOLERANCE IN FLAX (*LINUM USITATISSIMUM* L.): CHARACTERIZATION AT THE PHYSIOLOGICAL, METABOLIC, TRANSCRIPTOMIC AND GENETIC LEVELS.

Henri DESAINT^{1*}; Adèle DE GIULI ¹; Hanine IDELBI ²; Klara CIK ¹; Jean-Xavier FONTAINE ¹; Roland MOLINE ¹; Emmanuel PETIT ¹; Solène BASSARD ¹; Romain ROULARD ¹; Damien HERFURTH ¹; Hervé DEMAILY ³; Stéphanie GUENIN ³; Laurent GUTIERREZ ³; David MATHIRON ⁴; Nicolas MONTRELAY ⁵; David GAGNEUL ¹; Christophe PINEAU ²; Gaëlle MONGELARD ³; Anthony QUERO ¹

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8th International Conference on Advanced Plasma Technologies 3rd Workshop on Plasma Applications for Smart and Sustainable Agriculture



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Program of the 3rd Workshop on Plasma Applications for Smart and Sustainable Agriculture

Sunday, 14th May

16:30	Registration
18:30	Welcome cocktail
19:00	Dinner break

Monday, 15th May

8:30	Registration
8:50	Opening of the workshop
	Session Chairs: Nevena Puač, František Krčma
9:00	Nikola Škoro (Serbia); Properties of plasma-treated liquids used in agricultural and water purification applications; Invited
9:30	Mario Janda (Slovakia); Tuning plasma activated water composition generated by transient spark with electrospray; Invited
10:00	Rasa Žukienė (Lithuania); Cold plasma-induced effects in <i>Stevia rebaudiana</i> Bertoni in different experimental models; Invited
10:30	Coffee break
	Session chair: Kinga Kutasi
11:00	Dawid Zarzeczny (Poland); Influence of low-temperature plasma on the properties of beetroot seeds; STSM
11:15	Ludmila Čechová (Czech Republic); Study of the different plasma sources for water and seed treatment; STSM
11:30	Piotr Terebun (Poland); Impact of plasma activated water prepared by different systems on the selected soil properties; STSM
11:45	Klaas De Baerdemaeker (Belgium); The decontamination of bioaerosols using an in-house rotating dielectric barrier discharge (RDBD) plasma source; STSM
12:00	Lunch break
	Session chair: Romolo Laurita
14:00	Clemencia Chaves-López (Italy); Antifungal Efficacy of SDBD cold plasma on dried tomatoes and mechanisms of action; Invited
14:30	James Walsh (UK); Perspectives on the in-line decontamination of food-processing surfaces using cold atmospheric pressure air plasma; Invited
15:00	Fabio Palumbo (Italy); Plasma deposition as a possible innovative technology in plasma agriculture; Invited
15:30	Coffee break

Properties of plasma treated liquids used in agricultural and water purification applications

Nikola Škoro, Olivera Jovanović, Amit Kumar and Nevena Puač

Institute of Physics, University of Belgrade, Pregrevica 118, 11080 Belgrade, Serbia

Many applications of cold atmospheric plasmas (CAP) nowadays are connected to treatments of liquid samples. As CAP induces rich chemistry in active plasma volume creating short- and long-lived reactive species that affect the treated liquid by penetrating and reacting with molecules in the liquid phase. From the application point of view, this type of chemical interaction induced in plasma treatment could be used for liquid activation, i.e., creation of plasma-activated water (PAW) or plasma-activated medium (PAM), or for decontamination of pollutant molecules dissolved in water. For the latter case, plasma can be used as a chemically free advanced oxidation process (AOP) that can efficiently remove various organic micropollutants (OMP) from water. Both applications, PAW creation and plasma decontamination, are within the domain of plasma agriculture research. For all these applications, in order to understand the main processes governing the liquid treatment and to be able to optimize the treatment, clear knowledge of the entangled connection between input parameters, plasma properties, plasma chemistry, and interaction that takes place inside the liquid target is necessary. This is also a prerequisite in order to be able to tune the plasma for different targets. In this work, we will present several kinds of plasma treatments featuring an investigation of both plasma and treated liquid properties and their dependence on operating parameters. Results of treatments of pure and contaminated water (with organic contaminants) were performed by using a high-frequency pin-type atmospheric pressure plasma jet operating with He and Ar. For PAW creation, we demonstrated how the plasma operating in different regimes with He as working gas yield different concentrations of reactive nitrogen and oxygen species. Moreover, we investigated the influence of the working gas as well as sample volume on the RONS concentration and other physico-chemical properties of the activated water. For example, we observed much higher production of H₂O₂ in case of Ar plasma than in He. Moreover, scaling-up of the plasma system was investigated – we increased the number of jets and added recirculation of the liquid sample in order to enhance the treatment process. In all systems we precisely determined the power delivered to the plasma. For decontamination processes, this was crucial in order to estimate energy efficiency that is required for comparison to other decontamination processes.

Acknowledgement: This work was supported by COST Action CA19110 PIAgri and NOWELTIES project – EU H2020 MSCA ITN No. 812880.

Creation of reactive species by two atmospheric pressure plasma sources while treating water for biomedical applications

Neda Babucić*, Olivera Jovanović, Nikola Škoro and Nevena Puač

Institute of Physics, University of Belgrade, Pregrevica 118, 11080 Belgrade, Serbia

Low-temperature plasma (LTP) has gained significant attention due to its versatile applications in water decontamination, agriculture, and biomedical fields. One of the key challenges in plasma-water interaction is to enhance the flux of reactive species from plasma to liquid. Aerosols, in the form of micrometer-scale droplets, immersed in plasma have been proposed as a strategy to increase the contact area and potentially enhance rates of chemical interaction between plasma and liquid.

Recent research has focused on the development of plasma sources operating at atmospheric pressure in contact with liquid samples, driven by biomedical applications such as wound healing, cancer treatment, deactivation of bacteria and viruses, and sterilization. Plasma Activated Liquids (PALs) have been widely used in numerous successful experiments, but questions remain regarding the mechanisms governing the interaction between plasma and liquid, including the production of various chemical species.

In this work, we utilized atmospheric pressure non-equilibrium plasma sources for the treatment of distilled water and the production of PALs. The first device was a plasma jet with pin-electrode configuration, operated in kHz frequency regime, and the second was microwave launcher that operated at 2.45GHz. In all experiments, Ar was used as a working gas with varying flow from 1 to 3 slm. Optical emission spectroscopy and plasma imaging provided information about the excited species and the point where the concentration of reactive oxygen and nitrogen species (RONS) was largest. Diagnostics of liquid samples were performed to assess the effectiveness of plasma treatment, including measurements of conductivity, pH, temperature, and the amount of deposited RONS.

Overall, this work contributes to a better understanding of different plasma sources and their potential for effective usage in biomedical applications, through changing plasma parameters and treatment conditions.



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Plasma applications for biology, medicine, and agriculture

Session's Chairs:
Prof. Liutauras Marcinauskas, Lithuanian Energy Institute/ Kaunas University of Tech, **Lithuania**
Dr. Ana Megía-Macías, Comillas Pontifical University, **Spain**

14:00 - 14:30	Design of new antimicrobial osseointegrated dental implants S. Carvalho	Prof. Sandra Carvalho , University of Coimbra, Portugal
14:30 - 15:00	Drug Eluting Coatings to Enhance the Functionality of Implants M. Zilberman	Prof. Meital Zilberman , Tel-Aviv University, Israel
15:00 - 15:30	Fluid interfaces as platforms for understanding the interaction of pollutants with lipid barriers E. Guzmán	Prof. Eduardo Guzman Solis , Complutense University of Madrid, Spain
15:30 - 15:45	Room vs. body temperature to evaluate electrical interface parameters of state-of-the-art PEDOT-PSS electrode for neural stimulation K. Cysewska	Dr. Karolina Cysewska , Gdansk University of Technology, Poland
15:45 - 16:00	Plasma-deposited hydrophobic organosilicon coatings for sustainable leather in footwear applications C. Ruzafa-Silvestre , V.M. Serrano-Martínez and E. Orgilés-Calpena.	Mr. Carlos Ruzafa Silvestre , Footwear Technology Centre (INESCOP), Spain

16:00 - 16:30 **Coffee Break / Posters Session - Mezzanine Area**

Session's Chairs:
Prof. Eduardo Guzman Solis, Complutense University of Madrid, **Spain**
Prof. Meital Zilberman, Tel-Aviv University, **Israel**

16:30 - 17:00	Plasma applications for smart and sustainable agriculture (PIAgri) with the emphasis on biotechnologies N. Puač , O. Jovanović, S. Živković, M. Milutinović, S. Jevremović, G. Malović and N. Škoro	Dr. Nevena Puac , University of Belgrade, Serbia
17:00 - 17:15	Multifunctional transparent oxide thin films for photocatalytic and antimicrobial applications J.G. Cuadra , S. Molina-Prados, G. Mínguez-Vega, A.C. Estrada, T. Trindade, C. Oliveira, M.P. Seabra, J. Labrincha, S. Porcar, R. Cadena, D. Fraga and J.B. Carda	Mr. Jaime González Cuadra , University Jaume I, Spain
17:15 - 17:30	In vitro Analysis of Plasma Activated Water: A Step Toward Effective Plasma Medicine A. Megía-Macías , O.D. Cortázar, S. Moreno, A. Brun, Y. Ballesteros and E. Gómez-Casado	Dr. Ana Megía-Macías , Comillas Pontifical University, Spain
17:30 - 17:45	A Step Forward in Ulcer Management: The Clinical Findings of Cold Plasma Therapy for Torpid Ulcers in Legs A. Megía-Macías, O.D. Cortázar and B. Hontanilla	Dr. Daniel Cortázar , MEDICAL PLASMAS S.L, Spain
17:45 - 18:00	Treatment of microalgae by gliding arc plasma and pulsed electric field L. Marcinauskas , K. Jonynaitė, R. Uscila, A. Stirkė, Ž. Kavaliauskas, M. Aikas, S. Keršulis and V. Stankevič	Prof. Liutauras Marcinauskas , Lithuanian Energy Institute/ Kaunas University of Tech, Lithuania
18:00 - 18:15	Cold Atmospheric Plasma treatment of in-vivo B16/F10 Melanoma cancer cells and the immunological response A.S. Mashayekh , S. Yin, Z. Liu and K. Yan	Mr. Amir Shahriar Mashayekh , Zhejiang University, China
18:15 - 18:30	Plasma Polymerized Nano particles: Synthesis, Diagnostics, and Bio-functionalization L. L. Haidar , M. M. M. Bilek and B. Akhavan	Mrs. Laura Haidar , The University of Sydney, Australia

Plasma applications for smart and sustainable agriculture (PlAgri) with the emphasis on biotechnologies

N. Puač¹, O. Jovanović¹, S. Živković², M. Milutinović², S. Jevremović², G. Malović¹ and N. Škoro¹
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Abstract:

The continuous increase in demand for food caused by population growth represents a serious challenge for humankind. At the same time, the climate change is having significant impact on agricultural production and farmers are forced to adapt or change existing practices due to the change in weather patterns, pest appearance, water availability etc. Improving sustainability of agriculture and reducing adverse effects of agriculture on the environment requires efficient technologies that enhance productivity while maintaining food quality and safety. The main aim is to investigate the potential of non-thermal plasmas as a green alternative to conventional fertilizers in agriculture to improve yields, increase size and robustness of plants, to reduce (or eliminate) the need for pesticides and in the case of final products treatments of food and its packaging.

First studies pointed out that plasma created Reactive Oxygen Nitrogen Species (RONS) observed in treatments of seeds and plants led to successful application of plasmas for different aspects of agriculture and food industry [1, 2]. It was shown that, both low pressure and atmospheric pressure plasmas can be successfully used in stimulation of the seed growth, increase of germination percentage and decontamination, breaking of dormancy or lengthening of the seed sprout. In direct plasma treatments, i.e. where seeds are in direct contact with plasma or its afterglow, the surface of seeds undergoes a variety of changes. Depending on the plasma conditions the surface is activated so other functional groups can be attached (-COOH, -COH, -COO, -NH₂, -OH, -NO etc.) and at the same time seed surface can be etched and/or decontaminated from various types of microbes. Plasma treatment of seeds became one of the starting points in opening of a wide area of applications of plasmas in agriculture and related biotechnologies.

The plants can be grown not just from seeds but also from plant meristematic (stem) cells or from plantlets encapsulated in synthetic seeds. We

have performed the treatments of *Daucus carota* and *Iris reichenbachii* Heuffel. calli by using plasma needle that operates at atmospheric pressure. We induced significant morphological and physiological changes in non-embryonic calli toward somatic embryos (SE) formation that. Another type of discharge was used for treatment of synthetic seeds of several Chrysanthemum varieties. We have used Dielectric Barrier Discharge (DBD) that operates in air at 50Hz frequency. The difficulties related to sowing them under non-sterile conditions (common soil) are considered to be one of the main limitations of the wide practical use of Synthetic Seeds technique. Here we will show the influence of cold plasma treatment on germination, plant growth and development of plantlets sowed in sterile and in ex vitro conditions.

Keywords: atmospheric pressure plasma, Plasma Agriculture, non-equilibrium discharge, plant calli, synthetic seeds.

Acknowledgments: This publication is based upon work from COST Action “Plasma applications for smart and sustainable agriculture (PlAgri)”, supported by COST (European Cooperation in Science and Technology).

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SAPP XXIV

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27 Jan - 1 Feb, 2023

Edited by J. Országh, B. Stachová, D. Mészáros, P. Papp, Š. Matejčík



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DEVELOPMENTS OF PLASMA ACTIVATED LIQUIDS FOR AGRICULTURAL AND WATER TREATMENT APPLICATIONS

Nikola Škoro¹, Olivera Jovanović¹, Amit Kumar¹, Andjelija Petrović¹ and Nevena Puač¹

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In recent years many applications of cold atmospheric plasmas (CAP) are focused on treatment of liquid samples. CAP induces rich chemistry in active plasma volume creating short and long lived reactive species that penetrate and/or react with molecules in the liquid-phase producing short and long-lived species [1]. From the application point of view, the plasma treatment could be used for liquid activation and creation of plasma activated water (PAW) and plasma activated medium (PAM), or for destruction of dissolved pollutant molecules – for decontamination processes. For the latter case, plasma can be used as a chemically free advanced oxidation process (AOP) that can efficiently remove various organic micropollutants (OMP) from water. The major area of interest for PAW and plasma decontamination lie within the field of plasma agriculture [2]. Apart this field, liquid activation has a high potential in plasma medicine. For all these applications, clear knowledge of the entangled connection between plasma properties, plasma chemistry and interaction with the liquid target is necessary in order to tune the plasma for different outcomes of the liquid treatment [3,4]. In this work we will present several kinds of plasma treatments featuring investigation of both plasma and treated liquid properties and their dependence on the plasma jet type, operating parameters etc. Results of treatments of pure and contaminated water (with organic contaminant) were performed by using high-frequency pin-type atmospheric pressure plasma jet operating with He and Ar. For PAW creation, we demonstrated how the plasma operating in different regimes with He as working gas yield different concentrations of reactive nitrogen and oxygen species. Moreover, we investigated the influence of the working gas as well as sample volume to the RONS concentration in the activated water. We observed much higher production of H₂O₂ in case of Ar plasma than in He. For treatments of the cell media used in plasma medicine applications, we used a dielectric barrier discharge (DBD) jet configuration that operated at 80 kHz. We showed that the power deposited to the plasma in contact with target was the key parameter for monitoring the production of RONS in the PAW and PAM. Thus precise power determination is crucial for fine tuning of the produced species.

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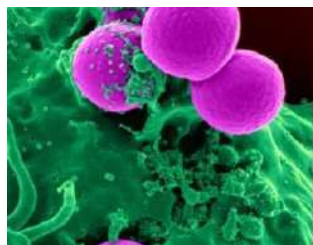
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P3

Varying the plasma-treated liquids' characteristics for applications in biomedicine

Olivera Jovanović^a, Nevena Puač^a, Anđelija Petrović^a, and Nikola Škoro^a

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The development of new plasma sources operating at atmospheric pressure in contact with liquid samples over the past 20 years has mostly been driven by new biomedical applications such as wound healing, cancer treatment, as well as the deactivation of bacteria and viruses and sterilization¹⁻². Recent research is focused on several aspects of plant biology and wastewater treatment³⁻⁴ because low-temperature plasmas are environmentally acceptable and the active gaseous environment is at room temperature.

Plasma Activated Liquids (PALs) are essential in this application. PALs have been used in a substantial number of successful experiments, but there are still many questions regarding the mechanisms governing the interaction between the plasma and liquid including the production of a broad number of chemical species.

In this work, we utilized atmospheric pressure non-equilibrium plasma jets for the treatment of liquids, and the production of PALs. PALs were employed for the treatment of plant tissue culture, then also for application on wound healing in diabetic mice and finally in a study that tested their toxicity after oral intake by rats. Diagnostics of liquid samples were performed to assess the effectiveness of plasma treatment. The analysis of physicochemical parameters was conducted by measuring conductivity, pH, temperature, and the amount of deposited RONS (H_2O_2 , NO_3^- , NO_2^-). The mechanisms of interaction between plasma and liquid samples were determined depending on the conditions in the system. Through this research, we wanted to show that the used plasma systems provide the possibility to adjust the properties of treated liquids for a particular application in biomedicine by changing the treatment conditions.

Acknowledgements: This work was supported by MSTDI Republic of Serbia grant number 451-03-68/2022-14/200024, and The Science Fund of the Republic of Serbia, grant No. 3114/2021 - Project APPerTAin-BIOM.

Keywords: plasma jet, plasma biomedicine, plasma activated liquids

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Can atmospheric pressure plasma technology be an efficient reactive chemistry tool for water treatments?

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In previous years many different studies were demonstrating ability of Cold Atmospheric Plasma (CAP) treatments to produce chemical interaction with various types of liquids. During treatments short-lived Reactive Oxygen and Nitrogen (RONS) Species generated in plasma (e.g. OH, O, O₃, NO etc.) interact with liquid target in such a way that, after the treatments, long-lived RONS are deposited and preserved in the samples. In case of pure water treatment, the obtained plasma activated water (PAW) contains species such as H₂O₂, ONOOH, NO_3^- , NO₂ [1]. Type of the reactive species depend considerably on type of plasma source, operating parameters but also on the liquid target properties (mostly conductivity) and even on the shape and the size of the sample container. Thus, linking the plasma chemistry with the obtained RONS content is an entangled task [2] and changes in the plasma treatment system could result in unanticipated changes in properties of the liquid sample. In this work, studies related to application of CAP formed over the water samples dealing with both decontamination [3] and production of PAW will be presented. We investigated several types of CAP sources for water treatment: pin-type jet, DBD jet and MW source.

As an illustration, comparison PAW created from distilled H₂O by pin-jet and DBD jet is given Fig.1 Power delivered to the plasma was much smaller for DBD jet (approx. 10 times).

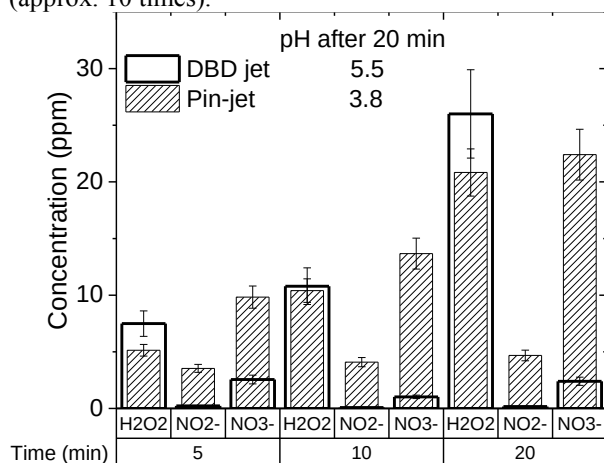


Figure 1. Measurements of RONS concentrations in PAW obtained from dH₂O for 3 treatment times. Plasma formed in air with He as working gas was used in both cases.

In both cases He (2 slm) was used as working gas producing a streamer discharge. The vessel with distilled water was placed 10 mm below the plasma source. The treated volume was 4 ml in the pin-jet case and 2 ml for

DBD jet. Just from H₂O₂ concentrations it could seem that changes in the sample volumes could yield similar PAW properties obtained in different systems, but NO_x concentrations and their ratios as well as pH are completely different. So, very similar gas phase plasma chemistry eventually results in completely different PAW properties which has direct influence on further application.

That is one of the reasons why PAW produced with different systems was used in different targeted applications - treating water polluted with micro organic pollutants as well as PAW produced for plant treatment (investigation of PAW influence to aquatic plant Duckweed). We exploited the combination between results of plasma diagnostics such as electrical characterization, optical emission spectroscopy and imaging, and treated physico-chemical liquid properties in order to select important input parameters and tailor desired treatment conditions for obtaining PAW with the properties suitable for particular application. Apart from creating different RONS concentrations we also studied pH adjustment as generating PAW with less acidic conditions can be beneficiary for some applications where low pH can induce negative effects (e.g. application related to seeds and plants).

When CAP is positioned above the water surface the effective surface that is in direct contact with the discharge is generally small. In our experiments we investigated increase of contact surface by increasing the plasma area with making a 3-pin jet and also by introducing recirculation of water in the system. Focusing on establishing relations between different discharge operating regimes, produced plasma-liquid chemistry and the obtained effects in applications we obtained information important for advancing towards achieving plasma reactivity at a larger scale, which is necessary step for many water-related applications.

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Plasma treatment of water for agricultural and water decontamination applications

PP1-11

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In recent years many applications of cold atmospheric plasmas (CAP) are focused on treatment of liquid samples. CAP induces reactive chemistry and create reactive species in the gas phase that penetrate and/or react with molecules in the liquid phase producing short and long-lived oxygen and nitrogen species. Regarding applications, the plasma treatment could be used for creation of plasma activated water (PAW) from pure water or for decontamination of polluted water. For all these applications that lie within the field of plasma agriculture clear knowledge of the complicated connections between plasma properties, reactive chemistry and interaction with the liquids is necessary to tune the PAW properties. In this work we will present several kinds of plasma treatments using a plasma jet type with pin-electrode (powered by 350 kHz high-voltage signal) and featuring investigation of both plasma and treated liquid properties and their dependence operating parameters. For PAW creation, we demonstrated how different He plasma regimes yield different concentrations of reactive species. Moreover, we investigated the influence of the working gas and sample volume to the RONS concentration in PAW. Much higher production of H₂O₂ was observed in case of Ar plasma than in He. We used PAW for treatment of the model plant system *Arabidopsis thaliana* and showed by using molecular approaches how PAW treatment altered expression of genes coding for specialized metabolites and hormones, thus regulating development and stress responses. Finally, the results of treatment of contaminated water by using high-frequency pin-type CAP jet showed efficient water decontamination.

Keywords: cold atmospheric plasma, plasma activated water, reactive species, water decontamination, plant metabolites

Acknowledgement: This work was supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia, Grant No. 451-03-68/2024-14/200024 and 451-03-68/2024-14/200007 and the Science Fund of the Republic of Serbia, Grant No. 3114/2021 - Project APPerTain-BIOM and 14916/2024 – Project PoWeRCAP-EGARDEN.

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Investigation of toxicity effects of Plasma Activated Water on a water model plant *Lemna Minor*

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Non-thermal atmospheric pressure plasmas play a crucial role in biomedical applications due to the highly reactive chemistry of Reactive Oxygen and Nitrogen Species (RONS) they produce in gas phase [1, 2]. When treating water, RONS produced in the gas phase interact with the liquid interface, creating Plasma Activated Water (PAW). The RONS concentration can be tailored by changing plasma chemistry in the gas phase, water type, sample vessel properties etc. In recent years PAW is being developed as part of a new field of plasma agricultural applications. When PAW is used as a watering agent it results in a better yield of plants and has a positive effect on germination percentage [3,4]. However, the toxicity of PAW and its impact on the environment if it is released in underground or fresh water has not yet been investigated in detail.

In this work, we used a plasma jet with pin electrode operating at atmospheric pressure to obtain PAW by treating deionized and tap water. The plasma jet was powered by a kHz sine signal and used Ar as a working gas, with a total gas flow of 1 slm. To investigate the toxicity of PAW, the primary goal was to produce a liquid with the highest amount of the measured reactive species (H_2O_2 , NO_3^- , NO_2^-). Deionized water treatments have demonstrated that, independent of the amount of treated sample, the pH of the water decreases significantly, which can be hazardous to plants. To eliminate the possible toxicity of PAW due to acidity, we selected to treat tap water, which retained its pH value even after treatment. Finally, PAW was obtained by treating 15 ml of tap water for 10 minutes.

The selected PAW was combined with two different matrices, namely tap water and Danube (river) water which were introduced by making PAW dilutions. Four different dilutions (100%, 60%, 30%, and 10%) were prepared for both matrices to assess their impact on *Lemna minor* samples. The plants were cultivated in beakers containing 25 ml of liquid, maintaining controlled conditions, such as temperature, humidity and daylight cycle, for a period of 17 days. The root lengths, leaf surface area, and chlorophyll percentage were then measured. Each experimental set was carried out in triplicate. The results showed that a smaller amount of PAW had a beneficial impact, whereas greater percentages were completely toxic. In all PAW samples, root lengths were reduced but leaf surface area increased. Notably, PAW diluted to 10% and 30% in either tap water or Danube water resulted in a higher chlorophyll percentage than the control samples.

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21st International Congress on Plasma Physics

September 8-13, 2024
Ghent, Belgium

Program & Abstracts



fwo



*inf*usion



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Foreword

Dear Colleagues,

On behalf of the Local Organizing Committee, the Program Committee, and the International Advisory Committee, we sincerely welcome you to the 21st International Congress on Plasma Physics (ICPP 2024), in Ghent. The event is organized by the Research Unit Nuclear Fusion (infusion) and the Research Unit Plasma Technology (RUPT) in the department of Applied Physics at the faculty of Engineering and Architecture of Ghent University (UGent).

We are very pleased to welcome a large number of distinguished presenters who will address a wide range of topics in the field of plasma physics. With participants hailing from 30 different countries all over the world, the conference will be a forum for discussion of cutting-edge research in fundamental plasma physics, fusion plasmas, laser-plasma interaction, plasma accelerators, astrophysical and space plasmas, applications of plasmas and plasma technology, complex plasmas, high energy density plasmas and quantum plasmas.

In addition, we are proud to offer you a plenary session on Public-Private Partnerships in Fusion Energy Development (Monday, September 9), in the form of a panel discussion among experts in the field. Furthermore, there will be an informal interest-group meeting regarding community engagement in repository and management of materials fundamental data for (fusion) plasma physics (Thursday, September 12). We invite all interested conference participants to attend these sessions.

We are deeply saddened by the premature decease of Prof. Robert (Bob) Dewar (Australian National University), who made an immense contribution to the field of theoretical plasma physics. Bob also had a significant presence at the ICPP and, in recognition of this service and commitment, a plenary memorial session celebrating his accomplishments will be held on Tuesday morning, September 10.

Finally, we hope that you take the occasion to explore Ghent as a vibrant city with an extensive historical center and an abundant architectural and cultural patrimony.

We welcome you here, to enjoy with us the conference, the many enchanting sights and the rich culinary tradition of the region!

Geert Verdoolaege

Chair of LOC

Professor, Ghent University

Bob Bingham

Chair of PC

Professor, Science and Technology Facilities Council (STFC)

Sponsoring

ICPP 2024 gratefully acknowledges its sponsors.



The [International Union of Pure and Applied Physics](#) (IUPAP) is the only international physics organization that is organized and run by the physics community itself. Its members are identified physics communities in countries or regions around the world.

To secure IUPAP sponsorship, the organizers have provided assurance that ICPP 2024 will be conducted in accordance with IUPAP principles as stated in the IUPAP resolution passed by the General Assembly in 2008 and 2011. In particular, no bona fide scientist will be excluded from participation on the grounds of national origin, nationality, or political considerations unrelated to science.



The [Research Foundation Flanders](#) (FWO) offers researchers in Flanders the opportunity to create knowledge. The FWO provides financial support for individual researchers, programmes and projects, and research infrastructure.

We are also very grateful to Prof. Hyeon K. Park and the other members of the local organizing committee of the ICPP 2022 conference, for the generous additional support.

Code of conduct

Free circulation of scientists

The principle of the Universality of Science is fundamental to scientific progress. This principle embodies freedom of movement, association, expression and communication for scientists, as well as equitable access to data, information and research materials.

In pursuing its objectives with respect to the rights and responsibilities of scientists, the International Union of Pure and Applied Physics (IUPAP) actively upholds this principle, and, in so doing, opposes any discrimination on the basis of such factors as ethnic origin, religion, citizenship, language, political stance, gender, or age. IUPAP should only sponsor conferences and events at institutions and in countries that uphold this principle. If scientists are excluded from attending IUPAP-sponsored international conferences by a host institution or country on the basis of any of these factors, IUPAP should register its concern at the highest level of that institution or country, and should not sponsor any future events in that country until such exclusions have been eliminated.

Harassment

It is the policy of the International Union of Pure and Applied Physics (IUPAP) that all participants in Union activities will enjoy an environment which encourages the free expression and exchange of scientific ideas, and is free from all forms of discrimination, harassment, and retaliation. The conference organisers have named an advisor who will consult with those who have suffered from harassment and who will suggest ways of redressing their problems, and an advisor who will counsel those accused of harassment. The conference organisers may, after due consideration, take such action they deem appropriate.

Committees

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Qiu-Gang Zong	Peking University	China

Local Organizing Committee

The Local Organizing Committee is chaired by Prof. Geert Verdoolaege and consists of the members of the [Research Unit Nuclear Fusion](#) (infusion) and the [Research Unit Plasma Technology](#) (RUPT) in the Department of Applied Physics at Ghent University.

Locations

Conference locations

Registration, coffee breaks and welcome reception: Ufo building, Foyer
Sint-Pietersnieuwstraat 33

Plenary sessions: Ufo building, Aud. 1 "Leon De Meyer"
Sint-Pietersnieuwstraat 33

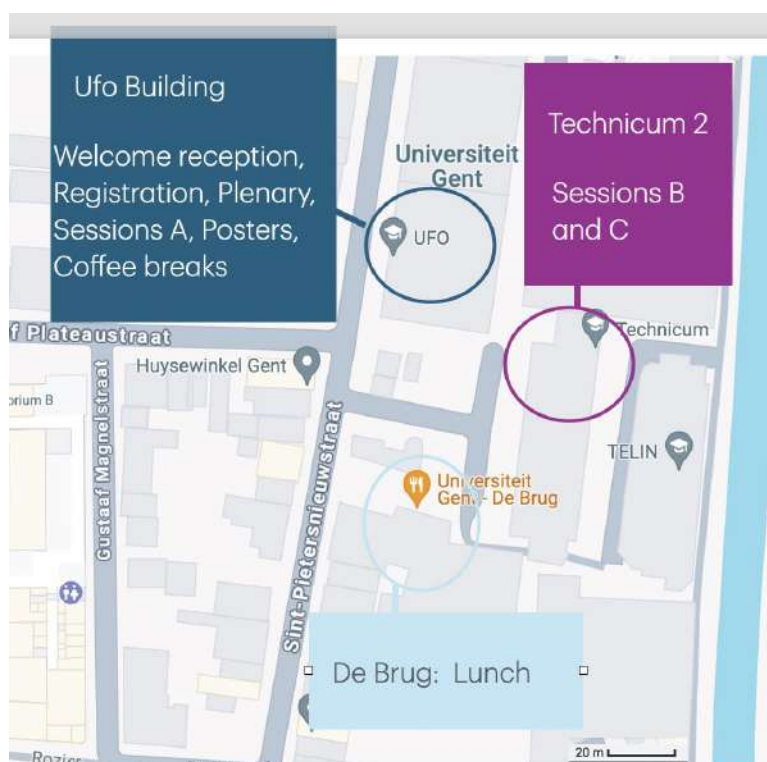
Sessions A: Ufo, Aud. 1 "Leon De Meyer"
Sint-Pietersnieuwstraat 33

Sessions B: Technicum building 2 (T2), floor 3
Aud. H
Sint-Pietersnieuwstraat 33

Sessions C: Technicum building 2 (T2), floor -3
Aud. A "Jean-Norbert Cloquet"
Sint-Pietersnieuwstraat 33

Poster sessions: Ufo building, Foyer
Sint-Pietersnieuwstraat 33

Lunch: student restaurant "De Brug"
Sint-Pietersnieuwstraat 45



Internet

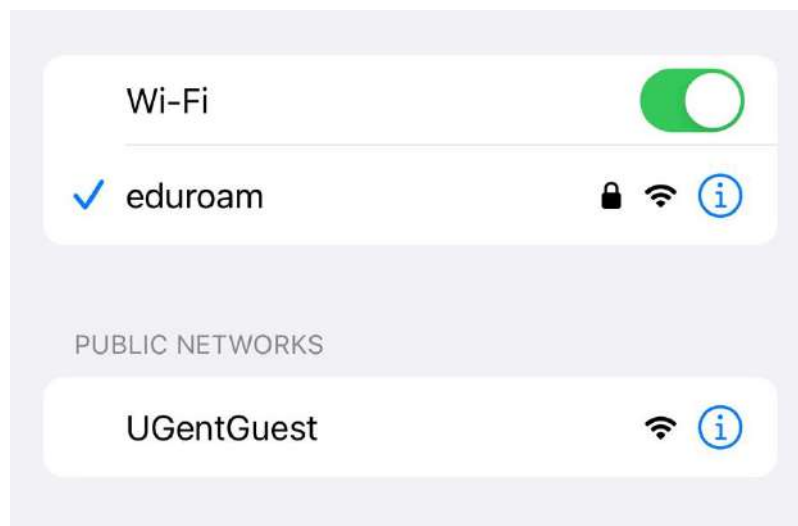
Wi-Fi internet connection is available at the venue. The credentials are:

Network: UGentGuest

Username: guestlcpp24

Password: gVmU8Lvg

The attendees may also connect via **Eduroam** using the credentials provided by their respective institutes.



Program overview

Sunday, September 8

13:00 - 18:00	Registration
18:00 - 20:00	Welcome reception

Monday, September 9

08:00 - 08:45	Registration		
08:45 - 09:00	Opening Ceremony		
09:00 - 09:40	[Mo.P.1] Frank Jenko, Towards Digital Twins of Fusion Systems		
09:40 - 10:20	[Mo.P.2] Rahul Pandit, The Solar Wind and Statistical Properties of Three-Dimensional Hall Magnetohydrodynamics Turbulence		
10:20 - 10:50	Coffee break		
10:50 - 11:30	[Mo.P.3] Jozsef Ongena, Overview and Initial Results of the ICRII Antenna for the Optimized Stellarator Wendelstein 7-X		
11:30 - 11:40	Short break		
11:40 - 12:30	[Mo1.A] Instabilities in Fusion Plasmas	[Mo1.B] Modelling for Plasma Processing Applications	[Mo1.C] Magnetic Reconnection in Space and Astrophysical Plasmas
	[Mo1.A.1] Hao Wang	[Mo1.B.1] Igor Kaganovich	[Mo1.C.1] Dmitri Uzdensky
	[Mo1.A.01] Aloisio Grondin-Edrington	[Mo1.B.02] Sanli Sivami (remote)	[Mo1.C.01] Kuldeep Singh
12:30 - 14:00	Lunch		
14:00 - 15:30	[Mo2.A] Fusion Plasma Modelling	[Mo2.B] Technological Applications of Plasmas	[Mo2.C] Solar Wind and Solar Physics
	[Mo2.A.1] Hideto Sugama	[Mo2.B.1] Rony Snyder	[Mo2.C.01] Sebastian Echeverria-Veras (remote)
	[Mo2.A.01] Richard Sydora	[Mo2.B.02] Leopoldo Soto	[Mo2.C.02] Victor Muñoz
	[Mo2.A.02] W. Guo	[Mo2.B.02] Jasna-Tijana Jelinsek	
	[Mo2.A.03] Arzoo Mahal (remote)	[Mo2.B.03] Moin Chaudhuri	
15:30 - 16:00	Coffee break		
16:00 - 17:40	[Mo3.A] Energetic Particles and Plasma Heating	[Mo3.B] Plasma Applications in Environmental and Agricultural Sciences	[Mo3.C] Numerical Methods and Plasma Modelling I
	[Mo3.A.1] Yevgen Kazakov	[Mo3.B.1] Alan Maššini	[Mo3.C.01] Supratik Banerjee
	[Mo3.A.12] Kenji Imadera	[Mo3.B.01] Alexandr Ustimenko	[Mo3.C.02] Anji Helder
	[Mo3.A.01] Julio Martinelli	[Mo3.B.02] Toshiro Kaneko	[Mo3.C.03] Mario Imbrogno
	[Mo3.A.02] Kohnosuke Sato		[Mo3.C.04] Xudong Ke Lin
17:40 - 17:50	Short break		
17:50 - 18:50	[Mo.P.4] Public-Private Partnerships in Fusion Energy Development		

Tuesday, September 10

08:00 - 09:00	Registration		
09:00 - 09:40	[Tu.P.1] Nicholas Dubil, Complex Interplay of Magnetic Islands and Turbulence in Fusion Plasmas		
09:40 - 10:20	[Tu.P.2] Christopher Denney, Pioneering High-Energy-Density Plasma Physics at the Laboratory for Laser Energetics		
10:20 - 10:40	Coffee break		
10:40 - 12:30	[Tu1.A] Bob Dewar Memorial Session		
12:30 - 14:00	Lunch		
14:40 - 15:40	[Tu2.A] Magnetic Reconnection in Fusion Plasmas	[Tu2.B] Plasma Accelerators I	[Tu2.C] Numerical Methods and Plasma Modelling II
	[Tu2.A.1] Yasushi Ono	[Tu2.B.1] Colin Whyte	[Tu2.C.01] Takayuki Umeda
	[Tu2.A.12] Hiroshi Taniabe	[Tu2.B.02] Devki Nandan Gupta	[Tu2.C.02] Harune Sekido
15:40 - 16:00	Coffee break		
16:00 - 17:20	[Tu3.A] Spherical Tokamaks	[Tu3.B] Laser-Plasma Interaction I	[Tu3.C] Nonextensive Plasma Physics
	[Tu3.A.1] Doniel Kennedy	[Tu3.B.1] Robert Batjak	[Tu3.C.01] Victor Muñoz
	[Tu3.A.12] Kazuaki Hanada	[Tu3.B.12] Dominika Maslova	[Tu3.C.02] Sebastian Seidvitz (remote)
		[Tu3.B.02] Ravina	
Poster Session 1 14:00 - 18:00			

Wednesday, September 11

08:00 - 09:00	Registration		
09:00 - 09:40	[We.P.1] Didier Maizot, Developing Numerical Tools for Tungsten Profile Measurement from X-Ray Diagnostics in WEST Plasmas		
09:40 - 10:20	[We.P.2] Eve Stenison, Highlights From the Path Toward Confined e+e- Pair Plasmas		
10:20 - 10:50	Coffee break		
10:50 - 11:30	[We.P.3] Emilia R. Solano, IET Isotope Studies and the L-H Transition		
11:30 - 11:40	Short break		
11:40 - 12:30	[We1.A] Data-Driven Plasma Science I	[We1.B] Laser-Plasma Interaction II	[We1.C] Astrophysical Plasmas I
	[We1.A.11] Zetseu Lin	[We1.B.11] Sylvie Depierreux	[We1.C.11] Taroough Downes
	[We1.A.01] Hao Wu	[We1.B.01] Vladimir Kochanovsky	[We1.C.01] Nicolas Bruggmans
12:30 - 14:00	Lunch		
14:30 - 17:30	Excursion		

Thursday, September 12

08:00 - 09:00	Registration		
09:00 - 09:40	[Th.P.1] Tammy Ma, Ignition Achieved: Next Steps in the Path toward an Inertial Fusion Energy Future		
09:40 - 10:20	[Th.P.2] Victor Malka, Laser Plasma Accelerators: Manipulating Relativistic Electrons with Intense Lasers		
10:20 - 10:50	Coffee break		
10:50 - 11:30	[Th.P.3] Osamu Sakai, Design of Plasma Shapes for Mercamaterials, Chemical Filters, and Maze-Solvers		
11:30 - 11:40	Short break		
11:40 - 12:40	[Th1.A] Plasma-Wall Interaction and Plasma-Facing Components	[Th1.B] Laser Applications	[Th1.C] Astrophysical Plasmas II
	[Th1.A.11] Renaud Delamaré	[Th1.B.11] Jessica Shaw	[Th1.C.11] Vinicius Duarte (IUPAP Early Career Scientist Prize)
	[Th1.A.01] Henri Kumpulainen	[Th1.B.01] Minsoo Hur	[Th1.C.01] Anoop Singh
12:40 - 14:00	Lunch		
14:40 - 15:30	[Th2.A] Edge-Localized Modes	[Th2.B] Materials Applications	[Th2.C] Data-Driven Plasma Science II
	[Th2.A.01] Jaehyun Lee	[Th2.B.11] Mark Koepke	[Th2.C.11] E. Paulo Alves
	[Th2.A.02] Tripur Kumar Borthakur	[Th2.B.01] Noriyasu Ohno	[Th2.C.01] Maria Jose Quezada Roco
15:30 - 16:00	Coffee break		
16:00 - 17:30	[Th3.A] Tokamak Scenarios and Ignition	[Th3.B] Laser-Plasma Interaction III	[Th3.C] Dusty Plasmas
	[Th3.A.11] Luca Garzotti	[Th3.B.11] Thales Silva	[Th3.C.11] Hubertus M. Thomas
	[Th3.A.02] Eun-Jin Kim	[Th3.B.01] Amit Dattotraya Led	[Th3.C.01] Rami Prasad Prajapati (remote)
	[Th3.A.01] Hyeon Park	[Th3.B.02] Rohit Juneja	[Th3.C.02] Sven Van Lee
17:30 - 17:40	Short break		
17:40 - 18:30	[Th.P.4] RM3FD Interest Group Meeting		
19:00 - 23:00	Conference banquet		

Poster Session 2
14:00 - 18:00

Friday, September 13

08:00 - 09:40	Registration		
09:40 - 10:20	[Fr.P.1] G. Ravindra Kumar, 10 ¹⁴ -Fold Field Amplification and Control of Relativistic Mega-Ampere Electron Beams in a Modest, Static Magnetic Solid		
10:20 - 10:50	Coffee break		
10:50 - 11:30	[Fr.P.2] Gianluca Gregori, Suppression of Pair Beam Instabilities in a Laboratory Analogue of Blazar Pair Cocorides		
11:30 - 11:40	Short break		
11:40 - 12:30	[Fr1.A] Tokamak Physics	[Fr1.B] Plasma Accelerators II and Laser-Plasma Interaction IV	[Fr1.C] Ionospheric Plasmas
	[Fr1.A.01] Aneel Kumar	[Fr1.B.11] Krishnamurthy Manickam	[Fr1.C.01] Anish Tawarwal
		[Fr1.B.01] Anish Dutt	[Fr1.C.02] Ioannis Kourakis
12:30 - 12:45	Closing Ceremony		
12:45 - 14:00	Lunch		

Session chairs

Monday, September 9			
Session	Start time	Chair	Location
Opening Ceremony	08:45	G. Verdoolaege	Ufo, Aud. 1 "Leon De Meyer"
[Mo.P.1] Plenary 1	09:00	S. Mohamed-Benkadda	
[Mo.P.2] Plenary 2	09:40	S. Mohamed-Benkadda	
[Mo.P.3] Plenary 3	10:50	G. Verdoolaege	
[Mo1.A] Instabilities in Fusion Plasmas	11:40	A. Das	Ufo, Aud. 1 "Leon De Meyer"
[Mo2.A] Fusion Plasma Modelling	14:00	Y. Ono	
[Mo3.A] Energetic Particles and Plasma Heating	16:00	L. Soto	
[Mo1.B] Modelling for Plasma Processing Applications	11:40	C. Leys	Technicum building 2 (T2), Aud. H
[Mo2.B] Technological Applications of Plasmas	14:00	A. Nikiforov	
[Mo3.B] Plasma Applications in Environmental and Agricultural Sciences	16:00	T. Kaneko M. Gromov	
[Mo1.C] Magnetic Reconnection in Space and Astrophysical Plasmas	11:40	R. Sydora	Technicum building 2 (T2), Aud. A "Jean-Norbert Cloquet"
[Mo2.C] Solar Wind and Solar Physics	14:00	S. Van Loo	
[Mo3.C] Numerical Methods and Plasma Modelling I	16:00	L. Soto	
[Mo.P.4] Public-Private Partnerships in Fusion Energy Development	17:50	M. Koepke	Ufo, Aud. 1 "Leon De Meyer"

Tuesday, September 10			
Session	Start time	Chair	Location
[Tu.P.1] Plenary 4	09:00	J. Martinell	Ufo, Aud. 1 "Leon De Meyer"
[Tu.P.2] Plenary 5	09:40	J. Martinell	
[Tu1.A] Bob Dewar Memorial Session	10:40	R. Mackay M. Hole	Ufo, Aud. 1 "Leon De Meyer"
[Tu2.A] Magnetic Reconnection in Fusion Plasmas	14:40	R. Sydora	
[Tu3.A] Spherical Tokamaks	16:00	N. Ohno	
[Tu2.B] Plasma Accelerators I	14:40	B. Bingham	Technicum building 2 (T2), Aud. H
[Tu3.B] Laser-Plasma Interaction I	16:00	K. Sato	
[Tu2.C] Numerical Methods and Plasma Modelling II	14:40	S. Mohamed-Benkadda	Technicum building 2 (T2), Aud. A "Jean-Norbert Cloquet"
[Tu3.C] Nonextensive Plasma Physics	16:00	F. Verheest	
Poster Session 1	14:00	Y. Zhang	Ufo, Foyer

Wednesday, September 11

Session	Start time	Chair	Location
[We.P.1] Plenary 6	09:00	G. Verdoolaege	Ufo, Aud. 1 "Leon De Meyer"
[We.P.2] Plenary 7	09:40	G. Verdoolaege	
[We.P.3] Plenary 8	10:50	G. Verdoolaege	
[We1.A] Data-Driven Plasma Science I	11:40	M. Hole	Ufo, Aud. 1 "Leon De Meyer"
[We1.B] Laser-Plasma Interaction II	11:40	T. Kaneko	Technicum building 2 (T2), Aud. H
[We1.C] Astrophysical Plasmas I	11:40	S. Van Loo	Technicum building 2 (T2), Aud. A "Jean-Norbert Cloquet"
Excursion	14:30	G. Verdoolaege	

Thursday, September 12

Session	Start time	Chair	Location
[Th.P.1] Plenary 9	09:00	B. Bingham	Ufo, Aud. 1 "Leon De Meyer"
[Th.P.2] Plenary 10	09:40	B. Bingham	
[Th.P.3] Plenary 11	10:50	M. Narimisa	
[Th1.A] Plasma-Wall Interaction and Plasma-Facing Components	11:40	N. Ohno	Ufo, Aud. 1 "Leon De Meyer"
[Th2.A] Edge-Localized Modes	14:40	K. Sato	
[Th3.A] Tokamak Scenarios and Ignition	16:00	M. Hole	
[Th1.B] Laser Applications	11:40	C. Leys	Technicum building 2 (T2), Aud. H
[Th2.B] Materials Applications	14:40	R. Ghobeira	
[Th3.B] Laser-Plasma Interaction III	16:00	J. Martinell	
[Th1.C] Astrophysical Plasmas II	11:40	A. Das	Technicum building 2 (T2), Aud. A "Jean-Norbert Cloquet"
[Th2.C] Data-Driven Plasma Science II	14:40	G. Verdoolaege	
[Th3.C] Dusty Plasmas	16:00	F. Verheest	
Poster Session 2	14:00	Y. Zhang	Ufo, Foyer
[Th.P.4] RM3FD Interest-Group Meeting	17:40	M. Koepke	Ufo, Aud. 1 "Leon De Meyer"
Conference banquet	19:00	A. Das	Oude Vismijn (Sint-Veerleplein 5)

Friday, September 13

Session	Start time	Chair	Location
[Fr.P.1] Plenary 12	09:40	Y. Ono	Ufo, Aud. 1 "Leon De Meyer"
[Fr.P.2] Plenary 13	10:50	Y. Ono	
[Fr1.A] Tokamak Physics	11:40	Y. Zhang	Ufo, Aud. 1 "Leon De Meyer"
[Fr1.B] Plasma Accelerators II and Laser-Plasma Interaction IV	11:40	K. Sato	Technicum building 2 (T2), Aud. H
[Fr1.C] Ionospheric Plasmas	11:40	S. Van Loo	Technicum building 2 (T2), Aud. A "Jean-Norbert Cloquet"
Closing Ceremony	12:30	B. Bingham	Ufo, Aud. 1 "Leon De Meyer"

Special session Monday:

Public-Private Partnerships in Fusion Energy Development

Public-private partnerships (PPP) allow large-scale government projects, such as roads, bridges, or hospitals, to be completed with private funding. These partnerships work well when private-sector technology and innovation combine with public-sector incentives to complete work on time and within budget.

A recent rapid increase in private-sector involvement in fusion R&D is a growing global trend with private funding being invested into companies based in Canada, UK, EU, China, Japan, and Australia.

This ICPP 2024 special session on Public-Private Partnerships in Fusion Energy Development takes place on **Monday 9 September 2024, 17:50-18:50 CEST** in the **plenary room** (Ufo, Aud. 1). PPP programs now exist having the following guiding principles:

- Public- and private-sector organizations must be able to fulfill their own institutional missions while also collaborating in a public-private partnership;
- Promote diversity in science & technology approaches to fusion energy;
- Government acts similar to an investor by selecting a portfolio that diversifies both technical and commercial risk;
- All applicants must devote a meaningful fraction of the total project cost from non-governmental funding sources;
- Flexibilities in contracting terms (including but not limited to intellectual property, data protection, etc.) and reduced reporting and cost accounting requirements.

The assembled panel is populated with subject matter experts who will share perspectives on present and future steps worth considering when capitalizing on the PPP approach.

Start 17:50 CEST

1. Welcome – Mark Koepke, West Virginia University
2. PPP's federal framework and goals – Colleen Nehl, U.S. Department of Energy [remote]
3. PPP's realization and impact within the fusion community's private sector – Andrew Holland, CEO, Fusion Industry Association [remote]
4. PPP plan and promise – Ambrogio Fasoli, Programme Manager, EUROfusion [remote]
5. PPP role in roadmapping high-gain inertial fusion energy development – Peter Norreys, Professor of Inertial Fusion Science, University of Oxford [remote or in-person]
6. PPP for collaboration and innovation benefit – Chris Deeney, Director, Laboratory for Laser Energetics, University of Rochester
7. PPP vision for success in inertial fusion energy – Tammy Ma, Lead for the LLNL Inertial Fusion Energy Institutional Initiative
8. Aspirational highlights, targeted metrics of PPP – Open question
9. Question and answer opportunity

End 18:50 CEST

Special session Thursday: RM3FD Interest-Group Meeting

This ICPP 2024 special session will consist of an informal interest-group meeting to facilitate community engagement, coordinate communication efforts, and build trust among stakeholders in the area of materials for the fusion energy community. It will be held on **Thursday 12 September 2024, 17:40-18:30 CEST** in the **plenary room** (Ufo, Aud. 1). This session precedes the 19h conference banquet (walking to the banquet involves a 20-minute walk from the Ufo building).

The aim of this open meeting is the recruitment of users, partners, databases, and facilities. This cooperation and/or collaboration will focus on enabling the fusion research community in accelerating the development of fusion energy through the repository, curation, management of materials fundamental data and the effective development and deployment of data-driven methodologies and cutting-edge computational resources.

The desired overall outcome is the creation of a comprehensive data infrastructure, within 3-4 years, that supports fusion energy research by providing federated access to heterogeneous data sources, advanced data processing tools, and integrated workflows for simulation, experimental, and engineering data.

The scope of the fusion data resource includes:

- Development and implementation of a comprehensive network of users and data providers;
- Productive coordination with conference organizers, journal editors, and international programs;
- Two-way communication to customize functionality and minimize response time for users;
- Monitor and report on best practices of tested or adopted methodologies;
- Evolve and refine the infrastructure as the data ecosystem expands and results are interpreted.

We seek a cross-cutting platform and hub aimed at accelerating the transfer of foundational open-science research into fusion energy solutions. Specifically, we want to enable the fusion energy mission by providing community-based solutions for the timely and efficient sharing of information, data, models and workflows between government lab scientists, individuals in academia, and commercialization-oriented teams in private industry.

The resource should (a) provide the data infrastructure and standards that will serve as the “community commons” for the efficient sharing of data, models, workflows and metadata, (b) provide the tools for the rapid discoverability of shared information, including capabilities for data aggregation, curation, analysis and visualization, and (c) provide the application interfaces for the seamless integration of data into user-defined workflows.

Start 17:40 CEST

1. Welcome – Mark Koepke, West Virginia University (FEDER Co-PI)
2. Data Ecosystem and Repository management – Raffi Nazikian, General Atomics (FEDER PI) [remote]
3. Building and operating the Data Ecosystem and Repository – Brian Sammulu, General Atomics (FEDER Co-PI) [remote]
4. Challenges in operating the Data Ecosystem and Repository – Frank Wuerthwein, San Diego Supercomputer Center at University of California-San Diego (FEDER facility partner) [remote]
5. Important features desired by the data community – Open question
6. Aspirational highlights, targeted metrics & foreseen impact within plasma physics – Open question
7. Question and answer opportunity

End 18:30 CEST

Social program

Welcome reception: Sunday, Sept. 8, 18:00 – 20:00

The reception will take place in the Foyer of the main conference building Ufo (Sint-Pietersnieuwstraat 33).



Excursion: Wednesday, Sept. 11, 14:30 – 17:30

Join us for a guided walking tour through the historical city center of Ghent, followed by a boat tour on the rivers and canals.

We will depart from the main conference building Ufo (Sint-Pietersnieuwstraat 33). Please check your badge for your group numbers.



Conference banquet: Thursday, Sept. 12, 19:00 – 23:00

The conference banquet will be held at the “Oude Vismijn” (old fish market), Sint-Veerleplein 5.
Please bring your badge.



Abstracts

Monday
September 9

Towards Digital Twins of Fusion Systems

Frank Jenko (Max Planck Institute for Plasma Physics).

Abstract

With the world's energy needs expected to rise by a factor of at least 4-5 until the end of the 21st century, and all existing options to cover these demands known to be subject to various disadvantages and limitations, fusion energy provides an attractive additional opportunity. As we edge closer to realizing practical fusion power, the integration of advanced computational tools becomes essential. This talk will explore the concept and development of digital twins in the context of fusion systems, highlighting their potential to revolutionize the design, optimization, and operation of fusion reactors.

Digital twins – virtual replicas of physical systems – enable real-time monitoring and simulation, providing a dynamic and predictive insight into system behavior under various conditions. For fusion systems, digital twins can simulate plasma behavior, reactor materials, and subsystems, facilitating enhanced plasma operation and control strategies, thus significantly reducing downtime and operational costs.

The talk will cover the foundational elements required to develop digital twins for fusion systems, including multi-fidelity modeling, data integration, and advanced algorithms for real-time data processing and decision-making. Emphasis will be placed on the challenges unique to fusion systems, such as the complex and highly nonlinear nature of plasma dynamics. Case studies of ongoing projects and collaborations in the fusion research community will be presented to illustrate the practical applications and benefits of digital twins.

The talk will also address future directions, emphasizing the role of artificial intelligence and machine learning in enhancing the predictive capabilities of digital twins. By leveraging these technologies, we can accelerate the path to achieving reliable and economically viable fusion energy.

The Solar Wind and Statistical Properties of Three-Dimensional Hall Magnetohydrodynamics Turbulence

Rahul Pandit (Department of Physics, Indian Institute of Science), Sharad Kumar Yadav (Department of Physics, Sardar Vallabhbhai National Institute of Technology) and Hideaki Miura (National Institute for Fusion Science).

Abstract

The characterization of the statistical properties of solar-wind turbulence is a major challenge in solar plasma physics. Satellite observations have shown that solar-wind-plasma turbulence displays a fluid energy spectrum with an inertial range with a scaling exponent that is consistent with $-5/3$, which follows from the Kolmogorov hypotheses of 1941 (henceforth K41). By contrast, the magnetic-energy spectrum displays two different scaling ranges (the first and second inertial ranges): (i) the first inertial range is consistent with the K41 power; (ii) the second inertial range is characterised by an exponent that lies in the range $[-4, -1]$. Furthermore, solar-wind-turbulence has also been analyzed to uncover (a) intermittency and multiscaling of velocity and magnetic-field structure functions and (b) the alignment of velocity and magnetic-field fluctuations. One study has found structure-function multiscaling (simple scaling), in the first (second) inertial range. We examine the statistical properties of three-dimensional (3D) Hall magnetohydrodynamics (HMHD) turbulence by carrying out high-resolution pseudospectral direct numerical simulations. We explore the dependence of 3D HMHD turbulence on the Reynolds number Re and the ion-inertial scale. We present several statistical properties of 3D HMHD turbulence, which we also compare with their counterparts for 3D MHD turbulence and solar-wind turbulence by calculating (a) the temporal evolution of the energy-dissipation rates and the energy, (b) the wave-number dependence of fluid and magnetic spectra, (c) the probability distribution functions of the cosines of the angles between various pairs of vectors, such as the velocity and the magnetic field, and (d) various measures of the intermittency in 3D HMHD turbulence.

Overview and Initial Results of the ICRH Antenna for the Optimized Stellarator Wendelstein 7-X

Jozef Ongena (LPP-ERM/KMS), Ye. O Kzakov (LPP-ERM/KMS), K. Crombé (LPP-ERM/KMS Brussels, Ghent University), D. Hartmann (Max-Planck-Institut für Plasmaphysik), D. Castaño-Bardawil (LPP-ERM/KMS), D. Lopez-Rodriguez (LPP-ERM/KMS, Ghent University), I. Stepanov (LPP-ERM/KMS), M. Verstraeten (LPP-ERM/KMS), M. Vervier (LPP-ERM/KMS), B. Schweer (LPP-ERM/KMS), A. Dinklage (Max-Planck-Institut für Plasmaphysik), T. Fornal (Institute of Plasma Physics and Laser Microfusion), D. Gradic (Max-Planck-Institut für Plasmaphysik), M. Gruca (Institute of Plasma Physics and Laser Microfusion), K. P. Hollfeld (Zentral Institut für Engineering, Elektronik und Analytik-Engineering und Technologie), J. P. Kallmeyer (Max-Planck-Institut für Plasmaphysik), I. Ksiazek (Institute of Physics, Opole University), A. Krämer-Flecken (Institut für Energie-und Klimaforschung/Plasmaphysik), M. Kubkowska (Institute of Plasma Physics and Laser Microfusion), Ch. Linsmeier (Institut für Energie-und Klimaforschung/Plasmaphysik), F. Louche (LPP-ERM/KMS), O. Neubauer (Institut für Energie-und Klimaforschung/Plasmaphysik), D. Nicolai (Institut für Energie-und Klimaforschung/Plasmaphysik), G. Offermanns (Zentral Institut für Engineering, Elektronik und Analytik-Engineering und Technologie), G. Satheeswaran (Institut für Energie-und Klimaforschung/Plasmaphysik), L. Syrocki (Institute of Plasma Physics and Laser Microfusion), M. Van Schoor (LPP-ERM/KMS), R. C. Wolf (Max-Planck-Institut für Plasmaphysik), the TEC team (LPP-ERM/KMS, Institut für Energie-und Klimaforschung/Plasmaphysik), and W7-X team (See author list Sunn Pedersen et al. Nucl. Fusion 62 (2022) 04202).

Abstract

The superconducting stellarator Wendelstein 7-X (W7-X) at the Max-Planck-Institute in Greifswald began operation in 2015. To demonstrate efficient confinement of energetic alpha particles, which will be essential for a future Helias fusion reactor, W7-X requires a population of fast ions with energies ranging from 80 to 100 keV in the core of high-density plasmas. This can be achieved with Ion Cyclotron Resonance Heating (ICRH) using minority heating of H in ^4He and D plasmas, as well as the three-ion scenarios of ^4He - ^3He -H and D- ^3He -H.

The ICRH antenna for W7-X consists of two poloidal straps. Each strap is terminated by a pre-matching capacitor at one end and short-circuited at the other, with RF power fed at an intermediate position along the straps. The antenna's shape is tailored to match the 3D shape of the Last Closed Magnetic Surface (LCMS) in the standard magnetic field configuration of W7-X, resulting in variable curvature in both toroidal and poloidal directions over the plasma-facing surface [1]. The antenna can also be moved radially up to 35 cm, and a gas puffing system is incorporated to inject gas in the region between the scrape-off layer (SOL) and the LCMS to locally improve coupling. The full system was commissioned on W7-X plasmas in February and March of 2023. During these experiments, only one of the two straps was powered due to a faulty pre-matching capacitor and vacuum feedthrough, resulting in operation with $k_{\parallel} \sim 0$.

Two significant milestones were achieved: operation at high power levels (up to 700 kW) and the generation of a target plasma using ICRH alone at magnetic fields below the usual 2.5 T, where the 140 GHz ECRH system is not resonant. In these experiments, the standard magnetic configuration in W7-X was used, the LCMS was positioned 17 cm from the first wall, and the distance between the antenna and the LCMS ranged from 3 to 10 cm. Despite the unfavourable heating conditions, $k_{\parallel} \sim 0$, there was a clear increase in the plasma stored energy at constant electron density, consistent with an increase in ion temperature. The Faraday screen is omitted in this antenna design, based on extensive experience with TEXTOR [2]. No edge interaction was observed. Small levels of Cu and C impurities were observed with VUV and Soft X-ray diagnostics. In plasma start-up experiments at 1.7 T, plasmas were sustained for the full duration of the ICRH pulse with approximately 300 kW RF power.

References

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11:40-12:10 [Mo1.A.I1] Nonlinear Excitation of Energetic-Particle-Driven Geodesic Acoustic Mode by Resonance Overlap with Alfvén Eigenmode

Hao Wang (National Institute for Fusion Science), Philipp Lauber (Max-Planck-Institute for Plasma Physics), Yasushi Todo (National Institute for Fusion Science), Yasuhiro Suzuki (Hiroshima University), Hanzheng Li (National Institute for Fusion Science), Jialei Wang (National Institute for Fusion Science), Malik Idouakass (National Institute for Fusion Science) and Panith Adulsiriswad (National Institutes for Quantum Science and Technology).

Abstract

The Alfvén instability nonlinearly excited the energetic-particle-driven geodesic acoustic mode on the ASDEX-Upgrade tokamak, as demonstrated experimentally [1-5]. The mechanism of the energetic-particle-driven geodesic acoustic mode excitation and the mode nonlinear evolution is not yet fully understood. In the present work, a first-principles simulation using the MEGA code [6] investigated the mode properties in both the linear growth and nonlinear saturated phases [7,8]. The simulation parameters and the equilibrium data are based on the realistic experiment, that is shot #34924 of ASDEX-Upgrade at $t = 1.9$ s. Here we show that the simulation successfully reproduced the excitation and coexistence of these two modes, and agreed with the experimental results well. The mode frequencies of these two modes are about 100 kHz and 50 kHz, respectively. In addition, the simulation reveals a radially inward energetic particle redistribution during mode activities, consistent with the experimental findings. Conclusive evidence showed that the resonance overlap is the excitation mechanism of the energetic-particle-driven geodesic acoustic mode. In the linear growth phase, energetic particles that satisfied different resonance conditions excited the Alfvén instability, which then caused energetic particle redistribution in phase space. These redistributed energetic particles caused resonance overlap, exciting the energetic-particle-driven geodesic acoustic mode in the nonlinear phase. The above process was demonstrated in toroidal canonical momentum P_ϕ and energy E phase space, and the time evolution of energetic particle distribution f_{total} and δf are investigated in detail.

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12:10-12:30 [Mo1.A.O1] Numerical Investigation of Fishbone Instability in Tokamak Plasmas

Alodie Grondin-Exbrayat (Aix-Marseille Université, CNRS), Matteo Faganello (Aix-Marseille Université, CNRS), Nicolas Dubuit (Aix-Marseille Université, CNRS) and Alexandre Poye (Aix-Marseille Université, CNRS).

Abstract

Understanding the transport and losses of particles in tokamaks is crucial for operating future fusion reactors in particular for energetics particles, as they can be expelled before being able to deposit their energy in the tokamak core. Focusing on energetic trapped particles, they can destabilize the so called fishbone instability [1, 2, 3] that finally induces their transport. Indeed, the resonant interaction between the toroidal precessional motion of particles and the low-frequency kink mode [2, 4] leads to a growing fishbone instability in the core of the tokamak. Those energetic particles can be generated by heating systems in D-D experiments, for instance, or correspond to the population of particles with a velocity mainly perpendicular to the ambient magnetic field in D-T experiments. In this work, we first present a model coupling a reduced MagnetoHydroDynamics model in cylindrical geometry, for the thermal plasma, with a kinetic description for the trapped energetic particles. The numerical work takes advantage of reduced MagnetoHydroDynamic code, AMON [5], developed at the PIIM laboratory, to which we added a gyro- and bounce-averaged Vlasov equation for the energetics particles. We ran linear simulations, using a small resistivity and different amounts of energetic particles for investigating the development of the fishbone instability close and far to the threshold [6], checking for the correct growth rate of the mode and the eigenfunction profiles. Non-linear simulations have been performed focusing on the saturation of the fishbone instability and the induced transport, by checking the phase space dynamics of the energetics particles.

References

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11:40-12:10 [Mo1.B.I1] Modelling of Modern Plasma Processing Reactors: Plasma Physics and Surface Chemistry

Igor Kaganovich (Princeton Plasma Physics Laboratory), Yury Barsukov (Princeton Plasma Physics Laboratory), Kallol Bera (Applied Materials, Inc, Santa Clara, USA), Jian Chen (Sun Yat-sen University, Zhuhai, China), Stephane Ethier (Princeton Plasma Physics Laboratory), Sathya Ganta (Applied Materials, Inc, Santa Clara, USA), Sierra Jubin (Princeton Plasma Physics Laboratory), Alexander Khrabrov (Princeton Plasma Physics Laboratory), Andrew Tasman Powis (Princeton Plasma Physics Laboratory), Shahid Rauf (Applied Materials, Inc, Santa Clara, USA), Dmytro Sydorenko (University of Alberta), Haomin Sun (École Polytechnique Fédérale de Lausanne (EPFL)), Sarveshwar Sharma (Institute for Plasma Research, Ahmedabad, India), Liang Xu (Soochow University, Suzhou, China), Abishek Verma (Applied Materials, Inc, Santa Clara, USA), Willca Villafana (Princeton Plasma Physics Laboratory) and Michael Tendler (KTH Royal Institute of Technology, Stockholm).

Abstract

For modern plasma processing, there is a need to perform kinetic simulations of large plasma devices using the particle-in-cell (PIC) technique due to relative ease of implementing the method, and that it can be parallelized effectively over many processors and accelerated on GPUs. At PPPL we have developed two codes EDIPIC-2D and LTP-PIC-3D. EDIPIC-2D is an open-source code that includes features for simulations of practical devices and has been used for modelling of several plasma devices. LTP-PIC-3D is a high-performance scalable PIC code which incorporates best programming practices and multi-level parallelism. This code was upgraded to operate efficiently on the latest CPU/GPU architectures for additional performance improvements. Energy conserving or implicit methods were implemented to speed up simulations [1]. Effects of numerical noise in simulations using PIC code need to be analysed and considered [2]. These codes have been applied to study plasma processing applications, such as capacitively coupled plasmas [3], electron beam produced plasmas [4], inductively coupled plasmas, and hollow cathode discharges. To model surface processes we used a combination of quantum chemistry methods and molecular dynamics [5].

Acknowledgments Supported by the US Department of Fusion Energy Science.

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12.10-12:30 [Mo1.B.O1] Optimization of Electron Cyclotron Resonance (ECR) Plasma Parameters for Enhanced Material Processing Applications

Sunil Swami (Department of Physics, Savitribai Phule Pune University, India), Vikas Mathe (Department of Physics, Savitribai Phule Pune University, India), Sudha Bhoraskar (Department of Physics, Savitribai Phule Pune University, India), Premkumar S. (Armament Research and Development Establishment, India), Supriya More (Research Unit Plasma Technology, Ghent University) and Rino Morent (Research Unit Plasma Technology, Ghent University).

Abstract

Surface modification of polymers is essential for tuning their surface properties, opening a myriad of applications across various technological areas. ECR plasma is one of the technologies used for surface treatment of polymers. However, various ECR plasma system parameters affect the plasma behavior and the plasma energy required to tune the surface of polymers. Therefore, in this study, COMSOL Multiphysics-based Finite Element Analysis [1,2] was utilized to investigate various crucial plasma parameters as a function of microwave power, pressure, and magnetic field strength. Variations in the resonance zone and properties such as ionization rate, electron mobility, mean plasma electrical conductivity, electron temperature, electron density, etc., were predicted. Further, the parameters were experimentally measured [3] and validated. The results demonstrated good agreement with experimental data. This study provides valuable insights for optimizing plasma parameters for various applications of ECR plasma, such as polymer surface modification, surface grafting, and applications in space and biomedical fields.

Acknowledgments I would like to express my sincere gratitude to Mahatma Jyotiba Phule Research and Training Institute, Nagpur, India for their Financial support. Their assistance was crucial in enabling the progress and completion of this work.

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11:40-12:10 [Mo1.C.I1] Radiative Relativistic Magnetic Reconnection in Astrophysical Plasmas

Dmitri Uzdensky (University of Colorado Boulder and University of Oxford).

Abstract

Magnetic reconnection – a fundamental collective plasma process of violent reorganization of the magnetic-field topology and associated rapid release of magnetic energy– is ubiquitous in many space and astrophysical plasma environments, powering intense high-energy flares. However, under the extreme physical conditions encountered around neutron stars and black holes, reconnection takes place in an exotic radiative relativistic regime, where particles energized by magnetic-energy release to relativistic energies emit copious amounts of synchrotron and/or inverse-Compton radiation. The resulting radiative losses feedback on the reconnection process, altering its dynamics and energetics and suppressing nonthermal particle acceleration. Under even more extreme conditions, QED processes like pair creation come into play, leading to a nontrivial self-regulating regime where the reconnection layer creates its own plasma. In this talk I will review the recent advances in our understanding of this fascinating radiative relativistic reconnection regime made possible by novel first-principles radiative-QED particle-in-cell numerical simulations in combination with analytical theory.

12:10-12:30 [Mo1.C.O1] Subsonic Shock Waves in Reconnection Jets

Kuldeep Singh (Khalifa University of Science & Technology, Abu Dhabi), Frank Verheest (Sterrenkundig Observatorium, Ghent University) and Ioannis Kourakis (Khalifa University of Science & Technology, Abu Dhabi).

Abstract

The Magnetospheric Multiscale Spacecraft (MMS) identified electrostatic solitary wave (ESW) signatures associated with localized bipolar electric field pulses in their connection jet region located in Earth's magnetotail [1]. Those observations unveiled the presence of two counterstreaming ion (proton) beams and hot electrons [1], thus suggesting a possible link between the generation of nonlinear waves and counterpropagating cold ion beams within the jet. ESW observed in the magnetosphere [2] are known to be affected by energetic particles, a common occurrence in various Space environments and in particular in the solar wind, associated with long-tailed non-Maxwellian distributions [3].

Inspired by the above observations [1] and following up on subsequent studies [4-5], we have developed a three-component plasma model consisting of two counterstreaming proton beams and suprathermal electrons, in order to investigate the occurrence of electrostatic waves in the Earth's reconnection jet region. A thorough study reveals a number of possibilities, including predictions of subsonic pulse-shaped solitary waves -associated with the slow ion-acoustic mode- occurring only at a certain threshold in the beam speed (value)[6]. Subsonic shock-shaped structures have also been shown to exist in the presence of weak dissipation [7]. Subsonic electrostatic excitations are not possible in the absence of a beam.

Acknowledgments Authors KS and IK acknowledge financial support from Khalifa University's Space and Planetary Science Center under grant No. KU-SPSC-8474000336. KS and IK also thank KU for support via grant CIRA-2021-064/8474000412.

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14:00-14:30 [Mo2.A.I1] Momentum Balance and Energy Exchange in Gyrokinetic Turbulent Systems

Hideo Sugama (National Institute for Fusion Science), Tetsuji Kato (Graduate School of Frontier Science, The University of Tokyo) and Tomo-Hiko Watanabe (Department of Physics, Nagoya University).

Abstract

Gyrokinetics is a powerful theoretical framework based on which a large number of analytical and numerical studies on microinstabilities and turbulent processes in magnetized plasmas have been done. This work consists of two parts of our recent theoretical and simulation studies on gyrokinetic turbulence. In the first part, a variational principle is used to derive governing equations of electromagnetic turbulent gyrokinetic plasma systems with conservation properties favorable for long-time global gyrokinetic transport simulation of high beta plasmas. Then, the invariance of the Lagrangian of the system under an arbitrary spatial coordinate transformation is used to derive the local momentum balance equation in which effects of collision and source terms are included [1]. In a way analogous to the derivation of the energy-momentum tensor in the theory of general relativity, the variational derivative of the Lagrangian density with respect to the metric tensor is taken to directly obtain the symmetric pressure tensor which describes both collisional and turbulent momentum transport processes. The derived symmetric pressure tensor is useful for treating momentum conservation in symmetric magnetic configurations. In the second part, the effects of turbulence on energy exchange between electrons and ions are investigated. Although the effect of turbulent energy exchange has not been considered significant in previous studies, it is anticipated to have a greater impact than collisional energy exchange in low collisional plasmas such as those in future fusion reactors. It is shown from gyrokinetic simulations that the energy exchange due to the ion temperature gradient (ITG) turbulence mainly consists of the cooling of ions in the grad B-curvature drift motion and the heating of electrons streaming along a field line [2]. The ITG turbulence transfers energy from ions to electrons regardless of whether ions or electrons are hotter, which is in marked contrast to the energy transfer by Coulomb collisions. This implies that the ITG turbulence should be suppressed from the viewpoint of sustaining the high ion temperature required for fusion reactions since it prevents energy transfer from alpha-heated electrons to ions as well as enhancing ion heat transport toward the outside of the reactor. Furthermore, linear and nonlinear simulation analyses confirm the feasibility of quasilinear modeling for predicting the turbulent energy exchange in addition to the particle and heat fluxes.

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14:30-14:50 [Mo2.A.O1] Magnetized Plasma Experiment on Layering and Inhomogeneous Mixing in an Evolving Vortex Lattice: Melting Staircases

Richard Sydora (University of Alberta, Canada), Thomas Simala-Grant (University of Alberta, Canada), Fredy Ramirez (University of California, San Diego), Patrick Diamond (University of California, San Diego) and Shreekrishna Tripathi (University of California, Los Angeles).

Abstract

The phenomena of layering in turbulent mixing processes is observed in a variety of physical systems, including magnetically confined plasmas. In this case, layering is connected with the so-called **ExB** staircase, which is a series of mixing zones interspersed by shear layer mini-barriers. Staircase formation can result from inhomogeneous mixing. This basic experiment addresses some of the key questions in layering and staircase dynamics, such as deviation from marginality and the percolation of density and thermal mixing.

In this work, we investigate layering and staircase dynamics using a melting vortex lattice (or fluctuating cellular array), which intrinsically drives inhomogeneous mixing [1]. To accomplish this, a vortex array is created in a large linear magnetized plasma device (LAPD) that is designed to have a system of tangent eddies, thus forming a staircase structure which can exhibit “melting” through the interaction of neighboring cells in the lattice. To create the vortex lattice in the LAPD experiment, a smaller lanthanum hexaboride (LaB6) cathode source is placed at the opposite end of the device relative to the main discharge large area LaB6 cathode. A carbon mask containing a patterned array of holes is placed in front of this smaller cathode. When this smaller cathode is biased to a mesh anode in front of the main discharge cathode, a stream of electrons flows through the lattice of holes in the mask and current channels are created in the pattern of the mask. The lattice of filaments that form has finite axial extent and the filament length is controlled by adjusting the magnitude of the biasing voltage. The initial experiments were carried out using a 3x3 square lattice of cells. Langmuir probes were used to make cross-field planes of density, temperature, and plasma potential.

The results indicate the establishment of a dynamical vortex lattice with local and global vortical azimuthal and axial flows. In the near field region of the lattice, the local plasma potential forms a well, which induces **ExB** differential rotation of the lattice resulting in azimuthally symmetric inhomogeneous boundary layers that are on the scale of the gaps in the initial lattice structure. A series of mixing layers is interspersed with the boundary layers and cross-field diffusion begins to reduce the gradients. A spectral analysis of the gradient-driven fluctuations and their relation to the diffusivity, as well as the coupling of this spinning lattice structure to the background plasma will be presented.

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14:50-15-10 [Mo2.A.O2] Developments and Preliminary Applications of Tokamak Magnetohydrodynamic Codes

W Guo (ASIPP).

Abstract

MHD instabilities are of considerable fundamental and practical importance for magnetic fusion plasmas, since they are believed to set limits on the achievable plasma pressure and current density, sometimes even leading to the termination of the plasma discharge. An eigenvalue and an initial value MHD codes in toroidal geometry have been developed to facilitate the related numerical investigations in understanding MHD stability in tokamak plasmas, since the exact solutions are impossible to analytically obtain due to the complexity of toroidal geometry.

A new full MHD eigenvalue code SCEL (Symbolic Computation aided Eigenvalue and Linear code for Tokamak) has been constructed by utilizing the symbolic computation technique for the first time [1]. A symbolic vector analysis module is first developed to conduct the automatic derivation of the tedious linearized full MHD equations in the magnetic flux coordinate system. And an automatic numerical discretization module is developed to implement the automatic numerical discretization. The reliability of the new full MHD eigenvalue code developed with help of these two modules is verified by the internal kink mode and tearing mode tests. This new method has the advantage to dramatically reduce the manpower and to avoid possible errors in code development. The feasibility and advantage of the methodology also has been further demonstrated by solving the more difficult challenge of MHD continua, i.e. global Geodesic Acoustic Modes (GAM), which ordinarily come with spatial singularities [2].

Also, a new initial value full MHD code using discontinuous Galerkin (DG) and Weighted Essentially Non-Oscillatory (WENO) methods has been developed to solve the conservative perturbed MHD equations in toroidal geometry [3]. A triangular mesh based on the flux of the fixed boundary equilibrium in the poloidal plane and a uniform division in toroidal direction were adopted during the code development. The code was linearly tested by performing the linear calculations of the internal kink mode and tearing mode. Nonlinear simulations of the resistive internal kink mode and tearing mode are also carried out.

Acknowledgments This work is supported by the National Natural Science Foundation of China under Grant Nos. 12075282 and 11775268. The numerical calculations in this paper were performed on ShenMa High Performance Computing Cluster in Institute of Plasma Physics, Chinese Academy of Sciences and Hefei Advanced Computing Center.

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15:10-15:30 [Mo2.A.03] Plasma Boundary Simulations of Limiter Ramp-up Phase of ITER

Arzoo Malwal (Institute for Plasma Research), Devendra Sharma (Institute for Plasma Research) and Richard A. Pitts (ITER Organization).

Abstract

The vast majority of ITER plasma boundary simulations have focused on the divertor phase since it is under these conditions that the principal plasma performance objectives will be achieved. However, as in all tokamaks, every ITER plasma pulse will have to pass through a current ramp-up phase, which, in common with many devices will take place in ITER in limiter configuration on the central column[1]. As for the rest of the main chamber first wall (FW), the central column will consist of FW panels (FWP) protecting the blanket shield blocks, with each panel shaped toroidally to ensure protection of leading edges between adjacent panels. This shaping leads to localized power deposition due to magnetic field line shadowing. Analysis based on 3D field line tracing coupled with a very simple, non-dissipative description of the scrape-off layer (SOL) parallel heat flow profile, $q_{||}(r)$ has shown that under some conditions (depending on plasma current, heating power and the degree of radial misalignment between FWP's), heat loads during the limiter ramp-up can approach, or even exceed the stationary values for which the panels are rated.

Given the potential for FWP power flux overload, it is important to assess the degree of conservatism introduced by the simplified SOL model employed in field line tracing study. A first step in such an assessment is reported here in which a 3D plasma transport study is performed using the coupled plasma-neutral transport code EMC3-Eirene, taking into account the full 3D FW CAD geometry from which an appropriate EMC3-Eirene numerical grid is constructed and a large enough portion of the full torus is simulated to fully capture all shadowing effects. Radial profiles of transport coefficients are chosen such that $q_{||}(r)$ at the outside midplane (OMP) closely follows the double exponential profile used to determine the optimum FWP toroidal shaping [1,2]. First simulations are reported for a fixed, slightly elongated magnetic equilibrium at $I_p \sim 2$ MA, $B_T = 5.3$ T with varying SOL input power and OMP separatrix density, chosen on the basis of DINA code ramp-up simulations. These are similar parameter sets to those chosen for a companion 2D SOLPS-ITER study of ITER limiter start-up, though this latter work includes impurity evolution from the limiter surface, while there the focus is on pure hydrogen simulations. The key first conclusion from this work is that at the low densities (and hence high SOL T_e) required for limiter start-up on ITER, dissipation due to hydrogenic radiation alone is negligible and the EMC3-Eirene deposited power distributions are similar to those obtained from the simple field line tracing approach. To provide a more quantitative assessment of the real reduction in power loading to be expected compared with the simple engineering treatment will require impurity release and transport to be included.

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14:00-14:30 [Mo2.B.11] Study of an Atmospheric Gliding Arc Discharge for Sustainable Nitrogen Fixation into NO_x

Rony Snyders (University of Mons).

Abstract

Nowadays, plasma-assisted nitrogen fixation (NF) processes have been demonstrated as a highly promising alternative to the environmentally impacting Haber-Bosch process. Therefore, the community develops numerous attempts to optimize these processes in term of energy cost and yield. Among the various plasma technologies, atmospheric gliding arc plasmas (GAP) is found to suit well thanks to their warm feature and the significant molecular vibrational excitation they allow. In this contribution, we overview our recent experimental efforts to contribute towards a better understanding of this plasma technology for nitrogen fixation into NO_x.

In this context, the present work will specifically evaluate a gliding arc plasma operating with N₂ and O₂ at atmospheric pressure and more precisely on the study of the electrical (arc) stability of the process, and on the resultant benchmarking of the plasma-based nitrogen fixation process from a techno-economic perspective.

Considering the electrical stability of the studied process, the conventional approach of introducing external resistors to stabilize the arc is challenged by studying both the performance and the stability of the plasma replacing the external resistor with an inductor. From this work, we conclude that similar stabilization results can be obtained while significantly lowering the overall energy cost, which decreased from up to a maximum of 7.9 MJ/mol N to 3 MJ/mol N. Then, considering these results, we evaluate to which extent a hypothetical small-scale fertilizer production facility based on a rotating gliding arc plasma for nitrogen fixation can be a local competitive alternative for the Haber-Bosch process. This is done by proposing a comparative model to understand how capital expenditures, gas price, CO₂ allowances, levelized cost of energy, and transport costs affect the fertilizer production costs. The model highlights how, with the current best available technology, plasma-based nitrogen fixation, while being an interesting alternative for the synthesis of NO_x, still requires a more efficient use of H₂ for a direct production of NH₃.

Acknowledgments This research is supported by the FNRS project "NITROPLASM", EOS 0005118F.

14:30-14:50 [Mo2.B.O1] Impulse Measurements in a Pulsed Micro-Energy Propulsion System for Nanosatellites

Leopoldo Soto (Chilean Nuclear Energy Commission), Camilo Vásquez-Wilson (Universidad Adolfo Ibáñez), Cristian Pavez (Chilean Nuclear Energy Commission), José Moreno (Chilean Nuclear Energy Commission), Jalaj Jain (Chilean Nuclear Energy Commission), Biswajit Bora (Chilean Nuclear Energy Commission), Marcos Díaz (Universidad de Chile), Felipe Asenjo (Universidad Adolfo Ibáñez) and Luis Altamirano (Dicontek Ltda.).

Abstract

In this work, miniaturized Plasma Focus (PF) technology, based on scaling laws for PF devices, is utilized to design and construct a pulsed plasma thruster for CubeSat nanosatellites. After the pinch phase in PF devices, a plasma shock appears, traveling at speeds of approximately 10^5 m/s, independent of the energy of the device. The ejected mass scales with the energy of the device; for a device operating at 400 J, the ejected mass is approximately 10^{-10} kg [1]. Theoretical estimations suggest that a pulsed plasma thruster (PPT) based on PF technology, operating at 1 J, will generate impulses ranging from fractions of μ Ns to several μ Ns per pulse [2].

It is important to note that while PF devices operate at millibar pressures, a PPT operates in orbit under vacuum conditions, where plasma is generated through the ablation of insulating material, typically PTFE.

A pulsed plasma thruster was constructed using a small capacitor of 1.5 μ F, 2 kV (3 J stored energy), with dimensions of 50 mm x 45 mm x 30 mm and a weight of 115 g. This capacitor is charged using a voltage multiplier rated at 6 W, $V_{in} = 12$ V, $V_{out} = 2$ kV, with dimensions of 57 mm x 28 mm x 12 mm and a weight of 37 g. The plasma gun weighs approximately 2 g. Consequently, the total weight and volume of the PPT system are around 150 g and 100 cm³. To measure the impulse, a new thrust stand based on a single-point load cell was developed. The calibration of the load cell, along with the associated electrical signals and PPT assembly, is analyzed.

Acknowledgments This work is supported by ANID FONDECYT Regular 1211695.

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14:50-15:10 [Mo2.B.O2] Synthesis of Polystyrene Nanoparticles via a Gas Aggregation Cluster Source

Jasna-Tinea Jelinek (Ghent University), Maryam Nilkar (Ghent University), Zdeněk Krtouš (Charles University), Ondřej Kylián (Charles University), Rino Morent (Ghent University) and Nathalie De Geyter (Ghent University).

Abstract

Due to their unique properties, polymer nanoparticles (PNPs) have found their application in photonics, drug delivery, sensing, environmental remediation, and more. Numerous chemical methods have been developed for their synthesis, which normally require the use of solvents, are time-consuming, and costly. Therefore, there is still a need for environmentally sustainable, efficient, and time-saving methods for the synthesis of PNPs [1]. One such method is magnetron sputtering, a quite straightforward process with high deposition rates, yielding nanoparticles of very high purity, and allowing the tailoring of particle size distributions [2]. However, despite the high potential of this method in developing PNPs, most existing studies have primarily focused on the production of metal nanoparticles. To address this gap, we present, for the first time, the solvent-free synthesis of polystyrene (PS) nanoparticles via a radiofrequency (RF) magnetron-based gas aggregation cluster source (GAS).

A PS target with a 81 mm diameter and 4 mm thickness is bombarded by high-energy plasma species, mainly ions, leading to the ejection of atoms, molecules, or molecular fragments from the target. The ejected species then travel and condense onto surrounding surfaces [2]. The PNPs were synthesized under constant pressure of 164 Pa in the aggregation chamber and a constant flow of 40 sccm of argon with a reaction time of 30 minutes. The effect of power on the PNPs' characteristics was investigated across a range of power levels: 40, 60, and 80 W. The PNPs synthesized at lower power (40 W) exhibited spherical morphology with a diameter of approximately 100 nm, while higher powers (60 and 80 W) led to a cauliflower-like morphology marginally larger than the 40 W particles. Despite variations in morphology, Fourier-transform infrared (FT-IR) spectroscopy revealed a consistent preservation of the aromatic structure, evidenced by characteristic vibrational modes of the styrene ring with the out-of-plane bending vibration at 669 cm^{-1} , C-C in-plane stretching [3], and various vibration bands associated with symmetric C-H stretching vibration of the methyl group ($-\text{CH}_3$) at 2871 and 2958 cm^{-1} , indicating substantial branching of the polymer [4]. For further comprehensive analysis, X-ray photoelectron spectroscopy (XPS) will be employed to provide insight into the elemental composition of the PNPs. Due to particles appearing transparent in the images obtained from scanning electron microscopy (SEM), ultraviolet-visible (UV-VIS) spectroscopy will also be performed to elucidate optical properties and the potential application of the synthesized PNPs in optoelectronic devices.

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15:10-15:30 [Mo2.B.03] Population Dynamics of Charged Particles in EUV and EUV Induced Plasma

Manis Chaudhuri (ASML), Luuk Heijmans (ASML), Andrei Yakunin (ASML) and Mark Van de Kerkhof (ASML).

Abstract

Particle contamination control for particles down to submicron sizes is a crucial aspect of semiconductor processing and etching technologies. In recent times this aspect has also become crucial for the lithographic patterning process with the introduction of extreme ultraviolet (EUV) lithography which uses highly energetic EUV photons (13.5 nm, 92 eV). One of the side effects of this development is the generation of EUV-induced plasma due to the interactions of highly energetic EUV photons with low pressure (1–10 Pa) background hydrogen gas. The lithography machines operate in pulsed mode with EUV pulses of < 100 ns in every 20 μ s. The spatial and temporal evolution of the EUV induced plasmas has been investigated using 3D particle-in-cell (PIC) model. This shows the EUV light beam creating the plasma in < 100 ns (Fig.1a), whereafter it spreads and decays in the remaining 19.9 μ s afterglow (Fig. 1b). This also impacts the particle contaminants: they become positive within the EUV beam due to the photoelectric effect and they become negative outside and after the beam passage due to EUV-induced plasma. The location of charged particles during the EUV pulse and in the afterglow is shown below in Fig. 1(c-d) . The multi-pulse population dynamics of charged particles is shown in Fig. 1e. This shows a peaked increase in positive particle population during every EUV pulse and a steady increase of negative particle population during multiple afterglows.

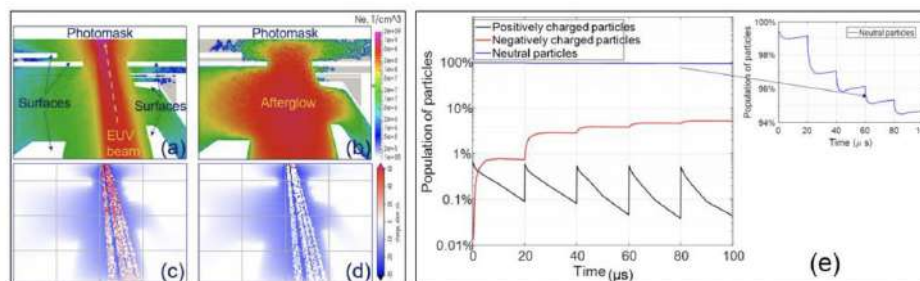


Figure 1 Spatial distribution profile of electron density at Reticle Mini-Environment within ASML lithography machine is shown (a) within EUV pulse (< 100 ns) and (b) after pulse (100 ns - 20 μ s) in the afterglow condition.(c-d) The typical population characteristics of 100 nm charged (positive: red and negative: blue) particles are shown in the two regimes mentioned in (a-b). For multi-pulse scenario, the population dynamics of charged and neutral particles are shown in (e). The population of neutral particle decreases over time at the expense of negatively charged particle generation. However, the population of positively charged particle remains steady over time.

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14:00-14:20 [Mo2.C.01] Quantifying the Expanding and Cooling Effects into the CGL Evolution Through the Expanding Box Model [online]

Sebastián Echeverría-Veas (Universidad de Chile), Pablo S. Moya (Universidad de Chile), Marian Lazar (KU Leuven & Ruhr University Bochum), Stefaan Poedts (KU Leuven & University of Maria Curie-Skłodowska) and Felipe Asenjo (Universidad Adolfo Ibáñez).

Abstract

One of the fundamental problems in space physics is the solar wind expansion and its interaction with different physical processes, e.g., collisions, wave turbulence or self-generated instabilities, conditioning the plasma dynamics. The expansion of the solar wind has been commonly described by the double adiabatic invariants or CGL theory [1], including the approach with the so-called Expanding Box Model (EBM) [2,3]. Despite the contributions made in the last decades, much remains to be understood and a realistically model, which includes plasma cooling and heating effects due to expansion concurring with other physical processes, is still an open problem. Our study introduces a new theoretical formalism to solve the CGL equations in an expanding framework, a significant step towards understanding what the expansion of the plasma entails but also what it implies. Our primary objective is to isolate the expanding effects and how they affect the conservation of double adiabatic invariants, the key aspect of the CGL theory.

To address the plasma expansion, we employed the Expanding Box Model transformations coupled with the ideal-MHD formalism used for CGL theory. This model provides a unique system of reference co-moving with the solar wind, allowing for the incorporation of transverse expansion into the double adiabatic equations. By following the same approximations and assumptions as in EBM and CGL theory, we developed a CGL-like description in which the expansion modifies the conservation of the double adiabatic invariants. Our results show that the double adiabatic equations are no longer conserved if plasma cooling is introduced through the EBM, with explicit dependence on expanding parameters, magnetic field profiles, and velocity gradients. Solving the equations for different magnetic field and density profiles (obtained self-consistently through the equations), we compute the evolution of anisotropy and plasma beta, which deviates from CGL predictions and empirical observations [4]. This deviation is attributed to the plasma cooling effect induced by the Expanding Box Model. Results suggest that heating mechanisms play an even major role in counteracting plasma cooling during expansion.

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14:20-14:40 [Mo2.C.O2] A 22-Year Cycle of the Network Topology for Solar Active Regions

Alejandro Zamorano (Universidad de Chile), Eduardo Flández (Universidad de Chile) and Victor Muñoz (Universidad de Chile).

Abstract

In this work solar cycles 21 to 24 were studied using complex network analysis. A network was constructed for each of the four solar cycles, where the nodes correspond to the active regions of the Sun that emanate flares, and on the other side, the connections correspond to the time sequence of solar flares. A similar network construction method is found in Ref. [1], following previous analyses for earthquake networks [2, 3]. In this way, we have constructed a directed network where we have also allowed self-connections. We calculate the incoming degree for each node, and subsequently the degree distribution, finding that for each solar cycle, the degree distribution obeys a power law, suggesting a preference of flares to appear in correlated active zones. Additionally, we found a variation in the characteristic exponent for each cycle, being higher in the even cycles than in the odd cycles. By means of a more detailed analysis based on moving 11-year networks, we find that the characteristic exponent varies with a period of approximately 22 years [4], which suggests that complex networks may provide information about the Hale cycle [5].

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16:00-16:30 [Mo3.A.11] Alpha-Particle Physics Studies in D-3He Plasmas at JET and JT-60SA in Support of ITER Rebaseline

Yevgen Kazakov (LPP-ERM/KMS), Jeronimo Garcia (CEA, IRFM), Massimo Nocente (Dipartimento di Fisica, Università di Milano-Bicocca), Rui Coelho (Instituto de Plasmas e Fusão Nuclear, Lisbon), Shunsuke Ide (National Institutes for Quantum Science and Technology (QST), Naka Fusion Institute), Vasily Kiptily (United Kingdom Atomic Energy Agency, CCFE), Jef Ongena (LPP-ERM/KMS), Hajime Urano (United Kingdom Atomic Energy Agency, CCFE), Maiko Yoshida (United Kingdom Atomic Energy Agency, CCFE), JET contributors (See author list of Maggi et al. to be published in Nuclear Fusion Special Issue from the 29th Fusion Energy Conference) and the EUROfusion Tokamak Exploitation Team (See author list of Joffrin et al. to be published in Nuclear Fusion Special Issue from the 29th Fusion Energy Conference).

Abstract

Mixed D-³He plasmas provide an opportunity to generate alpha particles with birth energies of 3.6 MeV without using tritium in a fusion device. The cross-section of the D-³He fusion reaction peaks at deuterium energies of ~450 keV. Recently, in preparation for alpha particle diagnostics in the DTE2 and DTE3 campaigns, dedicated alpha particle experiments were conducted at JET [1]. These experiments utilized a combination of ICRF and NBI heating systems to generate the necessary high-energy deuterons. The resulting alpha production rate of $\sim 1\text{--}2 \times 10^{16} \text{s}^{-1}$ enabled not only high-quality measurements of alpha particles [2], but also the observation of plasma instabilities driven by fusion-born alphas [3].

JT-60SA, the world-largest superconducting tokamak, will play a crucial role in supporting ITER and DEMO. It is equipped with a powerful NBI system, including both P-NBI and N-NBI, capable to deliver fast deuterons with energies up to 500 keV [4]. These characteristics make JT-60SA ideal for alpha-particle experiments in D-³He plasmas (there will be no tritium in JT-60SA to explore D-T fusion-born alphas), similar to those conducted at JET with a combination of P-NBI and ICRF systems. Following the closure of JET, JT-60SA is currently a unique tokamak for experimental studies of MeV-range fast ions and their impact on plasma confinement in large-scale tokamaks.

This proposed talk will first discuss recent JET experiments in D-³He plasmas, designed to prepare for future comparison experiments at JT-60SA. Specifically, in some of JET pulses discussed in this contribution, fast ions were deliberately generated off-axis, simulating the conditions expected with N-NBI ions at JT-60SA. We will proceed with discussing modeling results for a dedicated fast-ion scenario for alpha-particle studies in JT-60SA plasmas. The modeling shows that an alpha production rate of approximately $2\text{--}3 \times 10^{16} \text{s}^{-1}$ can be achieved with the high-power N-NBI system on JT-60SA at moderate ³He concentrations of 10-15%, surpassing the alpha production rate observed in D-³He plasmas on JET. The talk will conclude with a discussion on how future fast-ion experiments at JT-60SA can support the ITER rebaseline [5], particularly the development of alpha-particle diagnostics. This is a complex task, as most efforts have focused on exploiting alpha particle measurements using nuclear reactions between alpha particles and ⁹Be impurities. Due to the recent ITER decision to use a full-W wall, a new technique must be developed. We will present a proposed solution and explain how JT-60SA can contribute to this development.

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16:30-17:00 [Mo3.A.I2] Fuel Supply and Helium Ash Exhaust in Gyrokinetic Flux-Driven ITG/TEM Turbulence

Kenji Imadera (Kyoto University), Mikiya Muto (Kyoto University), Masatoshi Yagi (National Institutes for Quantum Science and Technology), Yasuaki Kishimoto (Kyoto University) and Akihiro Ishizawa (Kyoto University).

Abstract

Establishment of a refuelling method is an important issue for controlling nuclear fusion reactors. But, in DEMO-class high-temperature plasmas, a pellet injection reaches only up to 80-90% of the minor radius so that the central density peaking depends on particle pinch, making the prediction difficult. While turbulent particle transport has been studied based on local gyrokinetic models, it is also important to study global physics including the mean flow and the related neoclassical process. The global simulation is also meaningful to investigate impurity transport [1, 2] such as core Helium ash exhaust and edge impurity accumulation.

Based on this motivation, we perform flux-driven ITG/TEM simulations in the presence of ion/electron heating by means of the full- f electrostatic version of our global gyrokinetic code GKNET with a hybrid kinetic electron model [3]. This version enables us to precisely consider the self-consistent mean E_r determined by the radial force balance with the pressure and poloidal/toroidal flow profiles controlled by external source and sink. The global gyrokinetic ambipolarity condition can be also precisely treated so that we can investigate the physical mechanism of particle transport caused not only by non-axisymmetric $E \times B$ drift but also by axisymmetric $E \times B$ and magnetic drifts which is responsible for the interactions between turbulent and neoclassical transport.

It is found that ion heating can drive turbulent ion particle pinch by $E \times B$ drift ($n \neq 0$) because the negative thermo-diffusion term becomes dominant. Turbulent electron particle pinch is also driven in the case with electron heating. Such an electron particle pinch can trigger an ambipolar field, leading to up-down asymmetric density perturbations and resultant ion particle pinch by not only magnetic drift but also $E \times B$ drift ($n = 0$) [4]. It suggests that a density peaking of bulk ions due to turbulent fluctuations can be achieved by sufficiently strong both ion and electron heating. It also implies that a scale separation between neoclassical and turbulent transport process is not satisfied and their interactions become essential, when the macroscopic structure changes on a turbulence time scale shorter than a collision time scale. We also perform flux-driven ITG/TEM simulation for deuterium, helium, electron and find that both helium ash exhaust and fuel supply can be achieved simultaneously by the similar mechanism discussed above.

In this talk, we will report the heating condition as well as the interaction between helium and bulk ion and electron transport in details.

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17:00-17:20 [Mo3.A.O1] Alpha Particle Confinement with Magnetic Perturbations from Guiding Center and Full Orbit Simulations

Julio Martinell (Institute For Nuclear Sciences, National Autonomous University of Mexico) and Leopoldo Carbajal (Type One Energy).

Abstract

Fusion-produced alpha particles have to be well confined in order to maintain the plasma at thermonuclear temperatures. But the presence of magnetic perturbations which can be produced by instabilities of various kinds, modify the magnetic structure of the confinement device and hence the transport properties of fast particles. In previous works we have analyzed the transport of fast ions across the region where a magnetic island is present comparing the predictions from a guiding center description and the full orbit ion trajectory [1]. That study was limited to the ion energies in the range of NBI heating, from 10 to 50 keV and it was found that the full orbit outflows are larger than those predicted by guiding center transport [2]. In this work we study the transport of alpha particles with birth energies in the MeV range. The alphas are born in the plasma center and cross an island chain located at about the mid-radius. The analysis is done using a guiding center code (GCAF) and the full orbit code (KORC) for the case of a mid-size tokamak. Resonance effects are found to be important for the particles to cross the island region, regarding the island width and the particle Larmor radius. In addition, for rotating islands, the rotation frequency has a resonant effect on the particle flow when it is of the order of the transit frequency or the trapping frequency. The study with the guiding center code is also applied to the case of a stellarator with single helicity in order to estimate the non-axisymmetric effects on the transport.

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17:20-17:40 [Mo3.A.O2] Development and Basic Studies on the Cs-Free Negative Hydrogen Ion Source TPDsheet-U

Kohnosuke Sato (Tokai University), Kaito Miura (Tokai University), Naonori Okada (Tokai University) and Akira Tonegawa (Tokai University).

Abstract

The progress has been reported [1-3] toward realizing a high-performance caesium (Cs)-free negative-ion source based on volume production in the magnetized sheet plasma device TPDsheet-U. In the experiments, H⁻ions have been successfully extracted from sheet plasma by using single-aperture grids when argon gas is added to hydrogen plasma at the external magnetic field strength of 38 mT. The experimental results with the single-aperture grid show that the performance of negative hydrogen ion current density ($J_C(H^-)$) of Cs-free H⁻ ion beam in TPD sheet-U is about one-fourth of that of Cs-containing H⁻ ion sources in the negative-ion neutral-beam injector for ITER (NNBI) [4]. (i) The $J_C(H^-)$ was ~ 7.5 mA/cm² at an extraction voltage of 10 kV, a discharge current I_d of 90 A, and a gas pressure of 0.3 Pa without argon. (ii) Co-extracted electrons JEG(e) are successfully suppressed by setting a soft magnetic filter (SMF) on plasma grid. The JEG(e)/ $J_C(H^-)$ ratio has been reduced to below 1.0 for a single-aperture grid with the SMF.

The basic studies have been carried out extensively in order to understand the mechanisms of volume production of H⁻ions in the magnetized sheet plasma and its dependences on gas pressure, magnetic field strength and grid structures.

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16:00-16:30 [Mo3.B.11] Steam Thermal Plasma for Turquoise Hydrogen Production

Alan Maslani (Institute of Plasma Physics of the Czech Academy of Sciences), **Jafar Fathi** (Institute of Plasma Physics of the Czech Academy of Sciences), **Michal Hlina** (Institute of Plasma Physics of the Czech Academy of Sciences), **Vineet Sikarwar** (Institute of Plasma Physics of the Czech Academy of Sciences) and **Maksym Buryi** (Institute of Plasma Physics of the Czech Academy of Sciences).

Abstract

Thermal plasma offers great opportunity for processing of wide range of materials. For instance, different wastes can be efficiently decomposed into individual elements and as a result, valuable products can be obtained. One of such products is hydrogen, which is abundant in principle everywhere, its efficient large-scale production is inevitable for industry or as a fuel, but at the same time hydrogen production is still considered as a challenge. Indeed, hydrogen technologies are a very rapidly evolving field and there is a great demand for alternative methods of hydrogen production. At present, most hydrogen is produced with CO₂ as a by-product, namely by fossil fuels reforming. The fossil reserves of hydrocarbons/methane/natural gas are still huge, but their processing by currently used methods is apparently unsustainable. Research teams and petrochemical companies around the world are working intensively on this issue, and new technological solutions can be expected in the near future. In general, we can see in every day's life that the way in which energy is produced and used changes fundamentally.

This contribution presents the overview of the research related to the production of hydrogen from methane (or natural gas) and other hydrocarbons using thermal plasma. The hydrogen produced this way is categorized as a turquoise hydrogen and can be considered as a complementary to the well-known green and blue hydrogen production ways [1]. Produced hydrogen is characterized by high purity and at the same time we address the possibility of using the residual solid carbon, which can have a wide range of parameters. This is clearly the critical issue connected with turquoise hydrogen production, because once the produced solid carbon finds reasonable market, then the hydrogen produced this way becomes meaningful and competitive to other technologies. As an example from our laboratory research, we present the steam thermal plasma with very high enthalpy and low mass flow rate produced in the direct current arc discharge, which is in direct contact with water vortex surrounding the arc column [2, 3]. This thermal plasma source is attached to the reactor with internal volume 200 l, where interaction of the hydrocarbon flow with the plasma takes place. Results include characterization of the gaseous and solid product of the reaction. Both hydrogen and carbon are analysed with the intent of wide range of different applications and at the same time, almost zero CO₂ (or other greenhouse gases) production.

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16:30-16:50 [Mo3.B.O1] Plasma Application in Agricultural Waste Processing: a Thermodynamic and Experimental Study

Vladimir Messerle (The Institute of Combustion Problems), Alexandr Ustimenko (The Institute of Combustion Problems) and Oleg Lavrichshev (Al-Farabi Kazakh National University).

Abstract

A large amount of manure and its irrational use negatively affect the environment. By using plasma processing of manure, it is possible to enhance the process of obtaining synthesis gas ($\text{CO} + \text{H}_2$) and increase plant productivity by 150–200 times over biomass fermentation. Plasma processes are characterized by high temperatures, which greatly reduce waste processing time. This paper examines the plasma processing of biomass using the example of dried mixed animal manure (dung with a moisture content of 30%). Characteristic composition of dung, wt. %: H_2O – 30, C – 29.07, H – 4.06, O – 32.08, S – 0.26, N – 1.22, P_2O_5 – 0.61, K_2O – 1.47, CaO – 0.86, MgO – 0.37.

The thermodynamic code TERRA was used to analyze the plasma gasification and pyrolysis of dung. The calculations were conducted in the temperature range of 300 to 3,000 K and pressure 0.1 MPa for the following thermodynamic systems: 100% dung + 25% air (plasma gasification) and 100% dung + 25% nitrogen (plasma pyrolysis). At a temperature of 1,500 K, which provides complete gasification of dung carbon, the maximum yield of combustible components, and decomposition of toxic furan, dioxin, and benz(a)pyrene, the following composition of gas was obtained, vol. %: CO – 29.6, H_2 – 35.6, CO_2 – 5.7, N_2 – 10.6, H_2O – 17.9 (gasification); CO – 30.2, H_2 – 38.3, CO_2 – 4.1, N_2 – 13.3, H_2O – 13.6 (pyrolysis). Gasification and pyrolysis of dung require 1.28 and 1.33 kWh/kg of specific energy, respectively.

A reactor with a capacity of 50 kg/h with a 100 kW plasma torch was used to process dung experimentally. The dung was gasified in an air (or nitrogen during pyrolysis) plasma jet, which provided a mass-average temperature in the reactor volume of at least 1,600 K. Organic matter was gasified, and inorganic matter was melted. For pyrolysis and gasification of dung, the specific energy consumption was 1.5 kWh/kg and 1.4 kWh/kg, respectively. The maximum temperature in the reactor reached 1,887 K. A gas of the following composition was obtained, vol. %: CO – 25.9, H_2 – 32.9, CO_2 – 3.5, N_2 – 37.3 (pyrolysis in nitrogen plasma); CO – 32.6, H_2 – 24.1, CO_2 – 5.7, N_2 – 35.8 (air plasma gasification). The specific heat of combustion of the combustible gas formed during pyrolysis and plasma-air gasification of agricultural waste was 10,500 and 10,340 kJ/kg, respectively. Comparison of the integral indicators of dung plasma processing showed satisfactory agreement between the calculation and experiment.

16:50-17:10 [Mo3.B.O2] Precise Measurements of Short-Lived Reactive Species in Gas-Liquid Interfacial Plasmas with High-Speed Liquid-Column Flow

Toshiro Kaneko (Graduate School of Engineering, Tohoku University), Kazuki Takeda (Graduate School of Engineering, Tohoku University) and Shota Sasaki (Graduate School of Engineering, Tohoku University).

Abstract

Atmospheric pressure plasmas (APPs) in contact with liquid, which are defined as “gas-liquid interfacial plasmas (GLIPs)”, are widely used in chemical [1], medical [2], agricultural[3,4], and public health fields [5]. In these applications of GLIPs, the reactive species generated by the plasma in the gas and liquid phases, especially those with short lifetimes, are considered to play an important role.

The purpose of this study is to control the generation of short-lived reactive species using several lab-made GLIP devices, to measure them precisely, and to elucidate their chemical reaction mechanisms [6,7]. For this purpose, GLIP experiments using high-speed liquid-column flow [8] are conducted to investigate in detail the spatio-temporal dynamics of the short-lived reactive species in the liquid phase irradiated by a helium plasma. As a result, very fast decay (a half-life of ~ 0.1 msec) of OH radical was detected for the first time and explained with a numerical model assuming surface-localization of OH radical.

Furthermore, we are trying to measure not only OH radicals but also short-lived reactive nitrogen species (RNS). To achieve this purpose, we attempted to measure short-lived RNS, which are precursors of long-lived RNS such as nitrate and nitrite, using the GLIP system equipped with the high-speed liquid column flow. As a result, we have successfully measured the time decay of precursors of RNS using the reagent p-HPA (p-hydroxyphenylacetic acid), a scavenger of nitrite and nitrate precursors. The nitrite precursors were detected whereas nitrate precursors were below the detection limit, and the half-life of nitrite precursors was approximately 3 ms, which is obviously longer than 0.1 ms of OH. The results, such as the fact that only the precursor of nitrite decayed with time, led to the conclusion that the precursor of the reactive nitrogen species detected in the present study was N_2O_3 . These findings will contribute to the fully controlled generation of short-lived reactive species at the plasma-liquid interface, and the resulting selectively generated short-lived reactive species will be extended to a wide range of applications in environmental science, plant science, drug discovery science, material science, and other fields.

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16:00-16:20 [Mo3.C.O1] Universal Energy Cascade and Relaxation for Electron Magnetohydrodynamic Turbulence

Supratik Banerjee (Indian Institute of Technology Kanpur), Arijit Halder (Indian Institute of Technology Kanpur) and Amita Das (Indian Institute of Technology Delhi).

Abstract

The understanding of plasma turbulence is pivotal to describe several interesting features in both laboratory and space plasmas. For fluctuation length scales sufficiently larger than the ion inertial length scale, a plasma is described by magnetohydrodynamics (MHD), which is the simplest mono fluid model of plasma. However, space plasmas are often weakly collisional with sufficiently high thermal pressure and hence are associated with fluctuations with length scales sufficiently smaller than the ion inertial length scales. The corresponding fluctuations time scale is subsequently much smaller than the ion gyroperiod. In such a regime, the ions are practically immobile and the plasma can be described using electron magnetohydrodynamics (EMHD) where the electron fluid is primarily carrying both the inertia and the current while colliding with neutralizing medium of immobile ions.

Since the ion fluid velocity is negligible, the current ($\mathbf{J}$) and the electron fluid velocity ($\mathbf{v}_e$) are related as $\mathbf{J} = -ne\mathbf{v}_e$. In addition, the electron fluid is assumed to be incompressible such that $\nabla \cdot \mathbf{v}_e = 0$. Similar to ordinary magnetohydrodynamics, here too we neglect the displacement current ($\partial \mathbf{E} / \partial t$) assuming non-relativistic fluid velocities and the resulting governing equation for inviscid electron MHD becomes [1] $\frac{\partial}{\partial t} (d_e^2 \nabla^2 B - B) = \nabla \times [\mathbf{v}_e \times (d_e^2 \nabla^2 B - B)]$ which is simply a frozen-in field equation for $(d_e^2 \nabla^2 B - B)$ in the electron fluid with velocity $\mathbf{v}_e$ with $d_e = c / \omega_{pe} = (m_e / \mu_0 n e^2)^{1/2}$ being the electron inertial length. The total energy E , the sum of the kinetic energy of the electron fluid and the magnetic energy i.e. $E = \int \left(\frac{1}{2} \rho_e v_e^2 + \frac{B^2}{2\mu_0} \right) d\tau$, is an inviscid invariant of EMHD and hence is expected to show universal turbulent cascade.

In this work, we derive a compact exact relation where we express the average energy cascade rate ϵ in terms of two point increments of field variables and is written as $\langle \delta [\mathbf{v}_e \times (\rho_e \omega_e - ne\mathbf{B})] \cdot \delta \mathbf{v}_e \rangle = 2\epsilon$, (2) where ω is the electron fluid vorticity. By using the condition of vanishing cascade [2], it has been shown that the flow relaxes towards a pressure-balanced relaxed state given by $\mathbf{v}_e \times (\rho_e \omega_e - ne\mathbf{B}) = \nabla p_e$. The universal cascade and the relaxation are also numerically studied by the help of direct numerical simulations.

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16:20-16:40 [Mo3.C.O2] Non-Locality in Scale-to-Scale Energy Transfer in Hall Magnetohydrodynamic Turbulence

Arijit Halder (Indian Institute of Technology Kanpur), Supratik Banerjee (Indian Institute of Technology Kanpur), Manohar Kumar Sharma (LEGI, University of Grenoble Alpes) and Pablo D. Mininni (Departamento de Fisica, FCEN, UBA, Argentina).

Abstract

Hall magnetohydrodynamics (HMHD) is a single fluid plasma model often employed to study sub-ion scale plasma dynamics. Unlike ordinary MHD, HMHD system allows a finite difference between ion and electron fluid velocities. The Hall effect is modelled in the induction equation by adding a nonlinear term $-d_i \nabla \times (\mathbf{j} \times \mathbf{b})$ where d_i is the ion inertial length, $\mathbf{b}$ is the magnetic field (normalized to a velocity) and $\mathbf{j} = \nabla \times \mathbf{b}$. Therefore the Hall effect becomes important for length scales comparable or smaller than d_i .

In a turbulent plasma, the Hall effect plays a pivotal role by introducing a novel backscatter of magnetic energy which is found to be important for dynamo growth of magnetic field. By decomposing the Hall term in two parts namely $d_i(\mathbf{j} \cdot \nabla)\mathbf{b}$ and $-d_i(\mathbf{b} \cdot \nabla)\mathbf{j}$ one can separately measure the contributions of $\mathbf{b}$ and $\mathbf{j}$ -field to the dynamo action [1]. Along with the backscatter, the Hall effect is believed to introduce non-locality in the scale-to-scale energy transfer process [2]. In our current work [3], using data from simulation with 5123 grid points, we numerically investigate the locality of the energy transfer for two parts of the Hall term separately.

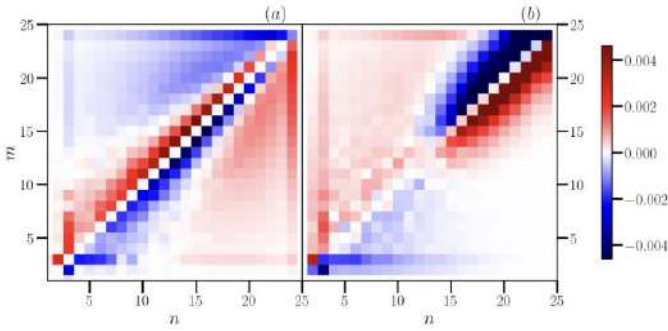


Figure 1 Shell-to-shell transfer rates for $\mathbf{b}$ -to- $\mathbf{b}$ (left panel) and $\mathbf{j}$ -to- $\mathbf{b}$ (right panel) transfers.

locally and nonlocally (Fig. 1b). However, for length scales smaller than d_i , the $\mathbf{j}$ -to- $\mathbf{b}$ transfer become strongly local and forward in nature. A phenomenological explanation is also provided based on the power spectra of $\mathbf{b}$ and $\mathbf{j}$.

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16:40-17:00 [Mo3.C.O3] Long-Living Equilibria in Kinetic Plasma Turbulence

Mario Imbrogno (Dipartimento di Fisica, Università della Calabria), Claudio Meringolo (Institut für Theoretische Physik, Goethe Universität), Sergio Servidio (Dipartimento di Fisica, Università della Calabria), Alejandro Cruz-Osorio (Instituto de Astronomía, Universidad Nacional Autónoma de México), Benoît Cerutti (Université Grenoble Alpes, CNRS) and Francesco Pegoraro (Dipartimento di Fisica “Enrico Fermi”, Università di Pisa, Pisa, 56122, IT).

Abstract

In classical fluids, turbulence is characterized by long-lasting patterns emerging from the chaotic environment. We use high-resolution, direct numerical simulations in two spatial dimensions to study the equivalent mechanism in fully kinetic plasma turbulence. We observe the creation of long-lived vortices whose profile is characteristic of macroscopic force-free states dominated by magnetic fields. We describe these metastable solutions with a self-consistent kinetic model in a cylindrical coordinate system centered on a representative vortex, inspired by the Harris pinch model for inhomogeneous equilibria, beginning with an explicit form of the particle velocity distribution function. These novel equilibria can be reduced to a modified force-free state of the Gold-Hoyle solution [1]. Long-lived structures facilitate turbulence, which is accompanied by transients when these vortices combine to generate self-similarly new metastable equilibria. Understanding this process can help shed light on several astrophysical events, such as the production of plasmoids around massive compact objects or the appearance of coherent structures in the heliosphere.

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17:00-17:20 [Mo3.C.O4] Novel Algorithms for the Accurate and Fast Solution of the Five-Moment Two-Fluid Plasma Equations

Xudong Ke Lin (University of Cambridge), Stephen Millmore (University of Cambridge) and Nikos Nikiforakis (University of Cambridge).

Abstract

The disparate time- and length-scales associated with the physics of magnetically confined fusion necessitate the deployment of a variety of simulation strategies, ranging from magnetohydrodynamic (MHD) to particle-based models. Single-fluid MHD, though computationally efficient, is invalid in regimes with small length and time scales, whilst particle-based methods are computationally expensive in simulations of full-domain phenomena, due to their high dimensionality. Five moment two-fluid plasma models offer an attractive compromise between the two approaches, by significantly reducing the dimensionality of the problem, but retaining important physics lost by standard MHD models. This work is concerned with the numerical solution of the two-fluid model by means of a finite volume approach and this work presents new innovations to improve the simulation efficiency. The cost of the calculation is reduced by relaxing the time step restrictions from the speed of light constraint with an implicit Maxwell solver and further improvement of the efficiency of the solution is achieved by using a locally implicit treatment of the stiff source terms. In this study, the multi-physics approach is extended in an attempt to capture the plasma-wall interaction. This is achieved through the implementation of a rigid body ghost fluid method, which allows the simultaneous consideration of both materials within the same computational domain. The algorithms are implemented in a highly parallelized software framework, and are validated against benchmarks from the open literature. It is shown that the new algorithms considerably reduce the computational times compared to traditional fully explicit methods.

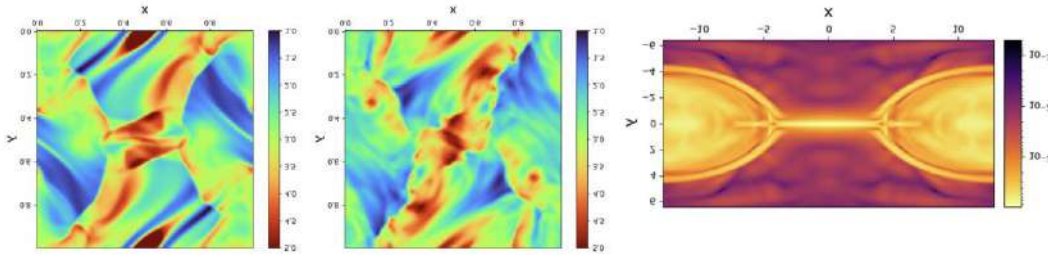


Figure 1 Validation test cases for both small and large length and time scales. From left to right - ideal MHD Orszag-Tang vortex, two-fluid Orszag-Tang vortex, GEM magnetic reconnection challenge.

1. Welcome – Mark Koepke, West Virginia University
2. PPP's federal framework and goals – Colleen Nehl, U.S. Department of Energy [remote]
3. PPP's realization and impact within the fusion community's private sector – Andrew Holland, CEO, Fusion Industry Association [remote]
4. PPP plan and promise – Ambrogio Fasoli, Programme Manager, EUROfusion [remote]
5. PPP role in roadmapping high-gain inertial fusion energy development – Peter Norreys, Professor of Inertial Fusion Science, University of Oxford [remote or in-person]
6. PPP for collaboration and innovation benefit – Chris Deeney, Director, Laboratory for Laser Energetics, University of Rochester
7. PPP vision for success in inertial fusion energy – Tammy Ma, Lead for the LLNL Inertial Fusion Energy Institutional Initiative
8. Aspirational highlights, targeted metrics of PPP – Open question
9. Question and answer opportunity

Tuesday
September 10

Complex Interplay of Magnetic Islands and Turbulence in Fusion Plasmas

Nicolas Dubuit (Aix-Marseille Université, CNRS), Daniele Villa (Aix-Marseille Université, CNRS), Bojana Stefanoska (Aix-Marseille Université, CNRS), Olivier Agullo (Aix-Marseille Université, CNRS), Magali Muraglia (Aix-Marseille Université, CNRS) and Xavier Garbet (CEA, IRFM).

Abstract

Magnetic islands are regions of reconnected magnetic field that appear frequently in magnetized fusion plasmas as well as in astrophysical plasmas. While they can be directly created by the current-driven "tearing" instability, in tokamak plasmas they frequently appear in situations where the tearing mode is stable. Therefore, the ubiquity of micro turbulence in tokamak plasmas has prompted many studies seeking to clarify the possible mechanisms through which turbulence may generate magnetic island. In the meantime, magnetic islands (regardless of their origin) perturb heat and particle transport, because they connect field lines across equilibrium magnetic surfaces. In addition to the associated degradation of confinement, this can lead to the explosive growth of so-called Neoclassical Tearing Modes. However, while the effect on collisional transport of single tearing-driven islands in simple MHD models is rather well understood, the situation with more detailed models, turbulence and/or multiple islands is another matter. Therefore, a consistent view of turbulence, transport and magnetic islands together is necessary.

Several different processes, discovered in the last decade, allow turbulence at small scales to drive large-scale magnetic islands. First, we show how the competition between those processes determines the size and shape of the generated large-scale magnetic islands, depending on the thermal power injected into the plasma [1]. Next, with use a 6-feld model [2, 3] to present a newly identified mechanism whereby the turbulence-generated "zonal" mean flows nonlinearly change the parity of the turbulent modes and, after a zonal flow-mediated coalescence process, drives large-scale islands. While this process depends on terms that are usually neglected in usual 2- or 3-field MHD models, it is dominant in the low-shear high- β regime of interest to advanced tokamak operation scenarios.

Next, we turn to the related issue of transport in magnetic islands. Even in the 2D mono-helicity geometry, the effect of magnetic islands can be much more complex than a flattening (or lack thereof) of the pressure gradient, in particular exhibiting self-heating [3] and/or several distinct flattened regions separated by sharp gradients. On top of this, the 3D multi helicity geometry enables the complex issue of stochastic regions of the magnetic field. Indeed, while such regions look deceptively uniform in Poincare plots, they feature complex local variations: in addition to sticky regions around islands, particular manifolds called Lagrangian Coherent Structures (LCS) which have been touted as possible transport barriers. However, the complex cohabitation of turbulence and turbulence-driven magnetic islands raises several questions which we address in this work: is stochasticity dominated by the small-scale magnetic flutter, or by the coupling of large-scale islands? Are the large-scale generated LCS resilient to the presence of small-scale turbulence?

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Pioneering High-Energy-Density Plasma Physics at the Laboratory for Laser Energetics

Christopher Deeney (Laboratory for Laser Energetics, University of Rochester).

Abstract

The pursuit of inertial confinement fusion created advanced pulsed-power and laser systems for five decades, culminating in the world's largest laser, the National Ignition Facility, demonstrating a target gain of more than 1 in 2022. In this journey, the laser systems, which deliver high-energy pulses in nanoseconds, have facilitated many other areas of high-energy-density science from studying materials at planetary conditions to simulating many aspects of astrophysics. The laser technology also advanced, including the development of chirped-pulsed amplification (CPA), to create picosecond pulses at petawatt levels of power. LLE is the home of two tens-of-kilojoule laser systems and one petawatt-class laser. The facilities support 2000 experiments per year for hundreds of users. In this presentation we will review the significant advances in fusion and high-energy-density science being conducted at LLE. This ranges from measuring fundamental plasma parameters like distribution functions to integrated experiments to study the role of magnetic fields in galaxy formation. In addition, the entire realm of short-pulse laser science, enabled by CPA, is expanding into advanced accelerator, THz radiation production, and intense x-ray sources. We will also discuss advances in short-pulse laser physics and a vision for 25-PW lasers to meet the scientific goals outlined in the recent international Multi-Petawatt Prioritization workshop.

Acknowledgments This material is based upon work supported by the Department of Energy [National Nuclear Security Administration] University of Rochester "National Inertial Confinement Fusion Program" under Award Number DE-NA0004144.

10:40-11:10 [Tu1.A.I1] Plasma Equilibria, Stability and Nonlinear Dynamics – a Celebration of the Contributions of Prof. Robert Dewar

Matthew Hole (Mathematical Sciences Institute, Australian National University), Arash Tavassoli (Mathematical Sciences Institute, Australian National University), Amitava Bhattacharjee (Princeton University), Stuart Hudson (Princeton Plasma Physics Laboratory), Adelle Wright (University of Wisconsin – Madison) and Zhisong Qu (Nanyang Technological University).

Abstract

Em/Prof. Robert (Bob) Leith Dewar, FAA, FAPS, FAIP (1944 – 2024) was a giant in the field of theoretical plasma physics, with important contributions in Magnetohydrodynamics (MHD) and in dynamical systems. These include MHD equilibrium and stability, MHD ballooning modes, Taylor relaxation and Hamiltonian maps. Bob worked closely with computer simulation and with experimentalists and has made important contributions to toroidal magnetic fusion research and to astrophysics. Over the last decade he had been instrumental in the development of a multiple region relaxed MHD model to describe general stellarator fields, and he was presently working on a generalization of such models to systems that preserve magnetic helicity with a weak ideal Ohm's law constraint. [1] Perhaps most importantly, he has left a legacy in both research and teaching, spanning 5 postdocs, 16 PhD, and many Masters and Honours students. Many of these now hold prominent positions in the field. Bob has also had a strong service contribution to the ICPP community, serving on the International Advisory Committee for decades, Chairing the 2002 ICPP meeting in Sydney, and was the architect of the ICPP statutes. In this proposed talk I will overview Bob's major contributions to the field, and focus on his work to describe magnetic fields in toroidally asymmetric configurations with flux surfaces, islands and chaos.

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11:10-11:30 [Tu1.A.O1] Tokamak Turbulence: a Pathway via a Series of Bifurcations.

Ben McMillan (University of Warwick), Chris Pringle (Coventry University) and Olly Smith (University of Warwick).

Abstract

Turbulence is often thought of as being constituted of a collection of interacting wave modes, but at the outset of classical fluid dynamics, was seen to arise as a series of increasingly complex structures that saturate and become successively unstable. We show that gyrokinetic tokamak turbulence follows this paradigm. The case of interest is subcritical turbulence that arises when there is both a driving gradient and a sheared background flow; this is known to give rise to isolated radially-propagating structures, and the formation and destabilisation of these structures is the pathway to turbulence in this system. Not only can we observe this in initial value simulations, it is possible to directly determine these structures via a nonlinear root-finding technique. These relative periodic orbits (RPOs) are turbulence states that repeat after some time, modulo some symmetry operation like translation; direct numerical solution allows us to determine these states even when they are unstable and to examine the bifurcation pathway as simple translation transitions via Hopf bifurcations into more complex nonlinear oscillations and finally space-filling turbulence.

11:30-11:50 [Tu1.A.O2] SPEculations (on Stepped Pressure Equilibria)

Robert MacKay (The University of Warwick).

Abstract

Extending work by Berk et al [1] on the case with a single interface, Bruno and Laurence [2] used KAM theory to make near axisymmetric magnetohydrostatic (MHS) fields with an arbitrary finite number of zones of constant pressure. The field is Beltrami in each zone and has a current sheet at each interface between zones, at which the pressure has a jump. Bob Dewar had the good idea, and the computationally expert team, to implement this strategy numerically [3]. The resulting stepped pressure equilibrium code (SPEC) is a hugely valuable tool in the search for suitable stellarator fields for confined nuclear fusion. In 2005, I began a discussion with Bob, in which I put the view that there could be limiting cases with infinitely many zones of constant pressure but for which the pressure is continuous, thereby eliminating the non-physicality of current sheets. The pressure gradient would be an L1 function supported on a fat Cantor set of flux surfaces. The same would hold for the current density. Such solutions would fit with Grad's hunch that most nonaxisymmetric MHS fields are pathological [4]. Bob was keen on the idea, but we never reached a conclusion. Here, I will present what I can on the proposal.

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11:50-12:10 [Tu1.A.O3] Periodic Nonlinear Phenomena (PNP) in Plasma Dynamics

Mark Koepke (West Virginia University).

Abstract

The basic elements (e.g., equivalent inductance and equivalent capacitance) of distributed-parameter and lumped-parameter characteristics will be described in a qualitative examination of multiple periodic nonlinear systems that are important as phenomenological models for demonstrating the salient features of the behavior of PNP that are encountered across a wide spectrum of discovery plasma science and beyond. This paper contains mostly theory but also contains many experimental examples from laboratory experiments.

- Basic, physical, and mathematical descriptions of PNP
- Prototype PNP systems
- Topological, analytical, variational, and perturbative methods
- Specific nonlinear characteristics of PNP
- Amplitude-frequency-phase relations and harmonic generation in PNP
- Experimental examples of PNP
- Coupled first-order systems
- Stabilization methods

Acknowledgments Seminal contributions from H. Lashinsky (deceased) and collaborative contributions from R. Majeski, D. Hartley, K.D. Weltmann, P. Miller, T. Klinger, and N. Brenning are greatly appreciated.

12:10-12:30 [Tu1.A.04] Tribute to the Late Professor Robert Dewar

Hideo Sugama (National Institute for Fusion Science).

Abstract

Professor Robert Dewar was a great theoretical plasma physicist who made many important contributions to the development of research in magnetic fusion and astrophysics. The first part of this talk highlights one of his great achievements: his theory on ballooning representation of instabilities in toroidal magnetic configurations [1]. He presented an elegant formulation of ballooning representation to efficiently investigate pressure-driven MHD instabilities in tokamaks and nonaxisymmetric systems such as stellarators and heliotrons. His work had a significant impact not only on analyses of MHD modes but also on studies of microinstabilities and turbulence through gyrofluid and gyrokinetic theory and simulation, in which his ballooning formalism is used as a standard helpful tool. The second half of the talk will touch on Professor Dewar's interactions with Japanese researchers. Professor Dewar has often visited Japan, sometimes for long periods of time, and has conducted many joint research projects. This talk will forward some messages from Japanese collaborators. It is also remembered that he participated in the AAPPs-DPP2023 in Nagoya last November and played very active roles as the program committee member, the plenary session chair, and the invited speaker. After the conference, he also visited NIFS and gave a seminar on plasma relaxation models, where he had a lively discussion and showed his energy. Therefore, we were even more shocked and saddened by the news of his sudden passing. We will never forget his great achievements and warm personality.

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14:40-15:10 [Tu2.A.11] High Power Ion Heating of Magnetic Reconnection in Two Merging Tokamak Plasmas

Yasushi Ono (University of Tokyo), Hiroshi Tanabe (University of Tokyo), Michiaki Inomoto (University of Tokyo) and Chio-Zong Cheng (Princeton Plasma Physics Laboratory).

Abstract

We have been developing high-power ion heating of magnetic reconnection using two merging tokamak plasma with high guide field $B_t \gg B_p$ (poloidal field) [1, 2]. The heated ion energy by magnetic reconnection scales with the reconnecting magnetic field component energy ($B_{rec}^2/2\mu_0$) where $B_{rec} \sim B_p$. The B_{rec}^2 -scaling of ion heating energy by reconnection can be understood by the fact that in the reconnection downstream the ion energy is mainly in the form of outflow kinetic energy before ions are thermalized in further downstream. The ion outflow velocity is produced mainly by the large $\mathbf{E} \times \mathbf{B}$ drift velocity associated with large poloidal electric field E_z , resulting from the formation of quadrupolar electrostatic potential structure in the downstream region and E_z depends linearly on $B_t B_p$ as observed in the experiments. Hence, the outflow velocity scales with B_{rec} , and thus the ion heating energy scales with B_{rec}^2 . High power ion heating with about 40-50% of $B_{rec}^2/2\mu_0$ converted into the heated ion energy can only be achieved when the reconnection current sheet thickness d is compressed to thinner than the ion gyroradius r_i . The B_{rec}^2 -scaling of high power reconnection ion heating provides an efficient way to produce burning plasmas with $T_i > 10\text{keV}$ by increasing B_{rec} to 0.6T (for $n_e \sim 1.5 \times 10^{19} \text{m}^{-3}$) without using any auxiliary heating methods like neutral beam injection (NBI). If $d \gg r_i$, the magnetic reconnection converts only 5-10% of $B_{rec}^2/2\mu_0$ into ion energy. This operation is suitable for magnetic helicity injection (current drive) of tokamak with small heating loss.

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15:10-15:40 [Tu2.A.I2] Ion and Electron Heating Characteristics During Magnetic Reconnection in ST40 and TS-6 Plasma Merging Experiments

Hiroshi Tanabe (University of Tokyo), Ryo Someya (University of Tokyo), Yunhan Cai (University of Tokyo), Tara Ahmadi (University of Tokyo), Mikhail Gryaznevich (Tokamak Energy Ltd.), Dmitry Osin (Tokamak Energy Ltd.), Hannah Willet (Tokamak Energy Ltd.), Hazel Lowe (Tokamak Energy Ltd.), Chio-Zong Cheng (Princeton Plasma Physics Laboratory), Michiaki Inomoto (University of Tokyo) and Yasushi Ono (University of Tokyo).

Abstract

Here we report ion and electron heating characteristics of magnetic reconnection under the influence of high guide field in ST40 and TS-6 plasma merging experiments using 96CH/320CH 2D ion Doppler tomography and Thomson scattering diagnostics. In addition to the extension of ion heating scaling $\Delta T_i \propto B_{\text{rec}}^2$ in keV range as demonstrated in MAST and ST40, our recent experiments explored the following 3 new findings using 2D ion Doppler tomography and Thomson scattering diagnostics: (1) formation of poloidally asymmetric global ion heating structure in TS-6 and highly localized electron heating around the X-point in ST40 via parallel electric field acceleration, (2) update of the heating scaling with $\Delta U_i \propto B_{\text{rec}}^2$ to 10kJ/m³ by including the contribution of electron density in collaboration with Thomson scattering measurement in ST40 from 2023 (the increment of ion thermal energy ΔU_i in the downstream region is ~30% of the upstream magnetic energy of reconnecting field B_{rec}), and (3) exploration of further electron heating via magnetic reconnection under the influence of high guide field in the keV range in ST40.

The poloidally asymmetric ion heating structure depends on the polarity of toroidal field B_t and the fine structure gets flipped when the guide field direction is reversed. Under the influence of high guide field, $\mathbf{E} \times \mathbf{B}$ drift is mainly driven by in-plane/poloidal electric field E_p from the quadruple potential structure, while parallel electric field $E_{\parallel}$ is mainly driven by reconnection electric field E_{rec} (spontaneously formed toroidal electric field E_t around X-point) and higher T_i appears where plasma potential is positive, while high T_e mainly appears around the X-point. The portion of toroidal electric field E_t for parallel electric field $E_{\parallel}$ is higher for high guide field condition ($B_t > 3B_{\text{rec}}$) and the peaked electron heating structure around the X-point becomes clearer when higher guide field is applied. Under the influence of toroidal effect to have higher guide field in the inboard side of outflow direction ($B_t \propto 1/r$), downstream heating also forms poloidally asymmetric structure, and more heating appears in the high field side. Perpendicular heat conduction in the outflow region is strongly suppressed by high guide field $\kappa_{\perp}^i / \kappa_{\perp}^e \sim 2(\omega_{ci} \tau_{ii})^2 \gg 1$ and the field-aligned transport process leads to the formation of poloidally ring-like characteristic fine structure after merging.

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14:40-15:10 [Tu2.B.11] The Laser-Hybrid Accelerator for Radiobiological Applications (LhARA)

Colin Whyte (University of Strathclyde) and The Lhara Collaboration The Lhara Collaboration (STFC).

Abstract

The “Laser-hybrid Accelerator for Radiobiological Applications”, LhARA, is being developed to serve the Ion Therapy Research Facility (ITRF). ITRF/LhARA will be a novel, uniquely flexible facility dedicated to the study of the biological impact of proton and ion beams. LhARA will combine laser ion acceleration, plasma lens with conventional accelerator technologies, to accelerate ions to 10's of MeV energies by using a high power laser focussed onto a thin target (target normal sheath acceleration). These ions, are captured and focussed by a non-neutral plasma lens known as a Gabor lens. The technologies can transform the clinical practice of proton and ion beam therapy (PBT) by creating a fully automated, highly flexible laser-driven system to:

- Deliver multi-ion PBT in completely new regimens at ultra-high dose rate in novel temporal-, spatial- and spectral fractionation schemes; and
- Make PBT widely available by integrating dose-deposition imaging with real-time treatment planning in an automatic, triggerable system.

The status of the ITRF/LhARA project will be described along with the collaboration's vision for the development of a transformative proton- and ion-beam system.

15:10-15:30 [Tu2.B.O1] Chaperoning Propagation of Laser Pulses and Laser Wakefield Accelerations in Plasmas

Devki Nandan Gupta (Department of Physics and Astrophysics, University of Delhi).

Abstract

The laser-plasma accelerator has advanced to the stage where control of beam parameters and stability are the main development challenges [1,2]. Achieving high quality electron beams in laser wakefield accelerators requires stable guiding of the driving laser pulse, which is challenging because of mode mismatching due to relativistic self-focusing. Here we show how an intense pre-pulse can be used to prepare the phase-space distribution of plasma electrons encountered by a trailing driver pulse so that it produces its own well-matched guiding channel that minimises wakefield evolution. We present a unique double-pulse scheme that creates a stable plasma channel waveguide intrinsically aligned with the trailing driver pulse. The plasma momentum distribution and electrostatic field are prepared by an intense, short duration pre-pulse that precedes the arrival of a second, co-propagating pulse. The time delay between the two pulses is chosen to be slightly less than the plasma period and the moderately intense leading, or chaperone, pre-pulse excites a weakly-nonlinear plasma wave that is below the threshold for wave breaking and self-injection. The pre-pulse does not directly produce a guiding structure for the trailing driver pulse, but rather its wake prepares the plasma by driving converging streams of electrons that are subsequently deflected by the ponderomotive force of the (trailing) driver pulse to produce a narrow, well-defined parabolic plasma channel. This acts as a self-aligned waveguide accompanying the driver over extended propagation lengths. The driver pulse initially undergoes relativistic self-focusing so that it matches the narrow density channel, which ensures high coupling efficiency. The chaperone pre-pulse stimulates guiding of the driver pulse, providing control over its evolution and enhancing the stability of the resulting wakefield [3].

Controlling high intensity driver pulses is an essential step in developing useful wakefield accelerators and compact radiation sources. Stable channels are necessary for developing next generation compact plasma undulators for compact synchrotron sources and free-electron lasers. Wide availability of ultra-compact accelerators and radiation sources could transform the way science is done.

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14:40-15:00 [Tu2.C.01] New Integrator for Relativistic Equations of Motion for Charged Particles

Takayuki Umeda (Information Initiative Center, Hokkaido University), Eiichiro Mizoguchi (Institute for Space-Earth Environmental Research, Nagoya University) and Riku Ozaki (Institute for Space-Earth Environmental Research, Nagoya University).

Abstract

Numerical methods for solving the relativistic motion of charged particles with a higher accuracy is an issue for scientific computing in various fields including plasma physics. The classic fourth-order Runge-Kutta method (RK4) has been used over many years for tracking charged particle motions, although RK4 does not satisfy any conservation law. However, the Boris method [1] has been used over a half century in particle-in-cell plasma simulations because of its property of the energy conservation during the gyro motion.

Recently, a new method for solving relativistic charged particle motions has been developed, which conserves the boosted Lorentz factor during the E-cross-B motion [2]. The new integrator has the second-order accuracy in time and is less accurate than RK4. Then, new integrator is extended to the fourth-order accuracy in time by combining RK4 [3]. However, it is not easy to implement the new fourth-order integrator into PIC codes, because the new method with RK4 adopted co-located time stepping for position and velocity vectors, which is not compatible with the charge conservation method.

In this paper, the two new relativistic integrators are reviewed. Then, a new leap-frog integrator with fourth-order accuracy in time is developed, which adopts staggered time stepping for position and velocity vectors.

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15:00-15:20 [Tu2.C.O2] Relaxation of the Courant Condition and Reduction of Numerical Errors in the Explicit Finite-Difference Time-Domain Method for Plasma Kinetic Simulations

Harune Sekido (Institute for Space-Earth Environmental Research, Nagoya University), Takayuki Umeda (Information Initiative Center, Hokkaido University) and Yoshizumi Miyoshi (Institute for Space-Earth Environmental Research, Nagoya University).

Abstract

This study provides a new numerical method for relaxation of the Courant condition and correction of numerical errors in the Finite-Difference Time-Domain (FDTD) method with the time-development equations using higher-degree difference terms. The FDTD method [1] is a numerical method for solving the time development of electromagnetic fields by approximating Maxwell's equations in both space and time with the finite difference of the second-order accuracy, which is widely used in plasma kinetic simulations. A staggered grid system is adopted in the spatial differences, in which Gauss's law is always satisfied. The FDTD method has a disadvantage that numerical oscillations occur due to the error between the numerical phase velocity and the theoretical phase velocity. The FDTD (2,4) method [2, 3], which uses the fourth-order spatial difference, is proposed for reduction of the numerical errors. However, the Courant condition becomes more restricted by using higher-order finite differences in space and a larger number of dimensions.

Recently, a numerical method has been developed by adding third-degree difference terms to the time-development equations of FDTD(2,4) [4]. Although the new method relaxes the Courant condition, there exist large numerical errors with large Courant numbers. In the present study, a new explicit and non-dissipative FDTD method is proposed with two types of the higher-degree difference operators for relaxation of the Courant condition and reduction of numerical errors. First, the one-dimensional third- and fifth-degree difference terms are added to the time-development equations of FDTD(2,6) [5]. Second, the third-degree difference terms including Laplacian are added to those of FDTD(2,4) [6]. The results of the test simulations show that numerical oscillations are not reduced so much with the one-dimensional difference operator, whereas the Laplacian operator suppresses an anisotropy in the waveforms and reduces the numerical oscillations. Furthermore, numerical instability is suppressed with large Courant numbers up to 1, which reduces the computational time of plasma kinetic simulations significantly.

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16:00-16:30 [Tu3.A.11] Electromagnetic Instabilities in Spherical Tokamaks

Daniel Kennedy (United Kingdom Atomic Energy Authority), Maurizio Giacomini (Dipartimento di Fisica “G. Galilei”, Università degli Studi di Padova), Plamen Ivanov (Rudolf Peierls Centre for Theoretical Physics, University of Oxford), Toby Adkins (Department of Physics, University of Otago), Facundo Sheffield (Max Planck Institute for Plasma Physics), Tobias Görler (Max Planck Institute for Plasma Physics), Arka Bokshi (York Plasma Institute, University of York), David Dickinson (York Plasma Institute), Harry Dudding (United Kingdom Atomic Energy Authority), Bhavin Patel (United Kingdom Atomic Energy Authority) and Colin Roach (United Kingdom Atomic Energy Authority).

Abstract

Electromagnetic micro instabilities are likely to limit performance in future advanced steady state tokamak plasmas and are expected to dominate transport in high β next generation spherical tokamaks (STs) such as STEP [1]. While gyrokinetic (GK) simulations have thus far proven to be a very accurate tool in modelling turbulent transport in predominantly electrostatic regimes, obtaining saturated nonlinear simulations in higher β plasmas with unstable kinetic ballooning modes (KBMs) and microtearing modes (MTMs) has proven computationally challenging (see e.g. [2]). Recent simulations of STEP-relevant equilibria that retain only MTMs and exclude KBMs (by neglecting compressional perturbations) saturate cleanly at very modest electron heat flux. However, local GK simulations find that including $\delta B_{\parallel}$ in such plasmas can unleash a hybrid KBM-like (hKBM) instability which drives very large heat fluxes (orders of magnitude greater than the available heating power) in the absence of strong equilibrium shear flows [3, 4]. These simulations underscore that understanding and mitigating hKBM-induced turbulence will be essential for the development of consistent flat-top operating regimes for future high performance ST devices.

In this talk we will present recent advances in our understanding of electromagnetic turbulence and discuss hKBM-driven turbulence in high β STs, with a particular focus on avoiding the high-transport state. We will present: (i) linear and nonlinear local simulations of hKBM turbulence, exploring the sensitivity of the turbulence and its associated transport to local parameters as well as different saturation mechanisms; (ii) a quasi-linear inspired reduced transport model for the hKBM turbulence; (iii) first flux-driven simulations for a high β ST that support the existence of a transport steady state in STEP with a fusion power comparable to that in the burning flat top of the conceptual design; and (iv) the first global nonlinear electromagnetic simulations of STEP to include $\delta B_{\parallel}$. These first-of-their-kind global simulations support the conclusions drawn from local GK simulations by: confirming that high-transport states in some equilibrium conditions are a robust prediction of GK and not simply an artefact of the local approximation; and tentatively supporting, in simulations that are currently running, the existence of a transport steady state in the vicinity of the conceptual STEP flat top. We will also discuss the most pressing priorities to be addressed in future work.

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16:30-17:00 [Tu3.A.12] Experimental Progress and Future Plan on Spherical Tokamak, QUEST

Kazuaki Hanada (Kyushu University), Hiroshi Idei (Kyushu University), Takeshi Ido (Kyushu University), Ryuya Ikezoe (Kyushu University), Yoshihiko Nagashima (Kyushu University), Makoto Hasegawa (Kyushu University), Takumi Onchi (Kyushu University), Toshiki Kinoshita (Kyushu University), Kengo Kuroda (Japan Coast Guard Academy), Makoto Oya (Kyushu University), Kazuo Nakamura (Kyushu University), Naoaki Yoshida (Kyushu University), Takahiro Nagata (Kyushu University), Aki Higashijima (Kyushu University), Shun Shimabukuro (Kyushu University), Ichiro Niiya (Kyushu University), Izumi Sekiya (Kyushu University), Kaori Kono (Kyushu University), Shoji Kawasaki (Kyushu University), Akira Ejiri (University of Tokyo), Sadayoshi Murakami (Kyoto University), Rojor Raman (University of Washington) and Masayuki Ono (Princeton Plasma Physics Laboratory, Princeton University).

Abstract

Spherical tokamak (ST) is a type of tokamak with low aspect ratio (A) that is the ratio of major radius (R) to minor radius (a) of torus and is intensively studied as a candidate for a cost-effective fusion power plant. Taking advantage of better MHD stability, higher bootstrap current fraction, and favorable confinement trend with magnetic field than traditional tokamaks (TTs), several designs of ST based fusion power plant have been proposed. But, the intrinsic natures of STs give rise to issues that need to be resolved. In particular, since the magnetic flux available for inductive plasma current generation is less than in TTs, it is important to develop methods to start and maintain the plasma current and to increase the toroidal magnetic field, B_T . Recently, a small-sized ST, ST-40 developed in a private company (Tokamak Energy Ltd.) in UK achieved 10keV of ion temperature for several tens ms at $B_T=2.2T$. The machine size of ST-40 ($R=0.4m$, $a=0.25m$) is significantly smaller than TTs that obtained more than 10 keV that is need to cause a DT nuclear fusion reaction. This proves a high potential of STs.

QUEST is a medium sized spherical tokamak with $R=0.64m$, $a=0.4m$, and $B_T<0.25T$ at $R=0.64m$. The QUEST aims at effective plasma current start up and stable maintenance of plasma discharge. To resolve the ST specific issues mentioned above, electron cyclotron current drive (ECCD) and coaxial helicity injection (CHI) are introduced on QUEST. An efficient ECCD could be achieved owing to energetic electrons (EEs), but the excess fraction of EEs may limit the application of toroidal electric field to drive plasma current to avoid a significant damage on the vacuum vessel due to local heat load caused by EEs. The methods we developed are to control the number of EEs with the assistance of control of injection refractive index to the magnetic field, $N_{||}$ of electron cyclotron wave (ECW) and/or to apply negative toroidal electric field using center solenoid. The combination of both methods could drive the bulk electron temperature up to 1keV. Another non-inductive start-up tool, the Transient coaxial helicity injection (T-CHI) has been successfully used in NSTX and a new electrode configuration is applied on QUEST. The electrode has been installed on lower divertor plates and electrically insulated by ceramic plates, and a plasma discharge between the electrode and the vacuum vessel is generated to supply magnetic helicity. The discharge develops in a force-free manner and magnetic reconnection drives a production of closed flux surface. The plasma induced by CHI is high density up to 1020 m^{-3} , but the temperature is still 10 eV. For long pulse operations on QUEST, it is impeded frequently due to particle imbalance. To resolve the issue, QUEST equipped a temperature controllable plasma facing wall (PFW) called hot wall. With help of the hot wall, 6 h discharges were obtained. In particular, the plasma duration of 40 min at 673K could be extended to more than 3 h through the temperature control of the hot wall.

Augmentation of B_T up to 0.5T and a CW gyrotron of 28GHz will be prepared as future plans of QUEST. Long pulse operations with higher plasma parameters will be expected.

16:00-16:30 [Tu3.B.I1] Optimised Direct Laser Acceleration of Electrons - Towards High-Brilliance Gamma-Ray Sources

Robert Babjak (GoLP - Institute for Plasmas and Nuclear Fusion).

Abstract

The pursuit of high-charge, high-density electron bunches driven by multi-petawatt lasers is essential for applications such as neutron generation, bright synchrotron radiation, and laboratory studies of electron-positron plasmas. Experiments have proven that at the hundred-terawatt scale, the OMEGA-EP facility can achieve electron energies of several hundred MeV and total charge over one hundred nanocoulombs via direct laser acceleration [1]. We push the acceleration mechanism towards multi-PW laser facilities to obtain electrons with energies surpassing GeV level and keeping the important property of high total charge. Our analytical scaling shows the dependence of an electron energy on laser intensity, and plasma density, and we provide a pathway to optimize this acceleration process for both existing and future facilities [2]. By precisely guiding a laser pulse over an appropriate distance, we can produce a wide Maxwellian spectrum of electrons with energies reaching several GeV and laser-to-electron conversion efficiencies in the tens of percent.

This presentation the nonlinear trade-off between the laser intensity and laser spot size will be explained. Understanding this balance provides insight into maximizing the transfer of laser energy to electrons, thus achieving the highest possible electron energies for a given laser power. We will compare our analytical scaling with Quasi-3D particle-in-cell simulations using the OSIRIS framework. Furthermore, we will discuss the theoretical aspects of plasma interactions under varying density conditions [3], crucial for understanding interactions in the preplasma of thin foils for ion acceleration. This analysis is necessary for experiments where the density profile of a gas jet varies along the laser propagation. In the end, we will highlight the potential of these electron bunches to emit high-brightness radiation at energies >100 MeV.

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16:30-17:00. [Tu3.B.I2] Scheme for Injecting Breit-Wheeler Positrons into a Plasma Channel Using Multi-PW Lasers

Dominika Maslarova (Department of Physics, Chalmers University of Technology), Bertrand Martinez (GoLP/Instituto de Plasmas e Fusão Nuclear, Instituto Superior Técnico, University of Lisbon) and Marija Vranic (GoLP/Instituto de Plasmas e Fusão Nuclear, Instituto Superior Técnico, University of Lisbon).

Abstract

Positron acceleration in plasmas has gained great interest due to its promising future applications, such as multi-TeV electron-positron colliders [1]. The challenge of this endeavor is greater for positrons than for electrons because self-generated fields from laser-plasma interactions are usually not well-suited for positron focusing and on-axis guiding. Additionally, an external positron source is required, while electrons are naturally available in the plasma. Here, we study electron-positron pair generation by an orthogonal collision of a multi-PW laser pulse and a GeV electron beam via the nonlinear Breit-Wheeler process [2].

We examine conditions favorable for positron deflection in the direction of the laser pulse propagation, facilitating injection into the plasma for further acceleration [3]. Using the OSIRIS particle-in-cell framework, we demonstrate that the radiation reaction triggered by ultra-high laser intensity plays a crucial role in positron injection by suppressing the initial transverse momentum gained from the Breit-Wheeler process. For the parameters used in our work, an intensity threshold is required to inject more than 1% of the created positrons, with the injection percentage rapidly increasing above this threshold.

Additionally, subsequent direct laser acceleration of positrons in the plasma channel using the same laser pulse can boost the final positron energy by a factor of two. The positron focusing and on-axis guiding are provided by significant electron beam loading, altering the internal structure of the channel fields. This study highlights the robustness and potential of this scheme for future applications.

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17:00-17:20. [Tu3.B.O1] Laser Wakefield Acceleration of Electrons in Double-Gas System

Ravina (University of Delhi) and D.N. Gupta (University of Delhi).

Abstract

The number of particles in an electron beam from laser wakefield acceleration is determined at the moment of trapping of background electrons. The longitudinal and transverse wave-breaking initiates the electron trapping. After some time, the trapping stops because of the repulsive force by the trapped particles. From many simulations and experiments, it has been well known that trapping of the background electrons begins much below the longitudinal wave-breaking limit [1,2]. This is related with transverse motion of the electrons. As an ultra-intense laser pulse propagates through a plasma, it pushes out the background plasma electrons and leaves behind a periodically-repeated bubble-like region. Inside the bubble, the electron density is very low, while the electron density at the rim of the bubble is very high. Highly energetic electrons make their trajectories along the rim of the bubble. Though many of such electrons turn around the rim and leave the bubble, some of those electrons are trapped in the transverse direction when their kinetic energies are lower than the depth of the potential well of the bubble [3].

The idea suggested in this paper is to use the double-gas system for electron injection. Based on the ionization of higher atomic number gas, enhanced injection has been estimated through particle-in-cell (PIC) simulations. The laser ionizes a lower atomic number gas to facilitate the laser wakefield via ponderomotive push of the electrons. However, the electron injection is weak in this case. The inclusion of higher atomic number gas (such as nitrogen) can enhance the electron injection via gas ionization. The advantage of this technique is to obtain the control of the beam charge in the laser-plasma accelerators, while keeping other parameters unmodified. Though the required gas ionization should be controlled via various parameters. A systematic bunch optimization is estimated in this work for future course of compact accelerator development.

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16:00-16:20 [Tu3.C.O1] Parametric Decays of Electromagnetic Waves in Electron-Positron Nonextensive Plasmas

Victor Muñoz (Departamento de Física, Facultad de Ciencias, Universidad de Chile), Gabriel Medel (Departamento de Física, Facultad de Ciencias, Universidad de Chile) and Roberto Navarro (Departamento de Física, Facultad de Ciencias Físicas y Matemáticas, Universidad de Chile).

Abstract

Wave propagation in relativistic plasmas is a subject of interest in many astrophysical and space systems, where electromagnetic fields may accelerate particles up to relativistic velocities, which in turn modifies the physics of wave propagation. Besides, kinetic effects further modify the dispersion properties of waves and their nonlinear interactions with the plasma particles. Thus, it is of interest to study wave propagation, and its nonlinear decays, considering both relativistic and kinetic effects.

However, the traditional approach of equilibrium statistics, where kinetic effects are described by Maxwellian velocity distributions, is not satisfactory in several environments where long range correlations, or memory effects, may lead the distributions to deviate from the Maxwellian case. Here, the proposal to describe plasma distribution functions in terms of nonextensive distribution functions, either of the Tsallis (where deviation from the Maxwellian case is given by a parameter q) [3] or the kappa type (where deviations are parametrized by a κ factor), [4, 5] allows to extend the traditional formalisms, to study wave linear and nonlinear propagation for systems out of thermodynamical equilibrium.

Following these ideas, in this work, parametric decays of an electromagnetic wave in an electron-positron plasma are studied. Kinetic effects are considered by means of the collisionless Vlasov equation, which is coupled to Maxwell equations. Relativistic effects on the particle motion are also taken into account. [6]

In the weakly relativistic case, although some of the instabilities involve strongly damped, electroacoustic pseudomodes, instabilities found using fluid theory are present as well in the kinetic regime. [1, 2] We study in detail the dependence on κ of the dispersion relation, the growth rate, and the phase velocity of the waves. As expected, results reduce to the Boltzmann-Gibbs statistics for $\kappa \rightarrow \infty$ ($q \rightarrow 1$).

Acknowledgements. This project has been financially funded by FONDECYT, grant number 1242013 (VM).

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16:20-16:40 [Tu3.C.O2] Using Visibility Graphs to Characterize Non-Maxwellian Turbulent Plasmas [online]

Sebastian Saldivia (Universidad de Chile), Pablo Moya (Universidad de Chile) and Denisse Pastén (Universidad de Chile).

Abstract

The Visibility Graph, a technique for mapping time series into complex networks, is employed to research underlying physical mechanisms in collisionless, turbulent plasmas. We analyze four distinct time series of magnetic field fluctuations obtained from Particle in Cell (PIC) simulations, initialized varying the κ parameter of its particle velocity distributions to explore departures from thermodynamic equilibrium. All studied cases exhibit a power law behavior in the degree distribution of the nodes. The critical exponent of this distribution unveils information about network properties, including particle correlations and heterogeneity. We compute the γ exponent for the degree distribution of the scale-free network and observe its evolution according to κ , peaking at $\kappa = 3$. This trend suggests that long-range correlations are more prominent in plasmas far from thermal equilibrium, while short-range correlations dominate in thermal plasmas following a Maxwellian distribution. These findings align with previous non-collisional plasma studies. Additionally, we investigate the μ and ν exponents associated with the slopes of power spectra of the magnetic fluctuations, obtaining insights into the energy dissipation and temporal persistence of the time series. Our findings reveal that low-frequency fluctuations exhibit the sharpest energy dissipation in thermal equilibrium environments, while high-frequency fluctuations dominate in systems described by velocity distributions with small κ . When comparing the correlation between these exponents and γ as a function of κ , we find a direct correlation for the exponent ν associated with high-frequencies, and an anticorrelation for the low-frequencies exponent μ . This finding underscores the connection between long- and short-range correlations and the Debye sphere of the plasma, revealing that the γ metric of the Visibility Graph is only able to see the smaller scales of a time series.

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Wednesday
September 11

Developing Numerical Tools for Tungsten Profile Measurement from X-Ray Diagnostics in WEST Plasmas

Didier Mazon (CEA), Y. Peysson (CEA), A. Jardin (Institute of Nuclear Physics, Polish Academy of Science), Gerenton (...). G. Verdoolaege (Ghent University), H. Wu (Ghent University, Southwestern Institute of Physics, CNNC, Chengdu), M. Chernyshova (Institute of Plasma Physics and Laser Microfusion), A. Wojenski (Warsaw University of Technology), J. Colnel (CEA), D. Guibert (CEA), T. Czarski (Institute of Plasma Physics and Laser Microfusion), K. Malinowski (Institute of Plasma Physics and Laser Microfusion), P. Linczuk (Warsaw University of Technology), D. Colette (ITER Organization), G. Kasprowicz (Warsaw University of Technology), K. T. Poźniak (Warsaw University of Technology), M. Walsh (ITER Organization) and the WEST team (<http://west.cea.fr/WESTteam>)

Abstract

In modern tokamaks like ITER—International Thermonuclear Experimental Reactor, or WEST – Tungsten (W) Environment in Steady-State Tokamak in Cadarache, the choice of W instead of traditional carbon as the main plasma facing material, to minimize tritium retention in the walls, has raised the essential issue of heavy impurity radiation. Monitoring and control of W concentration below 0.01% in the plasma core will be indeed necessary to avoid significant cooling of the plasma by impurity radiation, in particular in the soft X-ray(SXR) energy range of 0.1–20 keV.

In fusion devices, SXR plasma emissivity contains rich information not only about impurity transport, but also about the magnetohydrodynamic activity, magnetic equilibrium, plasma density and temperature. Since the radial impurity transport can be impacted by their poloidal distribution, SXR tomographic tools are valuable to infer the 2D impurity distribution and select adequate mitigation strategies.

In this context, this paper describes different efficient numerical tools, including artificial intelligence, neural network and Bayesian techniques for tungsten and/or electron temperature profiles evaluation in WEST plasmas based on experimental data from X-Ray measurements [1] and modelling tools [2], including synthetic diagnostics.

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Highlights from the path toward confined e^+e^- pair plasmas

E. V. Stenson (Max Planck Institute for Plasma Physics) representing the APEX Collaboration including V. C. Bayer (Max Planck Institute for Plasma Physics, Technische Universität München), E. Buglione-Ceresa (Technische Universität München), A. Card (Technische Universität München), J. R. Danielson (University of California San Diego), A. Deller (Max Planck Institute for Plasma Physics, University of California San Diego), P. Gil (Max Planck Institute for Plasma Physics), C. P. Hugenschmidt (Technische Universität München), P. Huslage (Max Planck Institute for Plasma Physics), J. von der Linden (Max Planck Institute for Plasma Physics), D. Mendoça (Technische Universität München), S. Nißl (Max Planck Institute for Plasma Physics), D. Orona (Massachusetts Institute of Technology), H. Saitoh (University of Tokyo), D. Schmeling (Columbia University), E. von Schoenberg (Concordia University), L. Schweikhard (University of Greifswald), M. Singer (Max Planck Institute for Plasma Physics, University of Greifswald), J. Smoniewski (Max Planck Institute for Plasma Physics), P. Steinbrunner (Max Planck Institute for Plasma Physics, University of Greifswald), M. R. Stoneking (Lawrence University), C. M. Surko (University of California San Diego) and A. Zetti (Lawrence University).

Abstract

The creation and study of confined, long-lived, electron-positron plasma in the laboratory is the grand challenge of the APEX (A Positron-Electron eXperiment) Collaboration. Conducting experiments with this unique hybrid of matter and antimatter will enable comparisons to fundamental plasma physics predictions for this uncommonly symmetric system. Ideally, these can in turn contribute to our understanding of positrons and/or pair plasmas in astrophysics --- or even the early universe. As part of striving towards this goal, we are employing, validating, combining, and advancing diverse, state-of-the-art science and technology [1]. This talk will give an overview of recent highlights --- including novel techniques in the areas of non-neutral plasmas [2], positron beams, and gamma diagnostics [3] --- as well as the progress on our two, complementary, table top-sized, pair-plasma traps: a levitated dipole and an optimized stellarator, both based on small, non-insulated, HTS (high-temperature superconducting) coils. Finally, it will summarize our roadmap for the next few years, when we will put all of these elements together.

Acknowledgments We gratefully acknowledge funding from the Helmholtz Association; the Deutsche Forschungsgemeinschaft (DFG); the Alexander von Humboldt Foundation; the UC San Diego Foundation; the Deutscher Akademischer Austauschdienst (DAAD) RISE program; the United States Department of Energy; the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme; the Japan Society for the Promotion of Science (JSPS); and the National Institute for Fusion Science (NIFS).

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JET Isotope Studies and the L-H Transition

Emilia R Solano (Laboratorio Nacional de Fusión, CIEMAT), for the JET L2H Team (See author list of E. R. Solano et al. Nucl. Fusion 63 112011 (2023)), and JET contributors (See author list of C. F. Maggi et al. Nucl. Fusion 63 110201).

Abstract

The transition between L and H-mode has fascinated plasma physicists since its discovery [1]. It is a clear phase transition between plasma confinement regimes, which takes place when the plasma is sufficiently heated. Here we present and discuss results from recent dedicated L-H transition experiments at JET [2]. Uniquely, we studied the power threshold (PLH) in plasmas composed of pure Tritium, to be compared with Hydrogen and Deuterium plasmas, D+T mixtures, and H+T mixtures [3]. We show that critical pressure profiles are required for the L-H transition to occur, and such critical profiles are independent of plasma content, but the power threshold itself depends strongly on isotopic content. PLH is in fact determined by the plasma transport characteristics in L-mode. We also show that an analysis of Doppler reflectometer measurements of the edge perpendicular velocity in D and He plasmas and observe that there are no critical radial electric field value or critical $\mathbf{v}_{\text{ExB}}$ rotation before the L-H transition [4]. Instead, there appears to a $\mathbf{v}_{\text{ExB}}$ profile that is characteristic of the L-mode. The diamagnetic velocity, proportional to ∇p , is a better indicator of proximity to the L-H transition. Together, these results enable us to challenge the widely accepted model of the L-H transition being associated to the stabilisation of electrostatic turbulence by sufficient $\mathbf{v}_{\text{ExB}}$ shear. An alternate model of the L-H transition is briefly discussed, based on a magnetisation transition of the plasma.

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11:40-12:10 [We1.A.11] Data-Driven Insights into Heavy Impurity Dynamics in Drift-Wave Turbulence

Zetao Lin (Aix-Marseille University), T. Maurel-Oujia (Aix-Marseille University), B. Kadoch (Aix-Marseille University), S. Benkadda (Aix-Marseille University) and K. Schneider (Aix-Marseille University).

Abstract

Confinement quality in fusion plasma is significantly influenced by the presence of heavy impurities, which can lead to radiative heat loss and reduced confinement. This study explores the clustering of heavy impurity, i.e., Tungsten in edge plasma of tokamaks. The two-dimensional Hasegawa-Wakatani model of drift-wave turbulence is used as a paradigm to describe edge tokamak turbulence. To this end high-resolution direct numerical simulations of the Hasegawa-Wakatani equations are carried out with millions of charged inertial particles. We use Stokes number to quantify the inertia of impurity particles. It is found that particle inertia will cause spatial intermittency in particle distribution and the formation of large-scale structures, i.e., the clustering of impurity particles. The degrees of clustering are influenced by Stokes number [1]. To quantify these observations, we apply a modified Voronoi tessellation method, which assigns specific volumes to impurity particles [2]. By determining time changes of these volumes, the impurity velocity divergence can be calculated, where negative values indicate cluster formation while positive values correspond to cluster destruction. Thus the clustering dynamics can be assessed. Additionally, Lagrangian statistics are used to provide further insights into the dynamics of heavy impurity behavior in the edge plasma.

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12:10-12:30 [We1.A.O1] Integrated Data Analysis of the Tungsten Concentration Profiles at WEST Using Soft X-Ray and Bolometer Diagnostics

Hao Wu (Ghent University), Axel Jardin (Institute of Nuclear Physics, Polish Academy of Sciences), Tianbo Wang (ITER Organization), Didier Mazon (Institute for Magnetic Fusion Research, CEA) and Geert Verdoolaege (Ghent University).

Abstract

Tungsten (W) has been selected as the divertor and first wall material for the International Thermonuclear Experimental Reactor (ITER) project [1, 2]. The tungsten divertor technology of ITER is tested on several devices, including the W Environment in Steady-state Tokamak (WEST) located at Cadarache. Despite the high melting point and low erosion rate of W, the interactions between the edge plasma and the plasma-facing components are an important source of tungsten impurities in the plasma. The accumulation of heavy impurities like tungsten in the plasma core of fusion devices poses a major risk to the core performance through strong radiative cooling. Therefore, reliable estimates of impurity concentrations in fusion devices are of vital importance. The soft X-ray (SXR) diagnostic system at WEST records the line-integrated SXR emissivity from impurities as well as the bulk plasma [2]. The two-dimensional SXR emissivity profile in a poloidal cross section can be reconstructed through Bayesian inference with a nonstationary Gaussian process prior, resulting in a closed-form expression for the posterior distribution [3]. Combined with density and temperature measurements, the tungsten concentration profile can be directly inferred from the reconstructed emissivity profile. However, such sequential analysis does not take into account the various sources of uncertainty from the individual diagnostic systems. Instead, we use Bayesian probability theory to jointly estimate the density, temperature and tungsten concentration profiles from their joint posterior distribution. This approach is called integrated data analysis (IDA) in the fusion community [4]. The IDA approach enables reliable uncertainty estimates without explicit error propagation analysis and allows exploiting the diagnostics' interdependencies. The high-dimensional joint posterior distribution of the quantities of interest can be explored by a Markov chain Monte Carlo (MCMC) sampler. Tests on synthetic data suggest large uncertainty on the inferred tungsten concentration near the plasma edge, due to the weak SXR signal from low ionization states of tungsten at low electron temperature. Therefore, to improve the edge inference we also investigate the possibility of including additional diagnostics, like bolometry. First results using synthetic data show that this is a promising approach.

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11:40-12:10 [We1.B.I1] Experimental Investigation of Laser Plasma Interaction in the Context of Inertial Confinement Fusion on the Laser Mégajoule Facility

Sylvie Depierreux (CEA), Véronique Tassin (CEA) and Marion Lafon (CEA).

Abstract

Since the end of 2021, the Laser Mégajoule (LMJ) at CEA in France can be operated in a symmetric configuration with 10 laser chains [80 beams distributed into 2 upper hemisphere cones (polar angle 33° and 49°) and 2 lower hemisphere cones (147° and 131°)] giving up to ~ 270 kJ delivered at 351 nm. LMJ presently offers more than 14 plasma diagnostics operating in the visible-UV wavelengths ranges and in multiple hard and soft x-ray ranges as well as particles diagnostics (neutrons, electrons and ions). Notably, Laser Plasma Interaction (LPI) is characterized on LMJ with (i) backscattering measurements (time-resolved spectra end energy) on one inner quadruplet and (ii) near backscattering [1] covering polar angles from 20° to 65° over $\sim 1/5$ of the azimuth of the LMJ target chamber.

In the indirect-drive configuration to ICF, the laser energy is converted into a bath of x-rays inside a high-Z enclosure (a hohlraum) that further implodes the capsule (containing the fuel), its symmetry of irradiation being adjusted through the energy balance between inner and outer cones of beams. A specific “rugby-ball shaped” hohlraum design (in contrast to the regular cylinder shape) is well suited to the LMJ energy partition with its $\frac{1}{2}$ - $\frac{1}{2}$ power balance. However, this particular prolate spheroid hohlraum shape introduces challenges in the control of the inner beams propagation. Namely, the significant expansion of the gold hohlraum wall into the path of the inners can give rise to significant Stimulated Brillouin Scattering (SBS). The latter involves the coupling of the incident laser with ion acoustic waves thus amplifying scattered electromagnetic waves. Large amounts of SBS may be expected for inner beams propagating in the high-Z mm scale gold bubble plasma.

In the direct-drive configuration to ICF, the laser beams irradiate the capsule in a spherical geometry giving rise to an expanding exponential density profile plasma. The coupling of the laser beams with such a plasma at the relevant scale (laser intensity, plasma temperature, density scale length) had been studied with a planar geometry. The unique angular coverage of the LMJ LPI diagnostics recently allowed to investigate the Raman scattering mechanisms responsible for the hot electrons generation in direct-drive ICF experiments thanks to this planar target platform. We will report on the first indirect-drive experiments performed on LMJ in rugby ball-shaped gas-filled hohlraums with laser pulses duration up to 9 ns. We will illustrate the challenges of getting a round implosion while mastering the LPI mechanisms. The configuration and preliminary results of the planar direct-drive experiments will also be presented.

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12:10-12:30 [We1.B.O1] Filamentation of Currents and Generation of Strong Magnetic Fields Due to Electron Weibel Instability in an Expanding Collisionless Plasma

Vladimir Kocharovsky (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences), Anton Nechaev (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences) and Mikhail Garasev (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences).

Abstract

The talk aims at the classical problem of the decay of a strong plasma discontinuity and a similar problem of the injection of a plasma with hot electrons into a vacuum or a rarefied cold plasma with a magnetic field in the absence of significant particle collisions. The main goal is to elucidate the transient phenomena of the formation of an anisotropic electron velocity distribution leading to quasi-magnetostatic turbulence associated with Weibel-type instabilities. We focus mainly on the formation and decay of current filaments and sheets and review the theoretical and numerical results on this account obtained previously for space and laser plasmas.

We describe and analyze in detail the original results of particle-in-cell modeling of a collisionless expansion of an anisotropic plasma cloud with hot electrons from a flat surface into a background magnetoactive plasma in different settings entailing [1] (i) a hot-electron spot of a circular or cylindrical form within an initial-value problem or a finite-time injection of electrons from the surface, (ii) an external magnetic field with three orthogonal orientations: perpendicular to the surface or along it, directed either across or parallel to a long axis of the hot-electron spot, and (iii) inhomogeneous layers of cold background plasma of different spatial scales and densities.

Bearing in mind typical laser-plasma experiments, we outline the development of the principal structures of currents and highly inhomogeneous magnetic fields linked with distinct forms of the anisotropic electron velocity distribution and sophisticated dynamics of the instability process for diverse sets of attributes (i)–(iii). Strong magnetic fields generated at the main transition stage are turbulent in nature, can reach, in typical laser experiments, mega-Gauss and higher values, and modify interaction between charged particles notably. In particular, according to the analytical estimates, numerical simulations and laser ablation experiments, the magnetic fields of self-consistent currents of cold and hot electrons quickly lead to the spatial separation of particle counter flows and suppress the beam instability of plasma (Langmuir) waves. This situation is qualitatively different from the well-studied decay of a weak plasma discontinuity, where the electrons obey a Boltzmann distribution and the formation of magnetic turbulence is replaced with the generation of ion-acoustic solitons. We discuss applications of the obtained results to the analysis of laboratory and space plasma problems involving an explosive development of the small-scale magnetic turbulence due to the filamentation of electric currents.

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11:40-12:10 [We1.C.I1] Dissipative Structures In Non-Ideal MHD Turbulence: The Hall Effect Unveiled

Turlough Downes (Dublin City University) and A. Kinsella (Dublin City University).

Abstract

Turbulence involves the transport of energy from large scales to the small scales at which it is dissipated. The properties of the structures which dissipate the magnetohydrodynamic (MHD) turbulence in star forming regions are important in determining the conditions within which stars and planets form. In MHD turbulence a significant fraction of energy is dissipated in current sheets which are formed as magnetic field lines are pressed together. In planet-forming discs these current sheets influence the chemistry and even the ionisation fraction, and thus the dynamics, of the plasma thereby potentially influencing star and planet formation.

Dissipation of energy in current sheets has been studied in the context of compressible and incompressible non-ideal MHD incorporating the effects of both Ohmic and ambipolar resistivity [1, 2]. However, these studies have not incorporated the Hall effect which is thought to be important during gravitational collapse (in molecular clouds) and certain parts of planet-forming discs.

In this work we report, for the first time, on the impact of the Hall effect on dissipative structures in MHD turbulence. We use the multi-fluid MHD code HYDRA [3, 4] to simulate driven, multi-fluid MHD turbulence and show that the size distribution of current sheets is changed, resulting in energy being dissipated equally across current sheets of all sizes, in contrast to previous results without the Hall effect and similar to previously published results for ideal MHD [5].

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12:10-12:30 [We1.C.01] Non-Axisymmetric Accretion Disk Instabilities: New Possibilities Beyond the Magnetorotational Instability

Nicolas Brughmans (CmPA, KU Leuven), Rony Keppens (CmPA, KU Leuven) and Hans Goedbloed (DIFFER).

Abstract

Black hole accretion disks are assumed to be turbulent as they are highly unstable to magnetic instabilities driven by shear flow, resulting in angular momentum transport and an effective turbulent viscosity. The axisymmetric, weak-field magnetorotational instability (MRI) and its derivation through local WKB approximations has been the topic of many studies [1]. In contrast, its non-axisymmetric counterparts have received relatively little attention as they require the full power of magnetohydrodynamic (MHD) spectroscopy for a general description of the eigenspectrum of waves and instabilities [2]. This is essential for global cylindrical disk models, where the influence singularities from the overlapping MHD continua complicates the analysis. Recently, rigorous MHD spectroscopy identified a new type of ultra-localised, non-axisymmetric instability in global cylindrical disks with super-Alfvénic flow [3]. These super-Alfvénic rotational instabilities (SARIs) make up 2D unstable regions in the complex eigenfrequency plane with (near-eigen)modes that corotate at the local Doppler velocity and are radially localised between Alfvénic resonances, utterly insensitive to inner/outer radial disk boundaries.

Here, we independently confirm the existence of these unprecedented modes using the novel spectral MHD code Legolas (<https://legolas.science/>), reproducing and extending our earlier study with detailed eigenspectra and eigenfunctions [4]. We also compare growth rates of the discrete SARIs and MRI in a variety of disk equilibria, highlighting the impact of field strength and orientation, as well as compressibility in suprathermal fields. We show that non-axisymmetric modes can significantly extend instability regimes at high mode numbers, with instability determined by the Alfvén frequency and maximal growth rates comparable to the MRI. Furthermore, we explicitly show the existence of modes that are highly localised in all directions, with possible applications to global and shearing box simulations. A visualisation in the time and space domain of MRI/SARI modes highlights their differences and similarities and gives further indication that the onset instability in accretion disks could very well be governed by localised non-axisymmetric SARI modes.

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Thursday
September 12

Ignition Achieved: next Steps in the Path Toward an Inertial Fusion Energy Future

Tammy Ma (Lawrence Livermore National Laboratory), Mariann Albrecht (Lawrence Livermore National Laboratory), M. John Edwards (Lawrence Livermore National Laboratory), Justin Galbraith (Lawrence Livermore National Laboratory), Clement Goyon (Lawrence Livermore National Laboratory), Thomas Lynch (Lawrence Livermore National Laboratory), Veronika Kruse (Lawrence Livermore National Laboratory), James McCarrick (Lawrence Livermore National Laboratory), Vincent Tang (Lawrence Livermore National Laboratory) and G. Jackson Williams (Lawrence Livermore National Laboratory).

Abstract

The achievement of ignition on the National Ignition Facility in 2022 demonstrated the fundamental feasibility of controlled thermonuclear fusion in the laboratory for energy gain, and was the first major hurdle in efficiently harvesting fusion energy through inertial fusion energy (IFE). Excitement has been growing worldwide, with notable activity in the public and private sectors. To make IFE commercially viable, however, there are still significant scientific, engineering, workforce, and economic hurdles. This talk will review the advancements that made the ignition breakthrough possible, provide an overview of the international IFE landscape, and describe the remaining gaps and challenges that must be solved to realize IFE laser inertial fusion as a path for clean energy and energy security.

Laser Plasma Accelerators: Manipulating Relativistic Electrons with Intense Lasers

Victor Malka (Weizmann Institute of Science).

Abstract

Laser-plasma-based wakefield accelerators are changing the scientific landscape bringing on new hopes for high energy physics, compact light sources, and societal applications. Many of these applications critically require the precise characterization of the plasma wakefield that largely affects the bunch's quality they provide. Advanced diagnostics of such highly transient, microscopic bunch and field structures, however, remains very challenging. After introducing the context and the status of this research, I will shortly explain the physical processes that are involved in plasma accelerators, I then will report on recent major results that demonstrate for the first time the real-time visualization of laser-driven nonlinear relativistic plasma wave[1], its transition to electron-driven wakefield [2] and the femtosecond microscopy of relativistic electron bunch [3].

This will be followed by a short review of the most mature applications including the status of our EIC project ebeam4therapy [4].

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Design of Plasma Shapes for Metamaterials, Chemical Filters, and Maze-Solvers

Osamu Sakai (The University of Shiga Prefecture).

Abstract

Plasma in a gas phase forms in a free space or in a container, and its shapes and motions have attracted much scientific attentions for decades. In the outer space, it exists magnetohydrodynamically in rich forms in the background of the external magnetic fields. Fusion plasma in a reactor is produced in a large vacuum vessel to configure its form to maximize its energy output, keeping its stability. Low-temperature plasmas, which are industrial tools as well as fundamental scientific models, have been reported in many shapes under elaborate controls since they play various roles with specific aims in non-equilibrium states, such as light sources, material processing, and medical treatments. For instance, we reported plasma photonic crystals and plasma metamaterials, which have spatial periodic structure and work as microwave regulators [1,2]. In another study, a filter-like thin plasma sheet is equivalent to a controllable chemical filter [3]. Although they are deliberately designed to optimize their functional outputs, in some cases we have no ideas on scientific principles in its spatial design since they are fixed after simple trial-and-error procedures.

In this study, using our previous research results [1-4] and other relevant scientific achievements, we revisit relationships between functions of low-temperature plasmas and their shapes. Recently, we experimentally developed a maze-solver based on plasma channel expansion [4] and examined such phenomena using Boltzmann-Gibbs (thermodynamical) and Shannon (information) entropies. From this point of view, we can reconsider functions of plasma shapes in our previous research results such as plasma metamaterials and plasma chemical filters, both of which possessed designed shapes for specific purposes in non-equilibrium states. A clue to overall understanding of functions and shapes may be given by entropy estimation, and low-entropy states will be of benefit to improved functions.

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11:40-12:10 [Th1.A.I1] Physics Drivers for the Plasma-Facing Components Design of the COMPASS-U Tokamak

Renaud Dejarnac (Institute of Plasma Physics of the Czech Academy of Sciences), Jakub Caloud (Institute of Plasma Physics of the Czech Academy of Sciences), Ekaterina Matveeva (Institute of Plasma Physics of the Czech Academy of Sciences), Jonathan Gerardin (Institute of Plasma Physics of the Czech Academy of Sciences), Jozef Havlicek (Institute of Plasma Physics of the Czech Academy of Sciences), Jakub Hromadka (Institute of Plasma Physics of the Czech Academy of Sciences), Martin Imrisek (Institute of Plasma Physics of the Czech Academy of Sciences), Fabien Jaulmes (Institute of Plasma Physics of the Czech Academy of Sciences) and Matej Peterka (Institute of Plasma Physics of the Czech Academy of Sciences).

Abstract

COMPASS-U is a new tokamak which is currently under construction at the Institute of Plasma Physics of the Czech Academy of Sciences in Prague [1]. Its relatively small size ($R = 0.9$ m, $a = 0.27$ m), compared to other existing or planned tokamaks, combined to a high magnetic field (5 T) and a high plasma current (2 MA) makes it very challenging to design, especially its plasma-facing components. Indeed, the thermal energy confined in the plasma needs to be extracted on a limited area in the divertor yielding extremely high energy flux densities, few times higher than the ones met at the surface of the Sun. The large magnetic pressure needed to keep the hot plasma far away from the wall can be, in case of a sudden loss of confinement so-called 'disruptions', accidentally transferred to in-vessel components in a fraction of millisecond, yielding large electromagnetic (EM) $J \times B$ forces, ranging from several tens of kN on some tiles up to 4 MN on the vacuum vessel [2]. COMPASS-U being a unique new machine, these loads cannot be estimated from previous experiments and need to be determined.

This contribution describes how plasma physics considerations are used for engineering purposes in order to design the plasma-facing components of a tokamak. Design requirements are driven by the physics to be investigated. The large variety of foreseen plasma scenarios and magnetic equilibria, for investigating different types of physics, adds to the challenge as a single first wall configuration must cope with a broad range of completely different constraints. The different loads are estimated from scaling laws derived from multi-machine databases, simulations and extrapolation from existing devices. As an example, heat loads at the divertor targets follow the Eich's law and profiles [3]. It yields for COMPASS-U a power fall-off length in the edge plasma close to the wall of $\lambda_{q^{omp}} \sim 1$ mm and consequently steady-state energy flux densities up to 100 MW/m² if no mitigation is applied. Other type of thermal loads, more transient but more powerful, have to be also considered, e.g., runaway electrons with energy in the range 100-300 kJ or edge localized modes, adding to the risk of melting the tungsten tiles. During disruptions, the different currents induced (eddy) and flowing (halo) into the structures due to the temporal variation of magnetic field, caused by vertical displacements and by the plasma current quench, are determined using an EM model based on finite element modeling and multi-machine databases created for ITER under the International Tokamak Physics Activity [4].

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12:10-12:40 [Th1.A.12] Impact of Co-Dependent Energy and Angular Atomic Impact Spectra on Tungsten Erosion in JET

Henri Kumpulainen (Forschungszentrum Jülich), Detlev Reiter (Heinrich-Heine-Universität Düsseldorf), Sebastijan Brezinsek (Forschungszentrum Jülich), Mathias Groth (Aalto University), Juri Romazanov (Forschungszentrum Jülich) and Sven Wiesen (Dutch Institute for Fundamental Energy Research).

Abstract

The erosion of tungsten (W) by fast deuterium (D) atoms from charge-exchange reactions, predicted to be the dominant cause of the observed W radiation in most JET ITER-like-wall plasmas [1], is determined by the co-dependent energy and angular distributions and the flux of incident D atoms. Monte Carlo simulations using conventional histogram binning methods compromise between high resolution and low stochastic noise. In contrast, tallying functional expansion coefficients of the angular spectrum for each energy bin provides both arbitrarily high angular resolution and reduced Monte Carlo noise compared to angular histogram binning.

EIRENE simulations of JET L-mode and H-mode plasmas using newly implemented functional expansion tallies with a Legendre polynomial basis indicate that the most common D impact angles near the JET low-field side divertor entrance are in the range of 60° to 85° to the surface normal at impact energies (E) of 100 to 700 eV and 30° to 50° at $E > 1$ keV. The predicted W sputtering due to atoms and the W density in the core plasma is increased by up to one-third due to the energy-resolved angular spectra, compared to the total energy-independent angular spectrum and to earlier simulations [1] which assumed a constant impact angle of 60° . As the atomic D flux density decreases with higher impact energy, the increased W sputtering yield due to larger impact angles at $250 \text{ eV} < E < 700 \text{ eV}$ is more significant than the reduced W yield due to more perpendicular angles at $E > 1$ keV.

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11:40-12:10 [Th1.B.11] Raman Amplification with a 1×10^{15} W/cm² Seed

Jessica Shaw (University of Rochester Laboratory for Laser Energetics), Manfred Virgil Ambat (University of Rochester Laboratory for Laser Energetics), Kyle McMillen (University of Rochester Laboratory for Laser Energetics), Jeremy Pigeon (University of Rochester Laboratory for Laser Energetics), Sara Bucht (University of Rochester Laboratory for Laser Energetics), Isabelle LaBelle (University of Rochester Laboratory for Laser Energetics), Hans Rinderknecht (University of Rochester Laboratory for Laser Energetics), Dan Haberberger (University of Rochester Laboratory for Laser Energetics), Kassie Moczulski (University of Rochester), Petros Tzeferacos (University of Rochester), John Palastro (University of Rochester Laboratory for Laser Energetics), Adam Sefkow (University of Rochester) and Dustin Froula (University of Rochester Laboratory for Laser Energetics).

Abstract

We present experimental results from the Raman Amplification experimental platform at the University of Rochester's Laboratory for Laser Energetics (LLE). This platform explores Raman amplification in a unique parameter space which includes a multi-joule pump and an adjustable-energy seed with intensities exceeding 1×10^{15} W/cm². Initial experiments have demonstrated single-pass Raman amplification in multiple focal configurations with energy gain factors as high as 30x and record efficiencies as high as 11.7%. Amplification factor and efficiency scalings with plasma density, focal geometry, pump energy, and seed energy are presented. The impact of propagation of both the pump and seed lasers on the amplification process is explored.

Acknowledgments This material is based upon work supported by the Department of Energy [National Nuclear Security Administration] University of Rochester "National Inertial Confinement Fusion Program" under Award Number DE-NA0004144, the U.S. Department of Energy under Awards DE-SC0021057 and DE-SC0016253.

12:10-12:30. [Th1.B.O1] Plasma Optics for Compression of Ultraintense Laser Pulses

Minsup Hur (UNIST).

Abstract

Exawatt or zettawatt laser pulses can be groundbreaking scientific tools for uncovering the mysteries of our universe. These high-power laser pulses enable experimental studies of phenomena such as vacuum boiling and pair-production, Hawking radiation, and quantum gravity, holding great potential for advancing our understanding of nature. However, the currently available CPA technology is stuck at petawatt level, mainly due to the material breakdown of compression gratings and alternative solutions are yet to come from conventional optics. Plasma, being in a state of matter already broken down, can endure highly intense laser fields without damage. Its resilience to strong fields, coupled with its optically dispersive properties, makes plasma an ideal medium for manipulating high-power laser pulses. In this presentation, I will present a recently devised novel concept for pulse compression using a density gradient, high density plasma. The point of the new idea is generating reflection path difference of photons of a chirped pulse using a near-critical, gradient density plasma. As the higher frequency photons take longer reflection path deeper into the plasma, the photons in the tail of a negatively chirped pulse can catch up those in the pulse front, resulting in concentration of the photons into a narrow region. So far this idea is still in theoretical and modelling level, but it has been already published to Nature Photonics in 2023 [1], and experimental proof-of-principle is in progress. In theoretical point of view, combining different plasma optics ideas with the original density gradient idea is being studied. Here I present the status of the research and future vision of this innovative concept, providing comparison of characteristics of previous plasma-based schemes such as Raman, Brillouin and plasma gratings. These advancements can be crucial steps toward realizing compact, exawatt, or zettawatt laser pulses.

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11:40-12:10 [Th1.C.I1] Relaxation of Energetic Particles in Plasmas and Dark Matter in Galaxies Through a Common Resonance-Broadened Kinetic Theory [IUPAP Early Career Scientist Prize]

Vinicius Duarte (Princeton Plasma Physics Laboratory).

Abstract

A quasilinear plasma transport theory that incorporates Fokker-Planck dynamical friction (drag) and pitch angle scattering is self-consistently derived from first principles for an isolated, marginally-unstable mode resonating with an energetic minority species. It is found that drag fundamentally changes the structure of the wave-particle resonance, breaking its symmetry and leading to the shifting and splitting of resonance lines. In contrast, scattering broadens the resonance in a symmetric fashion. Comparison with fully nonlinear simulations shows that the proposed quasilinear system preserves the exact instability saturation amplitude and the corresponding particle redistribution of the fully nonlinear theory. Even in situations in which drag leads to a relatively small resonance shift, it still underpins major changes in there distribution of resonant particles. This novel influence of drag is equally important in plasmas and self-gravitating systems. In fusion plasmas, the effects are especially pronounced for fast-ion-driven instabilities in tokamaks with low aspect ratio or negative triangularity, as evidenced by past observations. The same theory directly maps to the resonant dynamics of the rotating galactic bar and massive bodies in its orbit, providing new techniques for analyzing galactic dynamics and for constraining candidates of dark matter particles.

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12:10-12:30 [Th1.C.01] One-Dimensional Model for Plasma-Flow from the Accretion Disk Towards the Neutron-Star Poles

Anoop Singh (Institute for Plasma Research), Mrityunjay Kundu (Institute for Plasma Research) and Shishir P. Deshpande (Institute for Plasma Research).

Abstract

Accreting neutron stars (ANS) are unique cosmic laboratories for studying the exotic plasma phenomena. ANS are characterised by strong surface gravity $\sim 10^{14} \text{cm/s}^2$ and a wide range of strong dipole magnetic fields $\sim 10^8\text{-}10^{14} \text{Gauss}$ and have a binary companion that feeds the ANS with matter, often creating the accretion disk (AD) [1]. We consider the simplest case of steady-state mass accretion and find intriguing solutions by applying the well-known MHD model.

The large magnetic field causes the ions and electrons to flow along the field lines, given in this case by the dipole approximation [2]. A slowly rotating ANS is considered, with its magnetic axis aligned with the rotation axis for simplicity. We show that for a given accretion rate, the conservation laws define boundaries of parameter-space where meaningful solutions can be found. It is also seen that the region near the poles, where the plasma gets concentrated, has an incredibly small thickness in the θ -direction. As the highly conducting plasma, attached to the converging magnetic field lines of increasing strengths, is channelled to the polar region of the neutron star acquires remarkably high densities and, in most cases, remains sub-alfvenic [3]. As the plasma continues to move further towards the ANS poles, the gravity and the magnetic field topology causes a compression of the plasma [2]. Diamagnetic effects due to a sharp radial pressure gradient cause a toroidal sheet to form on the outer and inner sides of the envelope defined by field lines binding the accretion disk. A weak electrical resistivity is enough to drive a significant poloidal current in the ANS magnetosphere. Finally, the conditions that may mark the onset of ideal and resistive MHD instabilities are delineated.

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14:40-15:00 [Th2.A.O1] High-Frequency Observation of Pedestal Turbulence and Its Impact on ELM Dynamics in KSTAR

Jaehyun Lee (Korea Institute of Fusion Energy (KFE)), Minho Kim (Korea Institute of Fusion Energy (KFE)), Gunsu Yun (Pohang University of Science and Technology (POSTECH)), Minwoo Kim (Korea Institute of Fusion Energy (KFE)), Jae-Min Kwon (Korea Institute of Fusion Energy (KFE)), Juhyung Kim (Korea Institute of Fusion Energy (KFE)), Sumin Yi (Korea Institute of Fusion Energy (KFE)) and Sehoon Ko (Korea Institute of Fusion Energy (KFE)).

Abstract

Pedestal turbulence manifests as electron temperature fluctuations while the electron transport barrier remains intact until an edge-localized mode (ELM) crash occurs. Considering the crucial role of pedestal turbulence in the evolution and collapse of the pedestal, this study investigates the microscopic spatial structure and dynamics of these temperature fluctuations to elucidate the role of pedestal turbulence in the electron transport barrier [1,2]. We comprehensively compared electron temperature fluctuations observed during pedestal evolution under various instability conditions, explaining the role of pedestal turbulence in both the evolution and collapse phases. A novel diagnostic method was employed to enhance the turbulence characteristics in the KSTAR pedestal [3]. Utilizing a newly developed high-speed digitizer, we measured broadband electron cyclotron emission (ECE) to precisely observe high-frequency pedestal turbulence during both inter-ELM crash and resonant magnetic perturbation (RMP)-driven ELM suppression. Detailed comparisons with the characteristics of various instabilities revealed that the micro-tearing mode is associated with pedestal evolution and collapse. Conversely, the turbulence characteristics during ELM suppression align more closely with interchange modes, suggesting that turbulence-driven transport can induce distinct pedestal structures and ELM dynamics. To evaluate the impact of these turbulent fluctuations on pedestal evolution and collapse, we calculated the quadratic transfer function (QTF) [4]. The QTF results indicate that during the inter-ELM crash period, pedestal energy is nonlinearly transferred from turbulent eddies to magnetohydrodynamic (MHD) modes, causing the mode structure to expand radially. However, during RMP-driven ELM suppression, dominant modes do not grow due to energy exchange among turbulent eddies within the pedestal.

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15:00-15:20 [Th2.A.O2] Pulsed Hydrogen Plasma Stream Interaction on Tungsten Substrate

Tridip Kumar Borthakur (Centre of Plasma Physics Institute for Plasma Research), Azmirah Ahmed (Centre of Plasma Physics Institute for Plasma Research), Sumit Singha (Centre of Plasma Physics Institute for Plasma Research), Pallabi Baruah (Centre of Plasma Physics Institute for Plasma Research), Pradipta Prakash Kalita (Centre of Plasma Physics Institute for Plasma Research) and Nirod Kumar Neog (Centre of Plasma Physics Institute for Plasma Research).

Abstract

A Pulsed Plasma Accelerator (PPA) with coaxial electrodes system, is being used to produce a high speed (several km/s), high density ($\sim 10^{20}/\text{m}^3$) Hydrogen plasma stream by applying power from a 200 kJ Pulsed Power System (PPS). The PPS, which consists of two modules capacitor banks, can be charged up to 15 kV to achieve 200 kJ of energy. This PPS generates a peak discharge current of 100 kA for a half time period of 500 μs . A gas injection valve is used to supply the requisite gas during the application of high voltage discharge pulse in between the two electrodes. The high voltage thus applied breaks down the gaseous medium in between the electrodes to form plasma sheet which is driven by the $\mathbf{J} \times \mathbf{B}$ force towards the open end of the electrodes to form a high-density plasma stream. In this work, the high speed Hydrogen plasma stream is allowed to fall on Tungsten substrates placed at a distance of 10 cm from the end of the electrodes. The measured heat energy density of the Hydrogen stream at this position is 0.205 MJ/m² while it increases up to 0.224 MJ/m² under an influence of 0.1 Tesla external magnetic field. The plasma matter interaction at this condition creates blister formation to the surface of Tungsten material for single exposure. However minor and major cracks, displacement of cracked surface, dust formation, re-deposition are observed in scanning electron micrograph for surface exposed for 15 times to the same plasma stream. The interaction due to the heat energy density of plasma stream on Tungsten material in this work resembles either a mitigated or lower energy type-I Edge Localized Mode (ELM) [1] and the reported results are highly relevant for fusion reactor.

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14:40-15:10 [Th2.B.11] Developing Community Engagement for RM3FD: Repository, Management, and Modeling for Materials Fundamental Data

Mark Koepke (West Virginia Univ and Tokamak Energy).

Abstract

Taking the lead in developing, collecting, curating, modeling, and making accessible online a repository for fundamental data characterizing novel materials that can withstand enormous heat and neutron exposure is important for controlled fusion energy and the design basis for a fusion pilot plant (FPP). The RM3FD is envisioned as a partnership for open access of high-value, high-effort, fully referenced, intellectual property for mutually shared, straightforward to implement, and as a benefit toward commercial fusion energy development.

We are motivated to form an association for the repository, management, and modeling of materials fundamental data to address the recognized need for such a community data archive both for modeling consistency and for improved transparency in interpreting experiments. Resources of the envisaged RM3FD association could include annual membership dues, public or private donations and grants, and income from services rendered by the association. One model for this endeavor is the LXCat endeavor [1] (www.lxcat.net) for the low-temperature plasma science community. The objective of this presentation is to recruit interest in facilitating the exchange of data and numerical tools for modeling Plasma-Materials Interfaces in Advanced Materials and Fusion Nuclear Science, Engineering, & Technology. Interested partners are welcome to join the effort as stakeholders (users, contributors, advisors, and leaders within the association) in advancing a proposal to government agencies.

Notional objectives:

- Encourage the exchange and the open access of fundamental data via the internet.
- Develop tools to facilitate the exchange of on-line data.
- Contribute to the collection and evaluation of data.
- Communicate with international fusion-materials community.
- Appeal to the Modeling for Fusion Materials Fundamental Data Network/Forum.
- Encourage open access of numerical tools for predictive modeling & analysis.
- Contribute to the standardization of formats for storing of and exchanging of data

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15:10-15:30 [Th2.B.O1] Experimental and Modeling Studies of EIR and MAR Processes on Detached Plasmas Using the Linear Plasma Device NAGDIS-II

Noriyasu Ohno (Nagoya University), Ryoji Mano (Nagoya University), Yuta Uematsu (Nagoya University), Hiroki Natsume (Tokai University), Hirohiko Tanaka (Nagoya University), Yuki Hayashi (the University of Tokyo), Shin Kajita (the University of Tokyo), Jieli Shi (Dalian University of Technology) and Keiji Sawada (Shinshu University).

Abstract

Reducing the heat load on divertor plates is crucial for the development of a magnetic confinement plasma fusion power reactor. To achieve this goal, the formation of detached plasma through plasma-gas interaction is a promising method, and accurate modeling of detached plasmas is essential for designing DEMO reactors. In this study, we constructed a digital twin by comparing detached plasma experiments conducted using a linear plasma machine with simulations integrating calculation codes for plasma transport, neutral transport, and collisional radiative processes. This presentation describes the results of both the experiments and simulations on linear detached plasma.

The linear plasma machine NAGDIS-II is used for the experiments [1]. This device can produce steady and high-density plasmas exceeding 10^{19}m^{-3} using DC discharges. Laser Thomson scattering measurements are installed in the upstream and downstream regions [2], and a 2D-driven spectroscopic and electrostatic measurement system is located downstream. The importance of the role of volume recombination as a particle loss process in detached recombining plasmas has been experimentally demonstrated, mainly using helium plasmas. The two-dimensional (2D) spatial structure of detached recombining plasmas, where the electron-ion recombination (EIR) is dominant, has also been clarified [3]. Furthermore, in the recombining region, the increase in plasma fluctuation and the associated increase in radial transport and structural changes have been revealed by time-space resolved 4D tomography using an electrostatic probe and a high-speed camera [4].

For hydrogen isotope plasmas, the competing processes of molecular assisted recombination (MAR) associated with excited hydrogen molecules and EIR have been investigated in detail. In deuterium plasmas, a transition from MAR to EIR is observed with increasing gas pressure [5]. Furthermore, comparative experiments between hydrogen and deuterium plasmas have been carried out, and isotope effects have been observed in which MAR is dominant in hydrogen plasmas compared to deuterium plasmas. To reproduce the above experimental results, a simulation code (DISCOVER: Detached plasma Integrated Simulation COde with Various Elastic/ inelastic Reactions), which integrates fluid, neutral and collisional radiative codes (including atomic and molecular species), has been developed by considering the transport of excited atoms and molecules generated by volume recombination and wall recycling.

Acknowledgments This work was supported by JSPS KAKENHI Grant Numbers (20H00138, 22H01203, 24H00201)

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14:40-15:10 [Th2.C.I1] *Ab Initio Kinetic Simulations Meet Machine Learning: Opportunities in Plasma Physics*

E. Paulo Alves (University of California, Los Angeles).

Abstract

First-principles kinetic simulations have long been indispensable tools for advancing our understanding of plasma physics. With the advent of increasing computational power, these simulations can now tackle larger and more complex problems than ever before. However, as the size of these simulations grows, so too does the volume of data they generate, presenting new challenges in data analysis and interpretation. Scientific Machine learning (SciML) is emerging as a powerful solution to these challenges, offering new ways to harness and analyze the vast datasets produced by fully kinetic simulations. Beyond data analysis, the computational underpinnings of SciML also present new opportunities to innovate in the way we tackle computational problems in plasma physics.

In this talk, I will explore some of the avenues my group is pursuing at the intersection of kinetic simulations and SciML. First, I will discuss how SciML can be employed to derive accurate yet simplified dynamical models of plasmas directly from the data of first-principles particle-in-cell simulations. These approaches hold the potential to create new theoretical frameworks and computationally efficient simulation models of plasmas. Additionally, I will discuss how computational methods from SciML, such as physics-informed neural networks, can be applied to solve complex inverse problems in plasma physics, including the full state reconstruction of plasmas from partial measurements/observations. Here, first-principles kinetic simulations serve as numerical experiments, providing the data necessary to understand both the potential and limitations of these innovative computational techniques, and to guide their development for plasma physics.

15:10-15:30 [Th2.C.01] Iterated Vs One-Step Optimized System Science Models for Dst Index with Neural Networks

Maria Jose Quezada Roco (University of Chile), Juan Alejandro Valdivia (University of Chile), Jose Rogan (University of Chile), Sylvain Blunier (European Astronaut Center, European Space Agency) and Max Ramírez (University of Chile).

Abstract

The Earth's magnetic field undergoes constant perturbations caused by the solar wind, a stream of ions and electrons originating from the Sun. Understanding the dynamics of the solar wind driven magnetosphere is crucial, particularly as our modern society becomes increasingly dependent on technology that is susceptible to these phenomena. With the approach of the peak of the current solar cycle, it becomes imperative to anticipate potential disruptions to our magnetosphere and take precautionary measures to mitigate potential damage to our technologies.

In an effort to characterize the dynamics of the solar wind-magnetosphere-ionosphere system (SWMI), numerous geomagnetic indices (GI) have been introduced. One noteworthy index, DST, has been constructed to characterize the magnetic field fluctuations in the equatorial regions at the Earth's surface, aiming to capture the effects of the ring current at ground level with an hourly temporal resolution.

In this context, we adapt a machine learning approach [1] designed specifically as a system science discovery technique to study this system. First, given that the magnetosphere is a high dimensional system driven by a turbulent solar wind, we take advantage of the complexity of the SWMI interaction to construct an ensemble of neural net models, starting from a diverse set of seeds. Second, we optimize the models in terms of the iterated error, instead of the standard one step error. In addition, we try to identify what are the Robust Solar Wind Drivers that affect the system. By combining these strategies, we are able to construct diverse system science models that intend to concentrate on the relevant dynamics and drivers of the system. It unveils interesting insights into how to achieve an enhanced adaptability across distinct stages of a storm. For example, our results indicate that iterated models show improvements when employing iterative optimization compared to the one-step approach.

Additionally, the construction of a diverse set of models that consider robust solar wind drivers suggest that this approach could be used to study the magnetosphere as a dynamically driven multi scale system with interconnected subsystems, and how to forecast their behavior. Additionally, it introduces an innovative interdisciplinary approach that provides valuable clues about the inner workings of neural networks.

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16:00-16:30 [Th3.A.11] JET Experiments in Support of JT-60SA Scenario Development

Luca Garzotti (UKAEA), F. P. Orsitto (ENEA C. R. Frascati), G. Pucella (ENEA C. R. Frascati), S. Gabriellini (Università di Roma La Sapienza), F. Auriemma (CNR ISTP, Milano), M. Baruzzo (ENEA C. R. Frascati), J. Bernardo (CNR IST, Lisboa), A. Burckhart (IPP Garching), C. Haliis (UKAEA), R. Dumont (CEA Cadarache), N. Hawkes (UKAEA), D. Kelling (UKAEA), D. King (UKAEA), J. Mailloux (UKAEA), A. Patel (UKAEA), C. Piron (ENEA C. R. Frascati), C. Sozzi (CNR IST, Lisboa), V. K. Zotta (Università di Roma La Sapienza), JET Contributors (See author list of C. F. Maggi et al. 2024 Nucl. Fusion) and the EUROfusion Tokamak Exploitation Team (See author list of E. H. Joffrin et al. 2024 Nucl. Fusion).

Abstract

The integrated commissioning of JT-60SA tokamak was successfully completed in 2023 and the initial research phase of the machine is foreseen to start in 2026. The different operational scenarios envisaged for the extended research phase include a high current (5.5 MA / 2.25 T) inductive scenario, an advanced inductive scenario (3.5 MA / 2.28 T) and a fully non-inductive, high β_N scenario for advanced tokamak operation (2.3 MA / 1.72 T) and they will be developed progressively starting from lower plasma currents.

In support of the scenario development activity to be carried out on JT-60SA, and based on previous exploration of the hybrid and advanced tokamak scenarios, JET has performed experiments aiming at establishing a scenario with dimensionless parameters between the JT-60SA advanced inductive and fully non-inductive scenarios.

In particular, deuterium plasmas were realized at toroidal magnetic field $B_T = 1.7, 2.0$ and 2.4 T, plasma current $I_p = 1.4$ MA, elongation $k = 1.6$, triangularity $\delta \approx 0.4$, $q_{95} = 3.5-4.5$, and q_0 at the start of the main heating phase > 1.2 , with NBI power $P_{NBI} = 16-25$ MW. A small subset of the deuterium plasmas obtained was replicated also in deuterium and tritium during the DTE3 experimental campaign to explore possible isotope effects.

Emphasis was placed to obtain plasmas with the highest possible β_N , while maintaining a mild MHD activity not to dramatically affect the confinement and guarantee the possibility of studying these plasmas from the transport point of view.

Main characteristics of the scenario developed on JET were: good confinement properties and relatively high β_N values for input NBI power of 20 MW ($\beta_N > 3.5$ for 1.7 T / 1.4 MA); good control of q_0 at the beginning of the main heating phase by tuning the NBI starting time $t_{0,NBI}$ depending on the toroidal magnetic field value, as investigated in JET hybrid and advanced scenarios; maximum $\beta_N \approx 2.5-2.7$ with relatively mild/stable MHD in the hybrid-advanced scenario at $B_T = 2.4$ T and $q_0 > 1$.

In this talk we review some aspects of the JT-60SA research plan, especially from the scenario development point of view, and present the main experimental results in support of the JT-60SA experimental exploitation showing that the scenarios developed on JET can approach the JT-60SA parameter space (especially in the initial operation phase) and some initial transport analysis showing that existing transport models such as Bohm/gyro-Bohm, QuaLiKiz and CDBM can reproduce reasonably well the JET experiments.

16:30-17:00 [Th3.A.12] Stochastic Theory of Plasma Bifurcations and Advanced Operations

Eun-jin Kim (Coventry University & Seoul National University).

Abstract

A key challenge in magnetically confined fusion of high-temperature plasmas is that plasmas tend to be unstable and become turbulent, causing anomalous transport and confinement degradation. However, novel plasma self-organization can emerge spontaneously, playing a vital role in plasma confinement and advanced scenarios. For instance, when a heating input power exceeds a critical power threshold, the transition from a low-confinement mode (L-mode) to a high-confinement mode (H-mode) occurs spontaneously, where plasmas organize themselves into an 'ordered', high-confinement state. While reproduced in different fusion devices, its triggering mechanisms and causality relations are not fully understood. Furthermore, turbulence characteristics in the L and H modes are very variable, often with highly time-varying RMS values of fluctuating density and turbulence velocity. On the other hand, the H-mode is subject to quasi-periodic edge-localized modes (ELMs), which can potentially cause significant damage to wall-facing materials. Despite successful experiments of ELM suppression and mitigation, e.g., by using resonant magnetic perturbations (RMPs), what is necessary for successful control is not fully understood.

To address this, I discuss a statistical analysis method [1-4] to shed light on fundamental mechanisms involved in accessing high-confinement or other advanced improved confinement states. I show the limitations of a naïve picture of bifurcation in a deterministic system based on a mean-field type theory and elucidate how plasma statistical properties change over the L-H transition. In particular, stochastic noises produce random trajectories and phase mixing, leading to uncertainty in power threshold [1] and ELM suppression [2]. I elucidate self-regulation and causal relations by information geometry [3,4] that works better than other popular entropy-based methods (e.g., transfer entropy). Some of the theoretical findings are supported from the L-H transition experimental data analysis [5]. We discuss implications for understanding other advanced operation scenarios.

Acknowledgments This research is supported by Brain Pool Program funded by the Ministry of Science and ICT through the National Research Foundation of Korea (RS-2023-00284119) and EPSRC grants (EP/W036770/1, EP/R014604/1).

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17:00-17:30 [Th3.A.I3] Most Probable Ignition Approach for Magnetic Fusion Plasmas

Hyeon Park (UNIST).

Abstract

New aspects of fusion plasma physics such as role of magnetic configuration for edge confinement and heating effects in core confinement will be a basis for the test of ignition in magnetic fusion. Performance data ($n\tau_E T_i$) and confinement scaling (τ_E) in the past half a century is discussed through critical analysis. These new findings are used to project the most probable ignition test device (tokamak device with $V_p \sim 200\text{m}^3$) with the presently available engineering to demonstrate sustainment of $T_i \gg 10\text{keV}$ fusion plasma through sufficient α -particle heating.

16:00-16:30 [Th3.B.I1] Generation of Unstable Plasmas Leveraging Laser-Matter Interactions in Unmagnetized and Magnetized Regimes

Thales Silva (Instituto Superior Técnico).

Abstract

The study of plasma instabilities is essential for understanding various astrophysical phenomena and advancing laboratory astrophysics. This presentation focuses on recent advancements in using lasers to generate unstable plasma conditions, both in non-magnetized and magnetized scenarios, leveraging anisotropic heating in laser-plasma interactions and the generation of ring-shaped momentum distribution functions during laser-ionization of a neutral gas.

We first delve into the generation of anisotropic momentum distributions due to stimulated Raman scattering (SRS) in laser-plasma interactions [1]. We use a combination of theoretical predictions and particle-in-cell (PIC) simulations to explore regimes in which SRS will cause strong anisotropies due to trapping and wave breaking of the small wavelength plasma waves resulting from the SRS near back-scattered modes. This anisotropy is a source for the Weibel instability [2]. We show how this configuration may help to better understand the long-time evolution of the generated magnetic field filaments.

The second part is dedicated to the electron cyclotron maser (ECM) instability in laser-ionized magnetized plasmas [3]. Recent results have shown that laser ionization can also generate plasmas unstable to the Weibel instability [4]. Here, we extend this approach by using circularly polarized lasers to ionize plasmas in the presence of a guiding field, creating long-lived ring-shaped momentum distributions. This configuration allows the growth of the ECM in a controlled laboratory environment, simulating conditions found in space and astrophysical plasmas. This is a very timely topic, as ring-shaped momentum distributions have been recently discovered to be a general feature of the Vlasov dynamics in regimes where radiation cooling is relevant [5-6].

By bridging these two studies, we aim to provide a comprehensive overview of our recent work in the generation and study of unstable plasmas.

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16:30-16:50 [Th3.B.O1] Time-Resolved Two-Dimensional Velocity Mapping of the Hot-Dense Relativistic-Laser-Produced Plasmas

Amit Dattatraya Lad (Tata Institute of Fundamental Research), Kamalesh Jana (Tata Institute of Fundamental Research), Sagar Dam (Tata Institute of Fundamental Research), S. K. Rakeeb (Tata Institute of Fundamental Research), Yash Ved (Tata Institute of Fundamental Research), Ankit Dulat (Tata Institute of Fundamental Research), C. Aparajit (Tata Institute of Fundamental Research), Anandam Choudhary (Tata Institute of Fundamental Research), Alex Robinson (Rutherford-Appleton Laboratory), John Pasley (University of York) and G. Ravindra Kumar (Tata Institute of Fundamental Research).

Abstract

Ultra-intense, femtosecond laser pulses can produce hot, dense plasma and thereby generate intense shock waves. In the present study, we use extreme contrast pulses exhibiting an interesting consequence as the cold target is nanometric close to the probe critical surface. The cold target explodes under the influence of the intense pump pulse, driving a strong shock outward into the plasma, where it is witnessed by changes in the probe reflectivity and Doppler shift [1-3].

A detailed understanding of the critical surface motion of high intensity laser produced plasma is a very crucial parameter for understanding the interaction [1]. Experimentally resolving the ultrafast dynamics of high intensity laser driven plasma at both the relevant length scales and timescales simultaneously is challenging mainly due to the lack of diagnostic approach. Here, we present a novel technique based on pump-probe Doppler spectrometry to map spatially and temporally the ultrafast dynamics of hot-dense plasma generated by femtosecond, relativistic laser pulses [2,3]. Our technique offers hundreds of femtoseconds time resolution simultaneously with a few micrometers spatial resolution across the transverse length of the plasma. The experiment was carried out using the TIFR150 TW laser system. The extreme contrast laser pulses are generated by converting the main laser pulses to second harmonic (400 nm) with peak intensity of [4]. A normally incident time-delayed probe pulse reflected from its critical layer experiences a change in its wavelength due to the motion of the critical layer. Measuring the time dependent Doppler shifts at different locations across the probe pulse, we obtain two-dimensional velocity maps of the probe-critical plasma layer at ultrafast timescales [Fig. 1]. The time and spatial resolution offered by the proposed technique could be improved using a short duration probe pulse and increasing number of detection channels respectively. Early time measurements using this technique provide very important information about shockwave generation and propagation in dense medium [2,3].

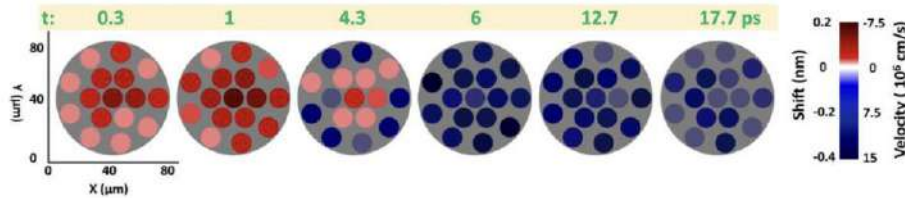


Figure 1: Spatially resolved Doppler shifts and corresponding velocity maps.

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16:50-17:10 [Th3.B.O2] Energy Absorption by Plasma Ions Under Inhomogeneous External Magnetic Field

Rohit Juneja (Indian Institute of Technology Delhi) and Amita Das (Indian Institute of Technology Delhi).

Abstract

Particle-In-Cell (PIC) simulations using the OSIRIS4.0 platform have been carried out to study the interaction of the laser pulse with an overdense plasma threaded by an inhomogeneous external magnetic field. The external magnetic field orientation is chosen to be along the laser magnetic field. The strength of the magnetic field at the plasma edge is such that the EM wave frequency lies inside the pass band, where the group velocity has a significant value. It enables the wave to enter the bulk plasma. The external magnetic field is then spatially tailored appropriately to have the LH resonance at a desired spatial location inside the plasma. This study demonstrates that the EM wave pulse comes to a standstill at the location of the resonance. The wave pulse is observed to break down subsequently, and the energy consequently goes dominantly to the local plasma ions. The absorption is significantly enhanced compared to the case in which the magnetic field profile was homogeneous [1, 2]. The dependence of absorption on the choice of magnetic field profile, the laser intensity, etc., will also be presented.

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16:00-16:30 [Th3.C.I1] 30 Years of Plasma Crystal Research

Hubertus M Thomas (DLR-Institute of Materials Physics in Space).

Abstract

In 1994 the research field of dusty plasmas changed fundamentally due to the discovery of the crystallization of dusty plasmas in the laboratory [1-3]. Before, dusty plasmas were mainly discussed theoretically concerning the interaction of dust particles with plasma in space, including interstellar clouds, circumstellar and protoplanetary accretion discs, nova ejecta, and planetary magnetospheres. In the late 80ies and early 90ies of the last century laboratory research in low-temperature plasmas started since dust particle growth became one of the leading problems in plasma processing industry. With the discovery of the so-called plasma crystals a new research field in classical condensed matter physics was born – a topic merging plasma and solid-state physics. One of the main properties of this new state of matter is the investigation of fluid and solid processes on the most fundamental – the kinetic – level due to the individual observation of the dust particles forming liquid and crystalline structures.

Gravity strongly affects micron-sized dust particles and leads to their sedimentation. In Earth-bound labs 2-dimensional plasma crystals can be formed and investigated by e.g. levitating the charged dust particles in the sheath electric field. For the formation of large 3-dimensional systems microgravity experiments are necessary and have been performed in parabolic flights, sounding rockets and onboard the International Space Station ISS. The latter generated a longstanding program on the ISS over more than 20 years up to now. In this presentation I will review the research over the last 30 years by showing some of the highlight topics of plasma crystal research.

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16:30-16:50 [Th3.C.01] Galactic Cosmic Rays Driven MHD Waves and Gravitational Instability Dusty Molecular Clouds [online]

Ram Prasad Prajapati (Jawaharlal Nehru University New Delhi).

Abstract

In this work, the impact of galactic cosmic rays (CRs) in terms of CR pressure and parallel CR diffusion is studied on the low-frequency magnetohydrodynamic (MHD) waves and linear gravitational instability in the typical dusty plasma environment of molecular clouds (MCs). The dusty fluid model is formulated by combining the equations of the magnetized electrons/ions and dust particles, including the CR effects. The interactions between CR fluid and gravitating magnetized dusty plasma have been studied with the help of modified dispersion properties of the MHD waves and instabilities using the hydrodynamic fluid–fluid (CR–plasma) approach. CR diffusion affects the coupling of CR pressure-driven mode with dust-Alfvén MHD mode and causes damping in the MHD waves. It persists in its effect along the direction of the magnetic field and is diminished across the magnetic field. The phase-speed diagram shows that for super-Alfvénic wave, the slow mode becomes the intermediate Alfvén mode.

The fundamental Jeans instability criterion remains unaffected due to CR effects, but in the absence of CR diffusion, the effects of dust-acoustic speed and CR pressure-driven wave speed are observed in the instability criterion. It is found that CR pressure stabilizes while CR diffusion destabilizes the growth rates of Jeans instability and significantly affects the gravitational collapse of dusty MCs. The charged dust grains play a dominant role in the sub-Alfvénic and super-Alfvénic MHD waves and the collapse of MCs, triggering gravitational instability. The consequences have been discussed to understand the gravitational instability in the dense photodissociation regions of dusty MCs [2].

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16:50-17:10. [Th3.C.O2] Sputtering and Grain-Grain Collisions in Weakly-Ionised Plasmas Using Grain Size Distribution Functions.

Sven Van Loo (Ghent University).

Abstract

Shocks in the interstellar medium occur as a result of a variety of phenomena, e.g. protostellar outflows, supernovae and cloud-cloud collisions. In dense, molecular clouds the ionisation fraction of the plasma is low and the magnetic fields threading the clouds can be significant. This results in the shocks from the bipolar outflows of young stellar objects being C-type, meaning there is a smoothing effect on the discontinuities in the fluid parameters through the shock. These shocks are important for the generation of molecules such as SiO which are otherwise heavily depleted into dust grains in these regions. The destruction of dust grains in shocks can occur due to gas-grain sputtering and grain-grain collisions, in which the grains undergo shattering and vaporisation. These processes can be modelled in a MHD code by taking different sizes of grains to be individual fluids. However, it is more realistic to incorporate the grain size distribution which is then followed through the shock. The novelty of the method presented here is that the distribution within each bin is taken into account, either using a piecewise linear or power law approach. Numerical tests show that the results closely follow the analytic solutions even for small numbers of size bins when using the power law approach. The piecewise linear method is accurate for large numbers of bins, but is inadequate in comparison to the power law method for small numbers of bins. This method can be implemented into any HD/MHD code in which changes in the dust grain size distribution occur.

1. Welcome – Mark Koepke, West Virginia University (FEDER Co-PI)
2. Data Ecosystem and Repository management – Raffi Nazikian, General Atomics (FEDER PI) [remote]
3. Building and operating the Data Ecosystem and Repository – Brian Sammulu, General Atomics (FEDER Co-PI) [remote]
4. Challenges in operating the Data Ecosystem and Repository – Frank Wuerthwein, San Diego Supercomputer Center at University of California-San Diego (FEDER facility partner) [remote]
5. Important features desired by the data community – Open question
6. Aspirational highlights, targeted metrics & foreseen impact within plasma physics – Open question
7. Question and answer opportunity

Friday
September 13

10⁴-Fold Field Amplification and Control of Relativistic Mega-Ampere Electron Beams in a Modest, Static Magnetic Solid

G. Ravindra Kumar (Tata Institute of Fundamental Research), Anandam Chaudhary (Tata Institute of Fundamental Research), Amit Dattatraya Lad (Tata Institute of Fundamental Research), Trishul Dhalia (Indian Institute of Technology Delhi), Aparajit Chandrasekaran (Tata Institute of Fundamental Research), Ankit Dulat (Tata Institute of Fundamental Research), Rohit Juneja (Indian Institute of Technology), Laxman Goswami (Indian Institute of Technology), Yash M Ved (Tata Institute of Fundamental Research) and Amita Das (Indian Institute of Technology).

Abstract

Generating a powerful and quasistatic magnetic field within the confines of a tabletop laboratory experiment has proven to be a persistent challenge. Magnetized high energy density physics through such experiments presents significant opportunities for exploring and studying several terrestrial as well as astrophysical phenomena, apart from controlling relativistic electron transport, directly relevant for fusion schemes.

Here we demonstrate that the modest magnetic field (0.1 tesla) in a common, readily available Neodymium magnet excited by an ultra-intense, femtosecond laser pulse leads to the generation and amplification of axial quasistatic magnetic field to megagauss levels lasting a few picoseconds. The experimental findings are strongly supported by particle-in-cell simulations, which not only validate the observations but also unveil a potential dynamo mechanism responsible for the enhancement and amplification of the axial magnetic field [1].

We also experimentally demonstrate that this modest (0.1 Tesla), static magnetic field guides 100s keV-MeV energy, mega-ampere electron pulses over macroscopic distances. These pulses, driven by an ultrahigh intensity, femtosecond laser, propagate like a beam a distance as large as 5 mm in a high Z target (neodymium), their collimation improved, and flux density enhanced nearly by a factor of 3 [2]. Unlike the previous studies that used 100s Tesla or kilotesla fields [3,4], our studies may prove more amenable for fast electron beam-driven radiation sources, fast ignition of laser fusion, and laboratory astrophysics.

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Suppression of Pair Beam Instabilities in a Laboratory Analogue of Blazar Pair Cascades

Gianluca Gregori (University of Oxford).

Abstract

Relativistic electron-positron plasmas are ubiquitous in extreme astrophysical environments such as black-hole and neutron-star magnetospheres, where accretion-powered jets and pulsar winds are expected to be enriched with electron-positron pairs. Their role in the dynamics of such environments is in many cases believed to be fundamental, but their behaviour differs significantly from typical electron-ion plasmas due to the matter-antimatter symmetry of the charged components. So far, our experimental inability to produce large yields of positrons in quasi-neutral beams has restricted the understanding of electron-positron pair plasmas to simple numerical and analytical studies, which are rather limited. Here we present the first experimental results confirming the generation of high-density, quasi-neutral, relativistic electron-positron pair beams using the 440 GeV/c beam at CERN's Super Proton Synchrotron (SPS) accelerator. We show that the characteristic scales necessary for collective plasma behaviour, such as the Debye length and the collisionless skin depth, are exceeded by the measured size of the produced pair beams. In the first application of this experimental platform, the stability of the pair beam is studied as it propagates through a meter-length plasma, analogous to TeV γ -ray induced pair cascades in the intergalactic medium. It has been argued that pair beam instabilities disrupt the cascade, thus accounting for the observed lack of reprocessed GeV emission from TeV blazars. If true this would remove the need for a moderate strength intergalactic magnetic field to explain the observations. We find that the pair beam instability is suppressed if the beam is not perfectly collimated or monochromatic, hence the lower limit to the intergalactic magnetic field inferred from γ -ray observations of blazars is robust.

11:40-12:00 [Fr1.A.O1] Isotopic Effect on Intrinsic Toroidal Rotation in ADITYA-U Tokamak

Ankit Kumar (Institute for Plasma Research), Joydeep Gosh (Institute for Plasma Research, Homi Bahabha National Institute), M. B. Chowdhuri (Institute for Plasma Research), Aman Gauttam (Institute for Plasma Research), N. Ramaiya (Institute for Plasma Research), Bharat Hedge (Institute for Plasma Research, Homi Bahabha National Institute), Kaushlender Singh (Institute for Plasma Research, Homi Bahabha National Institute), Suman Dolui (Institute for Plasma Research, Homi Bahabha National Institute), Ashok Kumawat (Institute for Plasma Research, Homi Bahabha National Institute), Dipexa Modi (Pandit Deendayal Petroleum University), Soumitra Banarjee (Institute for Plasma Research, Homi Bahabha National Institute), Injamul Houqe (Institute for Plasma Research, Homi Bahabha National Institute), Komal (Institute for Plasma Research, Homi Bahabha National Institute), Harshita Raj (Institute for Plasma Research, Homi Bahabha National Institute), K. A. Jadeja (Institute for Plasma Research), Ankit Patel (Institute for Plasma Research), Pramila Gautam (Institute for Plasma Research), Utsav (Institute for Plasma Research), K. Shah (Princeton Plasma Physics Laboratory), G. Shukla (Institute for Plasma Research), N. Yadava (Oak Ridge Associated Universities), Tanmay Macwan (University of California Los Angeles), S. Patel (Pandit Deendayal Petroleum University), Laxikanta Padhan (Institute for Plasma Research), Suman Aich (Institute for Plasma Research), A. Kanik (University of Petroleum and Energy Studies), Rohit Kumar (Institute for Plasma Research, Homi Bahabha National Institute), Priyanka Verma (Institute for Plasma Research), K. M. Patel (Institute for Plasma Research), Kalpesh Gadoliya (Institute for Plasma Research) and R. L. Tanna (Institute for Plasma Research).

Abstract

In both Hydrogen & Deuterium discharges, intrinsic toroidal rotation has been observed using Doppler shifts in spectral lines of the main impurity ions (C5+ ions, emitting at 529.05nm) in ADITYA-U tokamak without applying any external torque to the plasma. In purely Hydrogen discharges, intrinsic toroidal rotation reverses its direction from counter-current to co-current on increasing the plasma current beyond a certain threshold value. However, this intrinsic toroidal rotation (measured in the edge region) is observed to damp to zero as soon as the edge electron density is increased [1]. In Hydrogen discharges, $E \times B$ drift velocity is found to drive the observed intrinsic toroidal rotation in the edge region of ADITYA-U. A recent and a new study of the isotopic effect is performed on the intrinsic toroidal rotation in ADITYA-U tokamak, which will be discussed in this paper.

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11:40-12:10 [Fr1.B.I1] Laser Driven Microdroplet for Efficient Electron Acceleration

Krishnamurthy Manchikanti (TIFR Hyderabad).

Abstract

Intense ultrashort pulse lasers generate relativistic electrons when the intensity reaches relativistic scales, 10^{18}Wcm^{-2} for 800nm pulses. This requires Terra watt class lasers that are complex, cumbersome, expensive and deliver typically 10 pulses per second. While the electron/x-rays/proton beams generated from such system have shown a lot of promise, developing applications on such systems is very challenging. I will talk about experiments where even at a 1/100th of laser intensity, it is feasible to generate relativistic electron beam of 1 MeV energy with multi kHz few mJ/pulse lasers. We show that plasma wave instabilities generated and manipulated with suitable targetry is the underlying mechanism. The source size of the short pulse electron beam is amenable for x-ray radiography and shadowgraphy.

12:10-12:30 [Fr1.B.O1] Single-Shot, Spatio-Temporal Metrology of Relativistic Plasma Optics

Ankit Dulat (Tata Institute of Fundamental Research), **Amit Lad** (Tata Institute of Fundamental Research), **C. Aparajit** (Tata Institute of Fundamental Research), **Anandam Choudhary** (Tata Institute of Fundamental Research), **Yash Ved** (Tata Institute of Fundamental Research), **Laszlo Veisz** (Umea University) and **G. Ravindra Kumar** (Tata Institute of Fundamental Research).

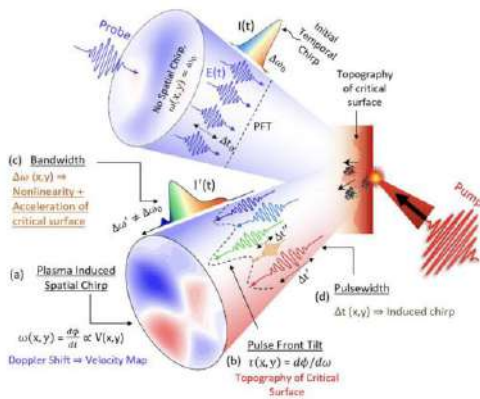
Abstract

Plasma optics are promising new tools to guide, shape, or even amplify ultrahigh-power, ultrashort laser pulses [1]. They have the unique potential to liberate ultrafast laser technology from the strong limitations posed by optical damage in conventional optical elements and so further increase the peak power of state-of-the-art lasers. However, the plasma is inherently dynamic, highly complex, and challenging to control, so it can easily degrade the spatio-temporal structure of the optical pulse. Therefore, to design and operate plasma optics, a precise characterization of the complex spatio-temporal and spatio-spectral profile of the laser pulse and the instantaneous dynamics of the plasma surface is highly desired. Furthermore, as ultra high-power laser systems have a low repetition rate down to single-shot operation, this spatio-temporal characterization is mandatory on a single-shot basis. This was not demonstrated before, so it represents a serious gap.

Here, we present three-dimensional (3D) spatio-temporal measurements of such pulses based on spectral interferometry [2,3]. We measure the complex space-time distortions induced in the laser pulses by relativistic plasma while simultaneously capturing the underlying plasma dynamics, all in a single shot. This all-optical technique can capture 3D spatio-temporal couplings within pulses at ultra-high peak powers, enabling further progress in ultra-high-intensity laser and plasma technologies.

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11:40-12:00 [Fr1.C.O1] Inverse Cascade Processes in the Development of Electrojet Plasma Instabilities in Ionospheric E Region

Arash Tavassoli (Department of Physics and Engineering Physics, University of Saskatchewan), Andrei Smolyakov (Department of Physics and Engineering Physics, University of Saskatchewan), Sasha Koustov (Department of Physics and Engineering Physics, University of Saskatchewan) and Raymond Spiteri (Department of Computer Science, University of Saskatchewan).

Abstract

In this work, we use two-fluid simulations to investigate the nonlinear regime of the electrojet plasma instabilities responsible for the formation of electron density irregularities in the ionospheric E region [1]. We employ a 2-D model of partially ionized plasma, originally developed in [2] for studying $E \times B$ plasma discharges. The model unifies closely related plasma instabilities, such as the Simon-Hoh/gradient-drift, Farley-Buneman and lower-hybrid instabilities, and captures multiscale effects, including electron inertia and finite-Larmor-radius effects. We report on the development of secondary nonlinear instabilities and formation of large-scale plasma structures (the inverse cascade processes) leading to the nonlinear saturated state. We demonstrate that these large-scale structures play a crucial role in the development of the electrojet plasma turbulence. A comparison of computational results with experimental data is provided.

Acknowledgments This work is partially supported by funding from the Natural Sciences and Engineering Council of Canada (NSERC), University of Saskatchewan Research grant 421980 to AVK, computational resources from the Digital Research Alliance of Canada, and Simons Collaboration on Hidden Symmetries and Fusion Energy.

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12:00-12:20 [Fr1.C.O2] Emergence of Electrostatic Solitary Waves in Titan's Ionospheric Plasma

Steffy Sara Varghese (Khalifa University of Science and Technology), Kuldeep Singh (Khalifa University of Science and Technology), Frank Verheest (Ghent University) and Ioannis Kourakis (Khalifa University of Science and Technology).

Abstract

Observations conducted in Titan's ionosphere have unveiled the presence of various ions possessing either positive or negative charge [1] along with different blends of electron populations (at different altitudes) practically following a Maxwell-Boltzmann distribution[2]. From a fundamental (theoretical) point of view, it is well established that the coexistence of ions with opposite charges leads to novel phenomena, notably including the possibility of polarity reversal in localized modes, such as electrostatic solitary waves.

In this work, we shall thoroughly investigate the potential occurrence of ion-acoustic solitary waves in the ionospheric plasma of Titan. A bi-ion plasma-fluid model with two electron populations (with different temperatures) in the background has been adopted as a starting point to explore the nonlinear propagation features of ionic scale electrostatic solitary waves that may occur in Titan's ionosphere from first principles. We have employed nonlinear (multiscale) perturbation techniques, in an effort to reduce the fluid model to a nonlinear evolution equation governing the electrostatic potential. Considering different plasma compositions, including critical configurations where the nature of nonlinearity is known to change [3], different partial-differential equations have been obtained, incorporating quadratic or cubic nonlinearity as well as combinations thereof, thus describing diverse scenarios for the solitary wave propagation characteristics. The parametric influence of the plasma composition, in terms of the relative electron concentration (cool-to-hot electron density) and the relative (negative-to-positive) ion density, on the solitary wave features has been investigated. Different possibilities have been predicted, including positive and negative polarity pulses and double layers, whose characteristics (amplitude, width, polarity) depend on the plasma configuration.

Our theoretical findings should provide valuable insight into the criteria for existence and the dynamics of electrostatic solitary wave structures occurring in Titan's ionosphere, with an ambition to contribute to the prediction and interpretation of future observations.

Acknowledgments The authors acknowledge financial support from Khalifa University's Space and Planetary Science Center under grant No. KU-SPSC-8474000336. IK also thanks KU for support via grant CIRA-2021-064/8474000412.

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Poster sessions

[Tu.Po.1] A Simulation Study of Impurity Radiation Excitation and Driven Tearing Mode

Zhiwei Ma (Zhejiang University) and Yiming Zu (Zhejiang University).

Abstract

Tearing modes excited and driven by impurity radiation are investigated by using three-dimensional toroidal MHD code with the impurity module (CLT). Various types of impurities applied at the boundary under an equilibrium condition where the tearing mode is stable. It is observed that impurity radiation-induced plasma contraction results in a current gradient that excites the tearing mode at the resonance surface. Through a scan of the initial atomic number (Z) and impurity concentrations, it is found that impurities with different Z values exhibit similar behaviors in the radiation-driven tearing mode. The impurity radiation can drive tearing mode growth through temperature cooling near the resonance surface, and there exists a linear relationship between the temperature perturbation caused by impurity radiation and the linear growth rate of the tearing mode. Additionally, the impurity can promote the growth of magnetic islands through the cooling inside the magnetic island, and there exists a correlation between the initial parameters of impurity and the width of the saturated magnetic island.

[Tu.Po.2] GKNET-X, a Global Gyrokinetic Code for Turbulence Simulations in Core and Edge Regions of Tokamak Plasmas

Shuhei Okuda (Kyoto University), Kenji Imadera (Kyoto University), Haruki Seto (National Institutes for Quantum Science and Technology) and Akihiro Ishizawa (Kyoto University).

Abstract

Turbulent fluctuations play an important role in predicting the confinement of plasmas at the edge region as well as the core region. Namely, understanding the fluctuations in the outer core region connecting to the SOL/divertor region is important for predicting fuel supply, impurity pumping, divertor heat load control, and L-H transition of tokamak plasmas. These issues are particularly important for predicting the confinement of future fusion devices like JA-DEMO [1]. Although gyrokinetic simulation is an essential tool for studying these issues based on the first principles of magnetized plasmas, it is still challenging to apply it to the edge region due to high q values and complex magnetic surface geometries that are not present in the inner core region. To address these issues, a core global gyrokinetic simulation code GKNET [2] has been extended to GKNET-FAC [3] by introducing a field-aligned coordinate system [4] with a shifted metric technique [5] and numerical MHD equilibria. The field-aligned coordinate can reduce computational costs for turbulence in the outer core region significantly. For calculating drift-wave instabilities with a high toroidal mode number in a JT-60SA plasma, GKNET-FAC requires only 1/94 smaller the number of grid points than that for GKNET.

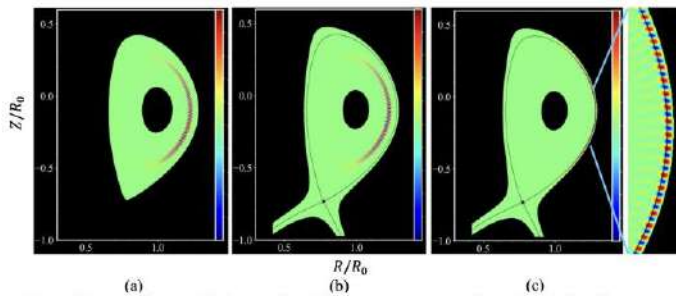


Figure. Electrostatic potential $\phi_{n=60}$ of the ITG mode in the core region calculated by (a) GKNET-FAC and by (b) GKNET-X. (c) $\phi_{n=60}$ of the ITG mode at the separatrix by GKNET-X.

In this study, we have extended GKNET-FAC to GKNET-X handling plasma turbulence in the edge region consisting of both the inside and outside of separatrix. Figures(a) and (b) show that the eigenfunction of the ion temperature gradient (ITG) mode calculated by GKNET-X is in good agreement with that by GKNET-FAC for a toroidal mode number $n=60$. In addition, we successfully calculate the ITG mode excited at the separatrix between the closed surface core region and the open surface SOL region as shown in Fig. (c). We will also present some nonlinear simulation results by GKNET-X in the conference.

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[Tu.Po.3] Estimation of the Optical Reflection Property of Plasma Facing Surfaces from Topography Measurements

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Abstract

In the spectroscopic measurement of fusion reactors, the reflected light from the plasma-facing surface has a significant effect on the measurement signal [1]. Therefore, to obtain accurate spectroscopic measurements, it is necessary to quantitatively assess the influence of the reflected light, which can be done by ray tracing calculations [2]. The accuracy of this assessment depends on the precision of the Bidirectional Reflectance Distribution Function (BRDF) of the reflecting surface used in the ray tracing calculations. Since the interaction between the plasma and the wall can change the surface condition, the BRDF of the plasma-facing surface will also change. Therefore, without the development of a method to measure this BRDF in situ, reliable spectroscopic measurements cannot be achieved, potentially leading to significant misunderstandings in spectroscopic diagnostics.

The aim of this study is to investigate the relationship between the surface geometry of plasma-facing materials and optical reflection models, and to clarify the possibility of in-situ estimation of the BRDF based on the surface topography. Among the plasma-facing components of ITER, the divertor surface will be made of tungsten, which may vary due to plasma-wall interactions, while the blanket surface will be made of beryllium, which has a large surface area and raises concerns about its substantial impact on optical diagnostics. Therefore, this study has conducted an analysis of BRDF for both tungsten and beryllium with various topographies.

Fitting was performed to optimize the free parameters in the optical reflection model. The topographic parameters were determined from the surface three-dimensional measured with a laser microscope. The relationship between the parameter (k_d) defining the diffuse reflection in the optical reflection model (Phong model), and the topographic parameters showed that k_d increased monotonically with increasing topographic parameters. In addition, topographic parameters using the slope of the surface geometry showed a higher coefficient of determination (R^2). Furthermore, the relationship between the parameter (k_s) defining the magnitude of the specular reflection in the Phong model and the topographic parameters did not show as strong a correlation as k_d . This is because the specular reflectance model does not accurately reproduce the measured BRDF, especially at the feet of the specular lobe where the BRDF is often underestimated, suggesting the need to investigate more accurate specular reflectance models. However, the topographic parameters that account for surface slope showed a certain correlation with k_s . Therefore, this indicates the potential to directly assess BRDF from measurements of surface geometry inside fusion reactors using tools such as laser interferometers, and to accurately assess the effect of reflected light in spectroscopic measurements.

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[Tu.Po.4] 2D-Emission Intensity Measurement of Hydrogen/Deuterium Plasma Detachment with ICR Heating on TPDsheet-ICR

Naonori Okada (Tokai University), Hiroki Natsume (Tokai University), Akira Tonegawa (Tokai University), Kohnosuke Sato (Tokai University) and Kunio Okimura (Tokai University).

Abstract

It is expected that the heat flux reaching the divertor region of the future reactor such as DEMO will be several times higher than that of ITER. To sufficiently reduce this heat flux, a fully detached plasma must be maintained while preventing X-point MARFE. In large fusion devices, conducting fundamental research focused on detailed divertor plasma physics is challenging due to port limitations and machine time, and other factors. Therefore, it is efficient to use a small linear divertor simulator that can generate steady-state detached plasmas and can be flexibly connected to various measurement devices.

A common issue with the general linear divertor simulator is that the ion temperature of the generated plasma is as low as several eV, which is one order of magnitude lower than that of fusion divertor plasmas. The ion temperature of fusion divertor plasmas is several tens of eV, which is close to or higher than the electron temperature (~ 10 eV). Therefore, it is required to increase the ion temperature from several eV to about 20 eV to study reaction processes in detached plasmas.

In this study, to investigate the transition process of detached plasma in detail, high-density sheet plasma ($\sim 10^{18}$ - 10^{19} m⁻³) was generated using the linear divertor simulator TPD sheet-ICR [1,2]. Hydrogen or deuterium was used as the discharge gas. Additionally, ions in the sheet plasma were heated by ion cyclotron resonance heating (ICRH) using parallel-plate electrodes. We performed the Langmuir probe measurement of plasma parameters (electron density and temperature) of the detached plasma outflowing into the divergent magnetic field region. Furthermore, two-dimensional distribution measurements of Balmer series emission (H α , D α , Fulcher, H γ and D γ) were performed using a fast-framing camera equipped with an Arbaa prism. When both hydrogen and deuterium plasmas transition from the attached plasma state to the detached plasma state, the Fulcher band emission intensity decreases, while the H γ and D γ emission intensity increases. Further, it is evidenced that the increase in the ion temperature by increasing the applied power of ICRH from 0 to 500 W (Ti: 2.4 to 6.5 eV) leads to rise in the electron temperature, resulting in a decrease in the emission from highly excited atoms such as H γ and D γ in the Balmer series.

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[Tu.Po.5] Optimisation of EBW Power Deposition and Current Drive in Spherical Tokamaks by Numerical Simulation and Resonance Condition Analysis

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Abstract

Auxiliary plasma heating and current drive (HCD) in the Spherical Tokamak for Energy Production (STEP) fusion reactor will be achieved by utilising a large number of high power microwave sources with a power of the order 1MW each. A combination of electron-cyclotron (EC) and electron Bernstein wave (EBW) HCD will be employed. In addition to the well-understood EC HCD method, the use of EBWs is considered due to their high efficiency current drive and ability to propagate in overdense plasmas. The proposed method to generate EBWs is by launching an O mode polarised microwave from the low field side at an optimal angle to the magnetic field. The O mode undergoes conversion to a slow X mode wave and finally to an EBW that can propagate into the overdense plasma interior. To obtain the most efficient power deposition and current drive, controllable access and power deposition of EBWs to all regions of the ST plasma is required. By employing GENRAY ray-tracing and CQL3D Fokker-Planck simulations, varying of the launch position and scaling the plasma parameters for a set of STEP Prototype Reactor (SPR) scenarios, we are able to optimise the propagation of the EBW and the locations of power deposition to achieve high levels of current drive. Analytical models have concurrently been developed through consideration of energy conservation and the fundamental resonance condition which give insight into the wave-plasma interaction. We also show how coupling of EBWs to Doppler-shifted electron cyclotron harmonics can impact the current drive through the excitation of oppositely directed currents.

[Tu.Po.6] Development of a Linear Divertor Plasma Simulator Based on Magnetic Mirror Device at KAIST

Donggeun Oh (Korea Advanced Institute of Science and Technology), Gwangwoon Baek (Korea Advanced Institute of Science and Technology), Dongha Kim (Korea Advanced Institute of Science and Technology), Yeono Jung (Korea Advanced Institute of Science and Technology), Kyu-Dong Lee (Korea Institute of Fusion Energy), Hyunyeong Lee (Korea Institute of Fusion Energy), Bong-Ki Jung (Q-BEAM SOLUTION), Kyoung-Jae Chung (Seoul National University) and Choongki Sung (Korea Advanced Institute of Science and Technology).

Abstract

It is crucial to understand physics of the edge region in fusion plasmas, since reducing heat flux at the divertor plate and reaching the thermo-mechanical engineering limit of the plasma facing materials are essential for the fusion energy development. Leveraging low construction and operation costs and simple geometry, linear devices have been utilized as a divertor simulator to investigate the edge plasma physics in closed magnetic field systems, including tokamaks [1–2]. This presentation will highlight the ongoing progress in developing a divertor plasma simulator using the magnetic mirror device at KAIST, KAIMIR [3]. The vacuum chamber of the device comprises three sub-chambers: source, center, and expander. We have modified the expander chamber as a divertor region of the tokamak to simulate the radiative divertor conditions and analyze their characteristics. To control the neutral pressure of the divertor chamber independently, we implemented a differential pumping system, allowing for a pressure difference of over 20 times between source and divertor chambers through the use of a skimmer structure. The gas injection valve, originally a piezoelectric valve (with a flow rate < 0.53 slm), has been replaced with a solenoid valve (with a flow rate ~ 17.8 slm) to enhance neutral pressure control capability at the source chamber. We also developed additional diagnostics, such as probes and a mm-wave interferometer system to understand the physics relevant to the divertor region, complementing existing diagnostics (spectroscopy, diamagnetic loops, and probes) in the center chamber. Future plans involve the integration of an Electron Bernstein wave (EBW) heating system to regulate the temperature of the incoming plasma flow to the divertor region and mitigate plasma pressure degradation along the axis. Further details on the experimental setup and results will be discussed.

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[Tu.Po.7] Innovative Magnetic Confinement Method for Fusion Plasmas Based on an Overlooked Historical Discovery

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Abstract

In the late 1960s, the American physicists Eugene Parker and Ian Lerche studied a simplified model of the flow of the solar wind around Earth's magnetic field, focusing on the region where the flow is parallel to the field lines (shown in red in the Figure 1). They discovered that, in the thin boundary layer between the plasma and the field, a secondary magnetic field arises, which is stronger than the primary field. Under certain circumstances, this effect causes the boundary layer to become unstable [1].

A decade later, Owen Storey and Laurent Cairó demonstrated that this effect, which they named "Parker's Effect", also occurs when a magnetic field encloses and confines a supersonically flowing plasma. However, in this scenario, it reinforces the confinement of the plasma which remains, in the bulk, field-free and current-free. They demonstrated analytically that a simple confinement system based on this effect would be intrinsically MHD-stable and have a global beta significantly higher than most magnetic confinement devices currently under investigation. They suggested that Parker's Effect may have applications to fusion and outlined a toroidal device exploiting it [2]. To date, Parker's Effect has not been observed experimentally or in simulation, mainly because it has not been sought.

Our research group has been reviewing these investigations and developing a full simulation model of a module of the device (figure 2), using an open-source software framework running on modern supercomputers. In this presentation, we will explain Parker's discovery, the insights contributed by Storey and Cairó, and the main device they proposed. We will mention our team's recent research efforts, share preliminary results and findings, and highlight potential avenues for collaboration.

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[Tu.Po.8] Potential Research Programme for JET with a Tungsten Wall and ECRH

Jozef Ongena (LPP-ERM/KMS Brussels), Emilia Solano (Laboratorio Nacional de Fusion, CIEMAT, Madrid) and JET petition contributors (Petition to extend JET beyond 2023).

Writing as independent, not necessarily representing the views of our institutions

Abstract

We propose a research program for the continued operation of the JET facilities over the next 10-15 years, thereby making optimal use of JET's unique capabilities (especially operation with Deuterium-Tritium mixtures) by integrating the ITER re-baselining decisions within JET. The primary objective is to transition JET to a phase with a tungsten wall and 10 MW of Electron Cyclotron Resonance Heating (ECRH) using 170GHz gyrotrons in addition to the existing additional heating systems, thereby aligning with the goals of both STEP and ITER.

Enhancing JET as proposed, would allow to increase the fusion performance for Baseline and Hybrid scenarios, and to test their compatibility with a Tungsten wall, aiming at steady conditions with equal electron and ion temperatures. Synergies between Electron Cyclotron (EC), Ion Cyclotron (IC), Neutral Beam Injection (NBI), and alpha heating could be investigated in detail. Investigations in the ion temperature clamping effect seen on smaller machines with dominant electron heating (and methods to avoid it) would now become possible in a device with a size and under operational conditions as close as possible to ITER. The compatibility of Boronization with high performance plasmas in the presence of a high-Z wall can be investigated in detail. Control, mitigation and suppression techniques for Edge Localized Modes (ELMs) could be tested in more fusion-relevant situations, and the effect of controlled impurity seeding with Ar and Ne on plasma wall interaction and confinement could be explored. DT campaigns in the enhanced JET would also uniquely enable to develop advanced fusion diagnostics, including precise neutron measurements and γ -ray imaging to detect alpha-particles (from D+T or B⁴He reactions), providing a unique opportunity to prepare necessary diagnostics for the rebaselined ITER. Preparing for ITER's DT campaigns (presently planned to begin in 2039) implies training the next generation of scientists, technicians, and engineers within a large fusion facility. Extending JET's operation as proposed offers a unique opportunity to sustain and enhance the expertise of fusion researchers globally, thereby accelerating progress in the world-wide quest for fusion energy.

[Tu.Po.9] Terahertz to near-Infrared Radiation Generation Using Two-Color Laser Pulses in Plasma

Dinkar Mishra (University of Lucknow) and Bhupesh Kumar (University of Lucknow).

Abstract

The generation of intense and coherent THz radiation has been a subject of intense research, with numerous methods developed to harness its potential. One of the promising techniques that has emerged is the generation of THz radiation using laser-induced plasma. This method utilizes the unique properties of ultrafast lasers to create plasma, which, in turn, emits THz radiation through a variety of mechanisms [1-5]. This study is significant for the generation of intense radiation fields having frequencies lying in the THz to near IR range. The novelty of the present mechanism is that the frequency of the generated radiation can be controlled by continuously varying the frequency difference between the two laser pulses. In order to portray the significance of the present mechanism laser frequency differences equal to twice and thrice the plasma frequency have been considered. However, arbitrary frequency differences can lead to a continuous range of tunable radiation. The present analytical mechanism of tunable radiation generation has been validated via simulation studies which show that the transverse orthogonal electric and magnetic fields generated in plasma are essentially radiation fields that can propagate in vacuum. The discrepancy in analytical and simulation results may be attributed to the approximation scheme used for the analytical case.

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[Tu.Po.10] Exploring the Possibility of Evaporating Macroparticles of Vacuum Arcs by an Electron Beam

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Martin Rudolph (Leibniz Institute of Surface Engineering (IOM)) and André Anders (Leibniz Institute of Surface Engineering (IOM)).

Abstract

Erosive plasmas sources creating dense, low-temperature plasma, in particular of the vacuum-arc type, are reliable and well-tested generators of metal plasma. They are widely used in academia and industry for the deposition of hard, protective and functional coatings. Moreover, they are used to modify surface properties of structural and decorative materials. However, the presence of microdroplets of the cathode material in the plasma prevents the use of erosive sources in applications requiring high-quality coatings, especially those demanding high uniformity and low roughness at the nanometer level. The use of modern filtering techniques to remove droplets, especially those of small sizes ($< 1 \mu\text{m}$), leads to a significant decrease in the deposition rate, which compromises one of the unique features of these sources, namely their high deposition rate. That is why alternative filtering concepts are of interest.

Here, we discuss an approach using energetic electrons injected into the arc plasma [1]. They introduce additional energy into the plasma which can promote evaporation from the droplets which may eventually lead to complete destruction of droplets. By solving a self-consistent system of equations for the balance of energy, current to and from a droplet, and the droplet mass, it is shown that favorable conditions for the evaporation of microdroplets may exist. It is shown that the main parameters influencing the droplet evaporation rate are the plasma density as well as the density and energy of the electron beam. It has been shown that droplets with a radius of $1 \mu\text{m}$ or less may completely evaporate during the time the droplet is in the system if the energy of the electron beam is 3 to 5 keV and the beam-to-plasma density ratio is greater than 0.01.

Acknowledgments This work is supported partly by the grant DFG - Deutsche Forschungsgemeinschaft (DFG) under project number 525228371.

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[Tu.Po.11] Factors Influencing Inactivation Efficacy of Plasma-Activated Water Against *Klebsiella Pneumoniae*

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Abstract

Plasma-activated water (PAW) has demonstrated potent antimicrobial efficacy, which is mainly attributed to the reactive oxygen and nitrogen species (RONS) [1]. Our study assessed bactericidal activity of PAW against multidrug-resistant *Klebsiella pneumoniae* in relation to plasma treatment time, volume of treated water and PAW application time on bacteria samples. The model organism we selected is an important nosocomial pathogen, capable of persistence on various surfaces and difficult to eradicate due to resistance to harsh conditions and antimicrobials [2].

The in-house developed plasma source was 3-pin jet with needle electrodes inserted inside three separate glass tubes and positioned vertically above the water sample. Working gas was argon (2.5 slm), frequency of high-voltage power supply 340 kHz and applied power 14 W. We have treated 150 ml of deionized water for 20 min (PAW1, pH=6.2) and 15 ml for 10 min (PAW2, pH=6.5). Characterization of the PAW chemistry was performed by using colorimetric methods: PAW1 (H₂O₂=10 ppm, NO₃=34 ppm, NO₂=0 ppm) and PAW2 (H₂O₂=80 ppm, NO₃=97 ppm, NO₂=41 ppm).

An aliquot of 100 µL of the suspension (10⁸ cells/ml, *K. pneumoniae* ATCC BAA-1705) was added to 900 µL of PAW in microtubes, homogenized, and maintained for 15 min and 60 min. Bactericidal effects of PAW were assessed by the standard plate count method and expressed in log CFU/mL. Inactivation activity of PAW against *K. pneumoniae* increased by reducing the water volume for PAW preparation and by extending the incubation time of the bacteria in PAW. PAW1 reduced *K. pneumoniae* by ≈1.1 log CFU after 60 min of application time, while no reduction was noted after 15 min exposure. PAW2 applied for 15 min resulted in ≈1.6 log CFU reduction, while 60 min application achieved complete eradication of *K. pneumoniae*.

Acknowledgments This research was supported by the Science Fund of the Republic of Serbia, 7739780, APPerTain-BIOM.

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[Tu.Po.12] Investigating Ultra-High Dose Rate Pulsed X-ray Effects on Cancer Cells Using a Kilojoule Plasma Focus Device

Jalaj Jain (Comisión Chilena de Energía Nuclear), José Moreno (Comisión Chilena de Energía Nuclear), Héctor Araya (Universidad de Chile), Rodrigo Andaur (Universidad de Chile), Sergio Davis (Chilean Nuclear Energy Commission), Leonardo Herrera (Universidad Andres Bello), Biswajit Bora (Chilean Nuclear Energy Commission), Ethel Velásquez (Chilean Nuclear Energy Commission), Cristian Pavez (Chilean Nuclear Energy Commission), Katherine Marcelain (Universidad de Chile) and Leopoldo Soto (Chilean Nuclear Energy Commission).

Abstract

Radiation therapy has been used for the treatment of cancer disease for many decades. Normally, the dose rates in radiation therapy are of the order of tens to hundreds Gy/h. The effects of ultra-high dose rate irradiations ($10^3 \sim 10^7$ Gy/sec) on cancer cells are less explored. In the present study, several cell cultures, two colorectal cancer cells (DLD-1, HCT-116), one breast cancer cell (MCF-7), one ovarian cell, and one non-cancerous colorectal cell (CCD-841-con) have been exposed to pulsed X-ray ($\sim$ ns duration time) with a high-dose-rate ($\sim 10^7$ Gy/sec) keeping the total doses low (≤ 1 Gy) using a kilojoule plasma focus device, PF-2kJ. The obtained results are compared with the conventional X-ray irradiation source results. The DLD-1 cell line shows the low-dose hyper-radio-sensitivity (LDHRS) effect at lower doses for pulsed X-ray than the conventional X-ray irradiation. The HCT-116 cell line shows the HRS effect in the case of pulsed X-ray irradiation, which is reported to be absent in the case of conventional X-ray irradiation. The radio-resistive cell MCF-7 shows a larger number of cell death in the case of pulsed X-ray irradiation. The ovarian cancer cell shows a reduced proliferation capacity in the case of 2D cell culture irradiation and reduced vasculogenic-mimic structures in the case of 3D model irradiation using pulsed X-ray. The X-ray energy used for cell culture irradiation was in the range of 8-10 keV. To study further the irradiation effects of different energies of X-ray on cells, a simulation using the Geant4 tool kit was performed. It was found that low-energy X-ray irradiation induces larger toxic effects.

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[Tu.Po.13] Methicillin-Resistant *Staphylococcus Aureus* Inactivation by Plasma Activated Water

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Abstract

Plasma-activated water (PAW) presents an effective strategy for microbial inactivation, which is linked to synergistic effects of reactive oxygen and nitrogen species (RONS) [1]. We evaluated the antimicrobial effect of PAW against a methicillin-resistant *Staphylococcus aureus* (MRSA) isolated from an odontogenic abscess. *S. aureus* strains, and in particular MRSA strains due to their resistance, have an important role in etiology of dental abscesses[2]. For the purpose of comparison, the reference MRSA strain routinely applied in disinfectant testing (ATCC 33591) was included in the study.

The plasma source for PAW preparation was 3-pin jet using argon as the working gas(2.5 slm), with frequency of high-voltage power supply 340 kHz and applied power 14 W. Needle electrodes were inserted inside three separate glass tubes and positioned vertically above the water sample. We have treated 15 ml of deionized water for 10 min (pH=6.5). The PAW chemistry was characterized by colorimetric methods (H_2O_2 =80 ppm, NO_3^- =97 ppm, NO_2^- =41 ppm).

Bacterial suspensions containing 108 cells/ml were prepared in sterile saline, and an aliquot of 100 μL per strain was added to 900 μL of PAW in microtubes. Following exposure to PAW for 15 min and 60 min, bactericidal effects were assessed by the plate count method and expressed in log CFU/mL. PAW treatment was ineffective against reference MRSA strain regardless of the exposure time. Only longer PAW treatment showed effectiveness in the clinical MRSA strain, resulting in log CFU reduction equivalent to a 81.67% reduction in the bacterial population. While these results show bactericidal capacity of PAW against MRSA, further optimization of the PAW generating parameters is required for achieving efficient eradication.

Acknowledgments This research was supported by the Science Fund of the Republic of Serbia, 7739780,APPerTain-BIOM.

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[Tu.Po.14] Hydrolysis of degradable plasma-polymerized poly(ethylene glycol)/ZnO nanocomposites in food simulants: identification of components and potential toxicity

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Abstract

Plasma polymers produced via atmospheric pressure plasma polymerization are an innovative alternative to polymers synthesized through wet chemistry. This solvent-free, eco-friendly technique creates advanced antibacterial polymer coatings suitable for food packaging. Despite advancements in plasma polymer manufacturing, less focus has been on the degradation of these coatings in contact with food. Proper identification of degradation products is crucial for assessing potential toxicity. This study uses an aerosol-assisted atmospheric pressure plasma system to deposit polyethylene glycol (PEG)-like coatings with 1 wt% zinc oxide (ZnO) nanoparticles on a polymer substrate. Fourteen degradation products released into various food simulants were identified, with the highest releases linked to $C_6H_{14}O_4$ and $C_{10}H_{22}O_5$, which differ mainly in the number of ethylene oxide groups. This highlights the efficiency of the plasma polymerization approach. Increasing the plasma input power from 200 to 350 W produced nanocomposites with higher degrees of crosslinking and a greater presence of ZnO nanoparticles (from 1.6 ± 0.3 to 5.9 ± 0.8 at. %), resulting in fewer degradation products being released. Toxicity evaluations, including *Daphnia magna* LC50 (48 hr) and oral rat LD50 tests, indicate that these substances are non-toxic, supporting the safe use of these plasma-polymerized coatings in antibacterial food packaging.

[Tu.Po.15] Comparative Study of Plasma Parameters by Cathode Size in Low Pressure DC Glow Discharge for Improving Tribological Properties

Ramnaresh Kumar (University of Delhi), Bornali Sarma (University of delhi) and D.N. Gupta (University of Delhi).

Abstract

The glow discharge is a widely-used technique for improving the tribological properties of materials, including the production of thin films using ionic beams and the treatment of surfaces through the sputtering process [1]. In this study, we concentrate on numerical analysis of the glow discharge in a cylindrical quartz chamber filled with argon gas. The chamber has a length and radius of 42 cm and 5 cm, respectively, and it is bounded by two copper electrodes with fixed potentials of 0 V (Anode) and -350 V (Cathode). The simulation has been done with two different conditions for different size of electrodes. The behaviour of plasma parameters presented at low pressure of 0.05 torr. To investigate this time-independent discharge and explore phenomena like the self-sustaining generation of secondary electrons and the formation of fixed regions, we use the Plasma Module of COMSOL Multiphysics software for numerical modelling [2]. The results presented in this paper provide valuable insights for the design and development of new technologies in the field of surface treatment.

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[Tu.Po.16] Elastic Overtaking Collisions of Large Amplitude Solitons

Carel Olivier (North-West University).

Abstract

In this poster presentation, a fluid simulation study of overtaking soliton collisions is undertaken for a plasma consisting of cold ions and Boltzmann electrons. An asymptotic analysis approach is applied to construct single soliton initial conditions directly from the Sagdeev pseudopotential solution. This approach allows for the construction of large amplitude solitons with Mach numbers of up to 1.5, that is, solitons that propagate 50% faster than the acoustic speed. These solutions are used to simulate overtaking collisions of solitons. In the small-amplitude regime, collisions are shown to closely resemble two-soliton solutions obtained from reductive perturbation analysis. At larger amplitudes, results show elastic collisions despite the higher order nonlinear effects. The only effect of the higher order nonlinearities is the reduction (in magnitude) of the phase shift compared to that predicted from reductive perturbation analysis.

[Tu.Po.17] Analytical Model of a Magnetopause Current Sheet with Various Energy Distributions of Particles and Features of Its Small-Scale Instability

Vladimir Kocharovsky (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences), Mikhail Garasev (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences) and Anton Nechaev (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences).

Abstract

We develop analytical kinetic models of quasi-stationary multicomponent current sheets that remind magnetopause configurations and allow for an arbitrary particle energy distributions [1, 2]. Particle-in-cell numerical simulations of their evolution demonstrate that, despite the overall stability of these sheets, the Weibel-type magnetic turbulence can develop within them or be inhibited depending on the model parameters. Both cases are studied and compared for two variants of the particle energy distribution — Maxwellian and Kappa. The purpose of this work is to elucidate the structure and parameters of current sheets, in which the Weibel-type instability is possible despite the presence of a self-consistent magnetic field and inhomogeneity of the plasma. Another aim is to study the main features of a weak magnetic turbulence that exists inside such current sheets of a magnetopause type.

In agreement with analytical estimates, simulations show that our simplest models without counterstreams of particles are stable with respect to the aperiodic Weibel-type instability. On the contrary, sheets with counterstreams and a wide region of weak enough magnetic field between them can be unstable. Nevertheless, in our simulations even they are not destroyed by the Weibel-type instability, which saturates at a relatively low level and leads only to a weak deformation of the large-scale current structure.

The obtained results make it possible to identify the features of growth and nonlinear evolution of Weibel-type turbulence in the presence of a non-uniform self-consistent magnetic field and an inhomogeneous plasma with a non-trivial particle velocity anisotropy. The gradients of the magnetoactive plasma's local properties significantly influence the spectrum of growing magnetic perturbations and the character of its gradual evolution from short to long wavelengths in comparison with the case of a homogeneous plasma.

We show that for both Maxwellian and Kappa particle energy distributions, the global structure of the current sheet and the character of the Weibel-type instability in its inner part are similar. Hence, the possibility of long-term existence of small-scale quasi-magnetostatic turbulence in distributed multicomponent current sheets seems to be quite universal. We discuss the applicability of the constructed current sheet models and the features of their internal small-scale magnetic turbulence to the analysis of the phenomena in the vicinity of the magnetopauses of planets and late-type stars.

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[Tu.Po.18] Analytical Relation Between the Main Parameters of Magnetic Turbulence at the Nonlinear Stage of the Weibel Instability in Collisionless Plasma

Vladimir Kocharovsky (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences), Alexey Kuznetsov (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences) and Anton Nechaev (A.V. Gaponov-Grekhov Institute of Applied Physics, Russian Academy of Sciences).

Abstract

Based on the energy invariants for the Weibel instability in a collisionless nonrelativistic plasma, an analytical relation is obtained between the instantaneous values of the space-averaged energy density of the magnetic field, its dominant wavenumber, and the average plasma anisotropy parameter [1]. The relation is valid for arbitrary particle velocity distribution functions, including ones that vary with time at the nonlinear stage of instability. It is obtained under the assumption that the plasma is homogeneous along a certain axis, determined, for example, by an external magnetic field, and taking into account only the modes with wave vectors orthogonal to this axis. This limits the justification of the relation's correctness, but does not rule out its approximate validity for a wider class of real systems with a sufficiently narrow spectrum of the Weibel magnetic turbulence.

Universality of the analytical relation is verified for several typical examples with different particle energy distributions using two-dimensional particle-in-cell modelling. The simulations also reveals important features and a self-similar nature of the evolution of the spatial spectrum of the Weibel magnetic turbulence. In particular, the power-law profiles of the small- and large-wavelength slopes of this spectrum and the temporal power-law behavior of its maximum are demonstrated.

Using the obtained relation, we give an analytical estimate of the maximum magnetic field energy achievable during the Weibel instability. Namely, we show that its ratio to the initial longitudinal energy of particles, even for a large initial plasma anisotropy parameter, cannot exceed the value approximately equal to 0.2. This estimate could be used to limit the potential mechanisms of the generation of magnetic fields observed in space and laboratory, especially since the performed simulations give reason to expect that the relation or its close analogues will be approximately valid for a class of quite diverse problems of quasi-two-dimensional Weibel turbulence under the conditions of a relatively narrow spatial spectrum and a weak inhomogeneity of the plasma number density.

We discuss possible applications of the newly found analytical relation to the analysis of explosive kinetic processes in laboratory and space plasmas leading to the small-scale magnetic turbulence due to self-consistent filamentation of the electric current.

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[Th.Po.1] Determining and Optimizing for Plasma Current, Centroid Position, and Shape in the DEMOnstration Power Plant Using Bayesian Integrated Data Analysis and Experimental Design

Jeffrey De Rycke (Ghent University), Alfredo Pironti (University of Naples Federico II), Marco Ariola (University of Naples Federico II), Antonio Quercia (University of Naples Federico II) and Geert Verdoolaege (Ghent University).

Abstract

For the benefit of accurate and precise plasma parameter inference in the upcoming DEMO demonstration fusion reactor, we aim to leverage the probabilistic framework of Bayesian inference. This framework provides a modular way of combining complementary diagnostics and delivers distributions for the plasma parameters of interest. Using prior physical knowledge of the expected plasma current density distribution, together with forward models to find the theoretical diagnostic measurements, we can infer the most probable plasma current density distribution and its covariance function derived from the diagnostic measurements and their uncertainties. From this, we calculate the plasma current, centroid position, and shape.

The inference is based on multiple diagnostics: pick-up coils, flux loops, and saddle coils. They are external diagnostics and therefore do not provide internal information of the plasma. Nonetheless, it has been shown that using a Gaussian process and a well-informed prior distribution, one can reconstruct the current density distribution and poloidal flux distribution with relatively good agreement [1]. The disagreement between the current density distribution and the ground-truth distribution increases rapidly towards the plasma centre, yet there remains a qualitative agreement. The reconstructed plasma boundary remains reliable, though the error on the poloidal flux distribution becomes quite large towards the centre [2].

In addition, we employed Bayesian experimental design (BED) to study the optimal amount and distribution of tangential and normal pick-up coils, complying with the DEMO design restrictions. BED aims to maximize a utility function, specifically the Shannon entropy, which in our case meant achieving D-optimality or maximizing the determinant of the Fisher information matrix of the plasma parameters.

Acknowledgments J. De Rycke acknowledges the Research Foundation - Flanders (FWO) via PhD grant1SH6424N.

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[Th.Po.2] Predictive Maintenance in Fusion Devices with an Application to Condition Monitoring of Plasma-Facing Components

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Abstract

In the context of Reliability, Availability, Maintainability, and Inspectability (RAMI) analysis for DEMO, achieving an availability of 30-60% and minimizing unscheduled shutdowns annually are essential prerequisites. Predictive maintenance (PdM) can play a crucial role in ensuring regular, rapid, and reliable maintenance of the plant. PdM proactively optimizes maintenance schedules by estimating the remaining useful life (RUL) of components or subsystems of a machine, from historical sensor measurements. Critical components, such as the divertor and the first wall, face extreme thermal loads, intense thermal shocks, and bombardment by plasma ions, neutral particles, and energetic neutrons. The options for real-time monitoring of Plasma-Facing Components (PFCs) in such conditions are limited. Moreover, computational models for reactor-scale surfaces can require significant computational resources. To overcome these challenges, we employ data-driven PdM to analyse infrared data and expert domain annotations, identifying patterns and anomalies that may indicate damage to the PFC. In particular, this study focuses on the application of modern statistical methods, including machine learning and deep learning on infrared data from steady state heat load experiments.

[Th.Po.3] Collisional Dynamics in Tokamak Plasmas

Y.S. Seitkozhanov (al-Farabi Kazakh National University, Satbayev University), E.O. Shalenov (Satbayev University) and K.N. Dzhumagulova (al-Farabi Kazakh National University, Satbayev University)

Abstract

Collisions between charged and neutral particles in plasma can lead to energy and momentum transfer, which can affect plasma temperature and density profiles. Understanding and controlling these transport properties is necessary to achieve and maintain the conditions required for nuclear fusion reactions to occur in a tokamak.

In this study, we investigate collision frequency and energy transfer between electrons and Helium atoms. The interaction between an incoming electron and He atom was studied by the optical potential method, which consists of three interaction potentials accounting for various effects. Momentum-transfer cross section and phase shifts are found using the partial wave expansion and the variable phase approach, respectively.

The resulting effective frequency has a maximum depending on the energy. The experimental data found in the literature are consistent with the detected maximum. At energies lower than the maximum energy, our results coincide with other works. Energy transfer calculations for the energy values above the detected maximum showed that temperature equalization between helium and electron occurs more slowly than has been known.

Large tokamaks, including JET and JT-60U, also produce electron-positron pairs in plasma, where collisions between several MeV runaway electrons and thermal particles can produce up to 10^{14} positrons. How many positrons are generated depending on the energy of the runaway electrons, the differential production rate can be calculated using the runaway electron distribution and the Coulomb logarithm.

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[Th.Po.4] Biased H Mode During Edge Plasma Biasing in the TJ-II Stellarator

Cesar Gutierrez-Tapia (National Institute for Nuclear Research), Daniel Lopez-Bruna (Laboratorio Nacional de Fusion, CIEMAT), Julio Martinell (Instituto de Ciencias Nucleares, UNAM) and Alexander Melnikov (National Research Center "Kurchatov Institute").

Abstract

The transition from the L-mode to H-mode confinement in stellarators is triggered by a sudden local increase of the radial electric field, E_r , which in turn reduces the fluctuation amplitudes and consequently suppresses the turbulence. The H mode is achieved only when certain thresholds in the control parameters are exceeded. In the case of biased H modes, the transition is obtained only when the bias voltage applied to the probe exceeds a critical value. In this work we study the biased H mode using a transport model that includes neoclassical and turbulent contributions [1]. The biasing electrode is assumed to produce a localized radial current which in turn affects the ambipolar radial electric field E_r . The sharp local change in E_r triggers the confinement improvement around the edge region, by suppressing turbulent fluctuations due to the shear of the poloidal ion velocity. For the anomalous transport we use the ballooning approximation including the stabilizing effect of the $\mathbf{E} \times \mathbf{B}$ shear. Starting from experimental profiles, the simulations are carried out using the ASTRA transport code [2] exploring different scenarios, in time and in space, with positive and negative bias. The results of the simulated radial electric field, electric potential and poloidal ion velocity are confronted with experimental data.

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[Th.Po.5] Turbulence Driven by Density Gradients and Biasing in Texas Helimak

Dennis L. Toufen (Federal Institute of Education, Science and Technology of São Paulo), Zwinglio O. Guimarães-Filho (Institute of Physics, University of São Paulo), Felipe A. Pereira (Institute of Physics, University of São Paulo), Giuseppe M. E. Silva (Institute of Physics, University of São Paulo), Iberê L. Caldas (Institute of Physics, University of São Paulo) and Kenneth W. Gentle (Department of Physics and Institute for Fusion Studies, The University of Texas at Austin).

Abstract

The plasma turbulence dependence with the equilibrium density profile and externally imposed electric fields was investigated in Texas Helimak [1], a toroidal plasma device suitable for turbulence studies with flexible configurations regarding the equilibrium magnetic field and electrostatic biasing. In Texas Helimak stable plasma discharges are obtained using ECRH resonant heating whose maximum absorption position depends on the magnetic field intensity. The turbulence is measured simultaneously by 96 Langmuir probes selected among the more than 700 available ones. Also, it is possible to impose the electrostatic potential in some radial regions, by connecting a DC power device in one of the four bias plate sets. Initially, with all the bias plates grounded, the magnetic field intensity was changed by selecting the coils current into 36 different values (one per discharge), which makes the density peak position vary by almost half of the machine radial extension. The turbulence level profile follows the density profile changes. Moreover, by comparing the turbulence radial profiles with the density peak positions, it is possible to see that the turbulence level is enhanced in the negative density gradient side of the peak, as predicted for ideal interchange modes [2]. Furthermore, when considering discharges with just one of the bias plates polarized (the others grounded), we observed strong changes in the density radial profiles for both positive and negative bias values due to biasing, with the peak displacement in different directions depending on the bias signal: the peak is moved out of the bias plate incase of negative bias and in the opposite direction with positive biasing. The turbulence level and the density radial profiles follow similar trends in general, but the link between them is not so clear as in discharges without biasing.

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[Th.Po.6] Novel Energy Absorption Mechanism in Plasma Microdroplets

Animesh Sharma (Indian Institute of Technology Delhi) and Amita Das (Indian Institute of Technology Delhi).

Abstract

Two dimensional (X-Y plane) Particle-In-Cell (PIC) simulation were performed using EPOCH [1] to study the evolution of plasma microdroplet irradiated by a laser pulse. The laser wavelength of 800nm, FWHM pulse duration of 25 fs and spot size of 19 micron were chosen. The target plasma droplet of diameters 15, 30, and 60 microns were chosen. The laser intensity was non-relativistic with $a_0 = 0.5$. The plasma density in the microdroplet had a gradient from zero to $10 n_c$ (n_c being the critical density) over a region of two microns. In the simulation the laser propagates along the X axis and its transverse profile is centered about $Y = 0$ and is incident normal to the target. At $Y = 0$ the electric field of the laser is transverse to the surface of the target. At all other Y locations, due to the curvature of the target, an electric component normal to the surface exists. This component of the electric field extracts the electrons out of the target (Fig1a) following the well-known vacuum heating process. The electrons acquire certain amount of energy irreversibly from the laser during this phase. The gain in kinetic energy of the electrons is apparent from the initial rise seen in average particle energy (APE) in Fig.1 a. Thereafter, two additional phases of energy increment are observed. It should be noted that the two subsequent phases occur after the laser pulse has left the simulation box. The energy acquired during these phases is a result of collision between the two surface waves (excited by the

laser as it grazes through the target surface Fig.1a) which traverse the top and bottom hemispheres to collide at the diametrically opposite point (Fig 1b). In addition, electrons from the bulk also arrive here. The collision of the two surface and the bulk disturbances lead to irreversible gain of the electron energy. Thus the second phase of growth in energy corresponds to this collision, the third happens at the time when the three colliding disturbances reverse their direction and again hit each other at the other side of the target (Fig 1c). A detail understanding of this process will be provided.

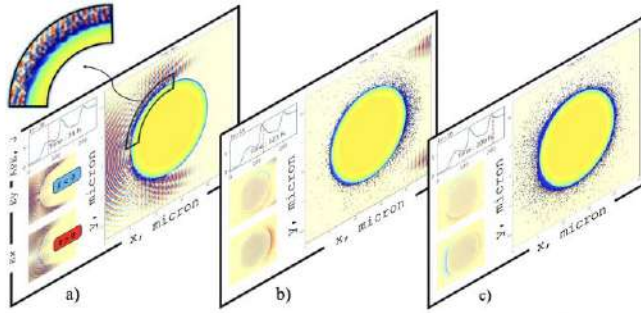


Figure 1. Temporal evolution of electric field, average particle energy and electrons for laser interacting with plasma microdroplets.

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[Th.Po.7] Full-Wave Simulations of Helicon Waves for Plasma Wakefield Accelerators

Luis Carlos Herrera Quesada (University of Stuttgart), Alf Köhn-Seemann (University of Stuttgart), Nihls Fahrenkamp (University of Greifswald), Stefan Knauer (University of Greifswald), Peter Manz (University of Greifswald) and Günter Tovar (University of Stuttgart).

Abstract

In fundamental particle physics experiments, energy requirements have become difficult to achieve in circular accelerators. Linear wakefield devices appear as a modern solution to produce particles of very high energies: plasma wakefield accelerators provide significantly higher E-fields to accelerate particles than conventional linear particle accelerators. However, to achieve these high electric fields, the plasma medium must have a large electron plasma density to create strong gradients. It is known that helicon plasma discharges achieve very high electron plasma densities, allowing the system to acquire the necessary density magnitudes for accelerator purposes [1].

This study aims to understand helicon wave propagation and dissipation in plasma wakefield systems, as well as the influence of different antenna geometries on the efficiency of helicon wave excitation. Furthermore, the evolution of the radial plasma density gradient on the efficiency of coupling to the helicon wave will be studied.

The research plan consists of performing a theoretical analysis with a full-wave simulation using the finite-difference time-domain (FDTD) 3D code FOCAL, which solves Maxwell's equations coupled to the fluid equation of motion for electrons in a cold magnetized plasma [2]. In parallel, computational simulations are made using the COMSOL Multiphysics code package. The simulations will be applied to the linear plasma device VINETA.75, located at the University of Greifswald. The device's geometry will be implemented in the numerical models as a first step. It is then planned to compare the results with measurements of the wavefield in VINETA.75. Also, external cooperation is made with the teams in the MAP device at the University of Wisconsin-Madison, and the PROMETHEUS-A and AWAKE experiments at CERN[3].

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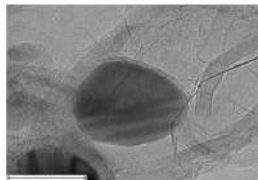
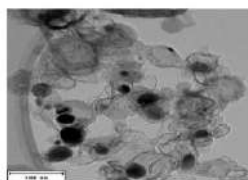
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[Th.Po.8] Nickel-Carbon Composite Nanomaterial Synthesis via Plasma Catalytic Pyrolysis

Jafar Fathi (Institute of Plasma Physics, Prague), Ondřej Jankovský (University of Chemistry and Technology Prague), Michael Pohořelý (University of Chemistry and Technology Prague), Vineet Sikarwar (Institute of Plasma Physics, Prague), Michal Hlína (Institute of Plasma Physics, Prague) and Alan Mašláni (Institute of Plasma Physics, Prague).

Abstract



The experiment was conducted using the PLASGAS plasma reactor, which has an internal volume of 200 liters and utilizes the Hybrid Water Stabilized Plasma torch. The characteristics of the Hybrid Water/Gas DC Arc Plasma Torch and the PLASGAS reactor are detailed by Hrabovský et al. [1] and Fathi et al. [2], respectively. Previously, this system has been employed for the pyrolysis of methane [3] and natural gas [4]. In this study, however, we investigate methane pyrolysis by adding Nickel

Oxide powder as a catalyst. This experiment introduced a 100 SLM methane feed into the reactor, with plasma power set at 110 kW. The resulting gas composition was 86.5 vol% hydrogen and 12.1 vol% carbon monoxide, with only 1.2 vol% methane remaining unconverted and 0.1 vol% acetylene produced. In addition to hydrogen production, a solid carbon-nickel (core-shell) structure nanocomposite material was synthesized. XRD and EDX analysis confirmed that the nickel oxide was reduced to zero-valent nickel nanoparticles with particle sizes less than 50 nm. The TEM images of the produced composite material are shown below.

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[Th.Po.9] Community Structure of Earth's Magnetic Field Measurements

Victor Muñoz (Departamento de Física, Facultad de Ciencias, Universidad de Chile) and Sebastian de la Maza (Facultad de Ciencias Físicas y Matemáticas, Universidad de Chile).

Abstract

The Earth's magnetic field has variations both in the time and spatial domains, which are due to the internal dynamics of the Earth's core, the forcing by external sources like the solar wind, or fluctuations induced by coupling between neighbouring regions. This leads to various levels of correlations between magnetic field readings on the Earth's surface, which map the interplay of these factors across multiple time and space scales. In this work, we propose to describe and study this complex dynamics of spatiotemporal correlations by means of tools derived from graph theory and complex networks, which have shown to be useful to describe the behavior of various systems of geophysical interest [1, 2, 3]. In particular, we study the evolution of magnetic field measurements on the Earth's surface along the 23rd solar cycle.

Based on records by 59 magnetometers during the 23rd solar cycle [4], we define a complex network where nodes are points on the Earth's surface (magnetometers), and their connections represent the degree of similarity between the time series observed at those points. Our results show that there is a correlation between the evolution of network community structure and geomagnetic activity. In addition, we study the dependence of the results of the methods used to define the similarity between time series (and, therefore, to define the connection between nodes), in order to establish the best possible sensitivity for the community structure with respect to the geomagnetic activity, as measured by the Dst index and the sunspots number. We show that the choice of similarity method is not as relevant as the choice of the correlation threshold which determines whether two nodes are actually connected or not. Our work suggests that analysis of the Earth's magnetic field variations using complex network and community structure analyses, can be useful to understand the geomagnetic activity along the solar cycle.

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[Th.Po.10] Characterizing the Solar Activity Using the Horizontal Visibility Graph

Tomás Zurita (University of Chile), Víctor Muñoz (University of Chile) and Denisse Pastén (University of Chile).

Abstract

The Sun and its behavior are studied by means of complex networks. The complex network was built using the Horizontal Visibility Graph (HVG) algorithm. This method maps time series into graphs in which every element of the time series is considered as a node and a visibility criterion is defined in order to connect them [1]. The HVG method has been widely used to analyze various systems such as pulsating variable stars [2], solar activity [3, 4] and blazars [5]. Using this method, we construct complex networks for magnetic field and sunspots time series encompassing four solar cycles, and various measures such as degree, clustering co-efficient, betweenness centrality and eigenvector centrality were calculated. In order to study the system in several time scales, we perform both a global, where the network contains information on the four solar cycles, and a local analysis, involving moving windows. Our results suggest that complex networks can provide a useful way to follow solar activity, and reveal new features on solar cycles.

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[Th.Po.11] Radiation Pressure- Driven Rayleigh-Taylor Instability in Compressible Strongly Magnetized Ultra-Relativistic Degenerate Plasmas in White Dwarfs

Ravinder Bhambhu (School of Physical Sciences, Jawaharlal Nehru University) and **Dr. Ram Prasad Prajapati** (School of Physical Sciences, Jawaharlal Nehru University).

Abstract

The radiation pressure and strong magnetic fields are prominent in the structures of Rayleigh-Taylor (R-T) instability in the interior of white dwarfs. We have investigated the radiation pressure-driven R-T instability in a compressible and magnetized ultra-relativistic degenerate strongly coupled plasma. The equation of state has been derived for such systems incorporating ultra-relativistic degenerate electrons with their radiation pressure and ion gas compressibility. The dispersion relation of the density gradients driven R-T instability is analyzed using the generalized hydrodynamic (GH) fluid model in the strongly coupled and weakly coupled limits [1]. We assumed the electron fluid is inertial-less, ultra-relativistic degenerate and weakly coupled while the ion fluid is non-degenerate and strongly coupled [2].

It is observed that the R-T instability criterion has been modified significantly due to radiation pressure, ion gas compressibility and degeneracy parameter. In the kinetic limit, the instability region is shorter than the hydrodynamic limit due to the dominance of plasma frequency over the viscoelastic relaxation frequency. The outcomes are explored in analyzing the development of R-T instability in the strongly magnetized carbon-oxygen white dwarfs. The radiation pressure, electron temperature and ion density strongly suppress the growth rate of the R-T instability in the interior of white dwarfs. The strong magnetic fields introduce asymmetry to the system by destabilizing the R-T unstable modes. The present results are also useful for understanding the R-T instability in the star formation and dense plasmas in inertial confinement fusion in some limiting cases.

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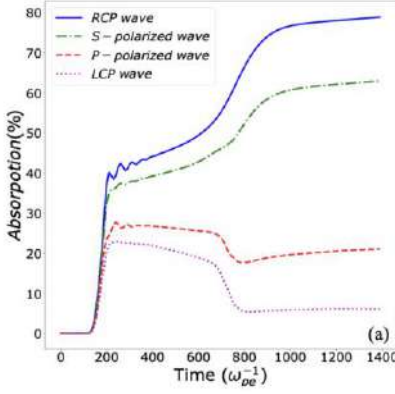
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[Th.Po.12] Two-Dimensional PIC Simulations for Electromagnetic Wave Propagation Oblique to Inhomogeneous Applied Magnetic Field in Plasma

Trishul Dhalia (Indian Institute of technology Delhi) and Amita Das (Indian Institute of technology Delhi).

Abstract

There has been significant interest lately in the study of Electromagnetic (EM) waves interacting with magnetized plasmas. The variety of resonances and the existence of several pass and stop bands in the dispersion curve for different orientations of the magnetic field offer new mechanisms of EM wave energy absorption [1,2,3]. However, earlier studies have been investigated only special cases of magnetized plasma geometry (e.g. RL mode ($\mathbf{k} \parallel \mathbf{B}_{\text{ext}}$ ($\theta = 0$) or X-mode $\mathbf{k} \perp \mathbf{B}_{\text{ext}}$ ($\theta = \pi/2$) configuration). In these cases, EM wave get absorbed at their respective resonances (e.g. for $\theta = 0$ electron cyclotron resonance ($\omega_{\text{EM}} = \omega_{\text{erc}}$), and for $\theta = \pi/2$, upper hybrid resonance ($\omega_{\text{EM}} = \omega_{\text{uh}}$)). We consider here the case of EM wave propagation at an oblique angle with respect to $\mathbf{B}_{\text{ext}}$. We tailor the magnetic field inhomogeneity such that the EM wave pulse encounters resonance layer ($n^2 \rightarrow \infty$) within the plasma. A 2-D Particle-In-Cell (PIC) simulation using the OSIRIS 4.0 platform has been carried out for this particular case. A significant enhancement in absorption leading to almost complete absorption of laser energy by the plasma have been observed (Fig.1). A detailed study characterizing the role of external magnetic field profile, EM wave intensity, etc., has also been carried out.



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[Th.Po.13] On the Gardner Equation for Nonlinear Waves in Multispecies Plasmas

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Abstract

The Gardner equation is a combination of the KdV equation (with quadratic non-linearity having constant coefficient B) and the modified KdV equation (with cubic non-linearity and constant coefficient C). For consistency, whereas C is of order one B should be very small, given the expansions used in the derivation of all KdV-like evolution equations. If B were of order one, the quadratic term would prevail over the cubic term, which could then be neglected. The sign of B is irrelevant because if $B < 0$ the Gardner equation can be rescaled to the case $B > 0$. The sign of C , however, is quite important. Even though the Gardner equation is fully integrable for both positive and negative C , the usual soliton solutions can only be generated for $C > 0$, which is of crucial importance should one want to study the collisions of solitons. A multispecies plasma model should therefore be detailed enough that the compositional parameters allow for a tiny B and a positive C . This aspect has been over-looked or neglected by several authors using the Gardner equation in their analysis, mostly due to the complexity of the plasma compositional parameters. In essence, a simple ion-electron plasma does not have enough compositional flexibility to go beyond the KdV equation.

It will be shown on a relevant dusty plasma model [1], comprising cold negative dust grains, Maxwellian electrons and nonthermal protons, that it is indeed possible to have B close to zero, while $C > 0$. When B gets larger, C can become negative, but in that case the absolute value of B is larger than that of C , leading to a regime where the relevant equation is the KdV, not the Gardner equation. In between, there is a region where the compositional parameter scan be adjusted such that, e.g., $B = 0.01$ and $C = 0.35$. In the theoretical studies of nonlinear electrostatic plasma waves there is another method, namely Sagdeev's pseudopotential analysis, where the Poisson equation is integrated leading to an "energy integral," with full nonlinearities but requiring a numerical integration of the Poisson equation to produce the profiles. The case of $B = 0.01$ and $C = 0.35$ will be discussed via the two approaches (Gardner and Sagdeev), and the results compared. Discrepancies might indicate the limits of the Gardner approach at higher amplitudes, given that in reductive perturbation theory the expansions limit the nonlinearities to the quadratic or cubic terms.

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[Th.Po.14] Landau Damping of Dust Ion-Acoustic Solitary Waves in Non-Maxwellian Dusty Plasmas

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Abstract

Landau damping is a captivating phenomenon, manifested as wave dissipation (damping) occurring – despite the absence of inter-particle collisions – due to resonant interactions between waves and particles [1]. Focusing on the ubiquitous presence of charged dust in space and in laboratory plasma environments [2], one may wonder how Landau damping may affect the propagation of electrostatic solitary waves (ESWs) and associated instabilities in space and astrophysical environments [3]. This investigation explores the effect of Landau damping on nonlinear dust-ion-acoustic waves (DIAWs) in a dusty plasma composed of inertial ions, energetic (suprathermal) electrons, and immobile dust. A cold-ion fluid model, coupled to a Vlasov-type kinetic equation for the electron dynamics, has been adopted. A kappa-distribution [4, 5] is assumed as equilibrium state for the electrons, in account of the non-Maxwellian particle behavior observed in Space [5].

A multiscale perturbation technique has been adopted and shown to lead to an evolution equation in the form of a modified Korteweg–de Vries (KdV) equation that incorporates a dissipative term, representing kinetic (Landau) damping [6]. Exact analytical solutions have been obtained, representing solitary waves undergoing amplitude decay over time. The combined parametric effect of Landau damping, non-Maxwellian electron statistics (via the kappa parameter) and dust density on the characteristics of DIAWs has been examined. The results of this investigation aim at generalizing earlier work [6, 7] by shedding some light on the dynamics of nonlinear waves in various space and astrophysical dusty environments [2,3].

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[Th.Po.15] Fluid Simulations of Electrostatic Solitary Waves

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Abstract

Electrostatic solitary waves (ESWs) are an ubiquitous occurrence in plasmas in the laboratory [1] and in Space [2], where they are observed widely in relation with bipolar electric field structures recorded by missions in planetary magnetospheres [3]. Their theoretical modeling represents a long standing challenge for theoreticians and experimental researchers alike. Even though an analytical framework has been established for the modeling of ESWs since the pioneering work of Sagdeev and coworkers [4] since decades ago, that framework provides “static” predictions of the solitary wave profile and provides no information on their propagation characteristics under realistic conditions. In particular, although the Sagdeev (pseudopotential) method -- and its small-amplitude Korteweg – de Vries (KdV) counterpart [5]-- successfully reproduce(s) the expected waveforms (i.e. a pulse for the ES potential and a bipolar form, in general, for the E-field), the (expected, but still not verified) soliton properties of the resulting -numerical- solutions from the Sagdeev formalism have not been analyzed.

We have undertaken a study of the dynamics of electrostatic pulses, from first principles. Based on a basic fluid model as a starting point, we have performed a series of numerical (fluid) simulations, in order to investigate the stability of electrostatic pulses and their interaction properties. Various propagation scenaria have been considered, and the pulses’ stability and (anticipated) soliton characteristics have been benchmarked. Solutions near the acoustic speed appear to be “KdV-like” (as expected), while strongly superacoustic (supersonic) pulses are characterized by a different amplitude-velocity relation but still possess the expected stability properties, throughout propagation and mutual interaction.

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[Th.Po.16] Modulated Electrostatic Wavepackets in the Presence of Damping and Forcing

Georgios Fotopoulos (Institute of Applied Technology, Abu Dhabi Polytechnic), Nikos Lazarides (Institute of Applied Technology, Abu Dhabi Polytechnic) and Ioannis Kourakis (Khalifa University).

Abstract

Electrostatic wavepacket modulation in plasmas is a widely studied nonlinear phenomenon: it is manifested as the variation (“modulation”) of a wavepacket’s harmonic amplitude in space and time, and may be attributed to various processes, such as self-modulation (self-interaction), cross-modulation, wave-wave interaction or due to ponderomotive processes [1, 2]. Modulated envelope (amplitude) dynamics is known to be described efficiently by a nonlinear Schrödinger (NLS) type equation [3], a nonlinear PDE that models the envelope’s evolution in space and time, in a slowly-varying amplitude approximation. In a fluid-plasma theoretical context, the NLS equation can be derived from a fluid model by means of a multiple scales technique [1,2,4]. Stability analysis may be performed, leading to predictions for modulational instability occurrence, a mechanism often postulated as a precursor state to freak wave (rogue wave) formation [2,4].

We have earlier undertaken a study of the modulational dynamics of an electrostatic wavepacket, from first principles [5]. Based on a cold-ion fluid model as a starting point, incorporating a phenomenological damping term in account of collisions, we have derived an NLS equation characterized by a complex-valued nonlinearity coefficient Q and a real dispersion coefficient P . The imaginary part of Q is thus associated with nonlinear dissipation of the envelope, as it results in a “damping” force that breaks the integrability of the NLS eq. and results in amplitude decay. Based on (and inspired by) [6], where numerical simulations revealed extreme wave events for the damped and forced NLS equation, we have performed a series of computer simulations in order to investigate the (in)stability and the evolution of an initial condition in the form of an envelope soliton (i.e. an exact solution of the unperturbed NLS eq.). Different possibilities have been explored, namely including the option of stabilizing the envelope pulse by imposing an ad hoc external forcing term to counteract dissipation.

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DIAGNOSTICS AND APPLICATIONS OF ATMOSPHERIC PRESSURE PLASMAS FOR TRIGGERING OF CELL MECHANISMS

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Abstract. Cold plasmas with their rich plasma chemistry with ample amounts of Reactive Oxygen and Nitrogen Species (RONS) are driving the development of applications in the fields of Plasma Medicine and Plasma Agriculture (see Adamovic et al. 2022). One of the applications is by using Plasma Treated Liquids (PTLs). When aqueous solutions (water, cell medium, saline solution etc.) are exposed to cold plasma, reactions occurring in the gaseous phase and at the gas-liquid interface, introduce short and long-living RONS, such as OH, O, O₃, H₂O₂, NO₂[•], NO₃[•], into the aqueous phase (see Bradu et al. 2020). We have used several configurations of atmospheric pressure plasma sources that were powered in the range of frequencies from kHz to GHz. They were chosen according to the application and characterized in detail by optical emission spectroscopy, electrical characterization, ICCD imaging, mass spectrometry etc. The applications by using PTLs of these sources include decontamination of bacteria, cancer cell treatment, wound treatments and toxicity tests.

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Time evolution of emission profiles and spatial structure of a plasma jet streamer discharge in contact with liquid

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Atmospheric pressure plasma jets have been intensively studied for the last two decades due to their wide field of application [1-2]. One of the most common targets exposed to cold plasma is liquid. Numerous investigations have demonstrated that the electrical properties of the plasma and its reactivity impact the water target by modifying physicochemical features such as pH, electrical conductivity, and the transfer of reactive species into the liquid [3]. In order to fully comprehend the complex interaction between discharge characteristics and treated liquid properties, an in-depth diagnostic of the plasma jet is required. Analysis of emission profiles from the discharge can provide various information. Ultrafast cameras can capture clear discharge images on a timescale ranging from ns to μ s, which allows insight of development of discharge profiles and interactions with the water surface [4].

In this research we used two ultrafast framing cameras to record temporal evolution of the streamer emission generated by using a specially designed pin-type atmospheric pressure plasma jet operating in contact with a liquid sample. The experimental setup of the atmospheric pressure pin-electrode jet operating in contact with liquid sample is presented schematically in Figure 1. The powered electrode was connected to the high-voltage (HV) and the second electrode was the target – a water sample placed in a grounded quartz vessel.

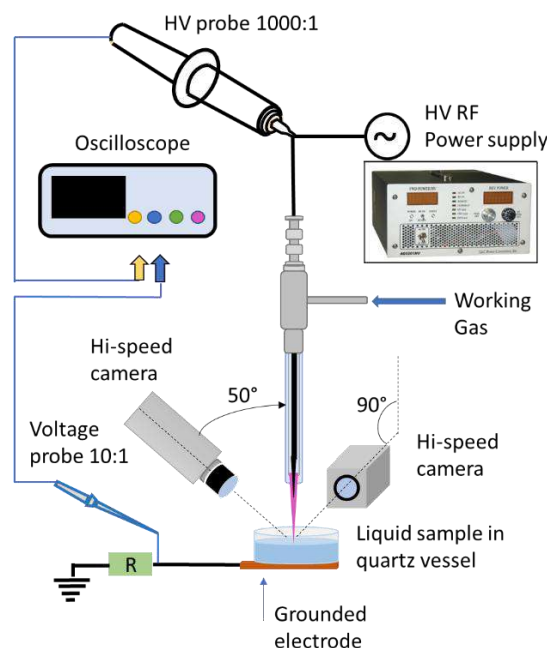


Fig. 1: Schematic representation of the experimental setup.

Distilled water, tap water, and saline solutions were used as targets in order to test targets with different conductivities. Pure He or Ar were used as working gases with gas flows of 1 slm and 2 slm,

respectively, adjusted by a mass flow controller. The plasma in the experiments was generated and controlled by using a commercial HV high-frequency power supply providing continuous sinusoidal signal with the frequency of 350 kHz. To monitor voltage and current waveforms we used an oscilloscope, and corresponding probes. The discharge current flowing through the ground line was obtained by measuring voltage drop across a 1 k Ω resistor.

Ultrafast frame imaging of the streamer discharge was performed by using of two Hi-speed cameras (I-Speed 726, IX Cameras, and FASTCAM SA-X2, Photron). The development and propagation route of streamers formed between powered pin electrode of the jet and the liquid surface were observed from side-on positions. The first camera was positioned perpendicular to plasma jet axis in order to observe the development and expansion of the streamer between the powered electrode and the liquid. To capture formation and propagation of smaller filaments on the liquid's surface, the second camera was installed above the sample, at an angle of 50° from plasma jet axis. Fast-framing cameras allowed observation of the transient behaviour of the streamer by recording of the emission profiles in a time frame short enough to see the effect. The spatial distribution of the emission generated by the streamer discharge and branching of the filaments at the water surface were recorded for different jet operating parameters and the liquid target conductivity.

Figure 2 shows discharge profiles in argon (a) and helium (b) in the case when the target was 4 ml of distilled water (conductivity 2.1 $\mu\text{S}/\text{cm}$). The images were taken with a camera placed at an angle of 90° with respect to the vertical axis of the plasma jet. The results showed that the contact surface of the Ar streamer and water was much larger than in the case of helium. Branching at the interface level was more pronounced in Ar streamers. The streamer was more intense, less distorted and with more homogeneous emission along its axis in the case of He plasma.

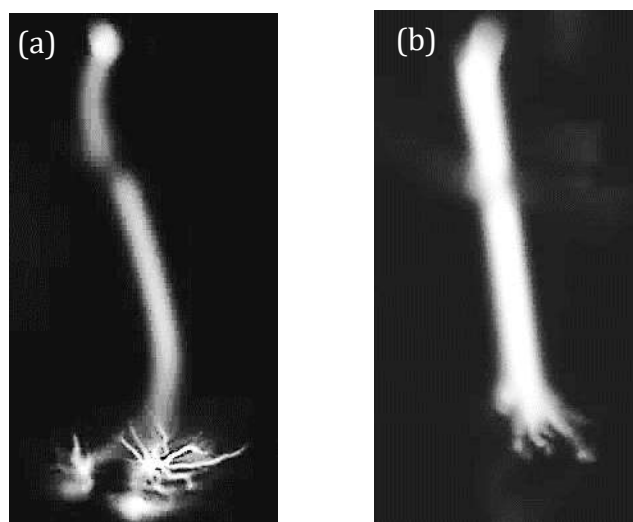


Fig. 2: Streamer spatial profiles in argon (a) and helium (b) in the case when the target was 4 ml of distilled water.

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Characterization and optimization of 3-pin atmospheric pressure plasma jet system for water treatment

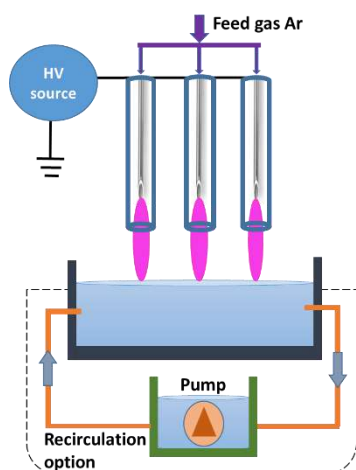
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Cold plasma-based treatment processes are eco-friendly because they operate without any additional chemicals at atmospheric conditions. Contact of plasma with water generates a rich mixture of Reactive Oxygen and Nitrogen Species (RONS) (e.g. $\text{HO}\cdot$, $\text{O}\cdot$, O_3 , H_2O_2 , $\text{NO}\cdot$, ONOOH , NO_3^- , NO_2^- , ...) that are deposited in the water making Plasma Activated Water (PAW). Treatment of water for production of PAW has been studied extensively in the last decade [1]. This led to development of various electrode geometries for atmospheric pressure plasma sources and new generation of power supply units. Increase number of investigations of different plasma systems led to a discovery of some new phenomena like Pulsed Atmospheric Plasma Streamer (PAPS) [2]. But still the biggest obstacle in the field is the volume of these discharges i.e. their upscaling so that they can be used in water treatments for industry, agriculture, medicine etc. Usually the water treatment is done by atmospheric discharge positioned above the water surface or with discharge inside the water sample. In case of discharge positioned above the water the effective surface that is in direct contact with the discharge is small. The increase of contact surface can be done by increasing the plasma area or by introducing recirculation of water in the system.

In this work we present the results of water treatment by 3-pin Atmospheric Pressure Plasma Jet (APPJ) (shown in Fig. 1) which was utilized in a system for PAW production. Two options of the system were investigated: without and with the recirculation system for water samples [3]. 3-pin APPJ was made by using syringe needles as powered electrodes connected to a high voltage power supply. The inner diameter of needles is 1 mm and the outer diameter 1.8 mm. Each needle is placed in the glass tube and the distance between the tip of the needle and the edge of the tube is 5 mm. During treatments the water sample was positioned at the distance of 20 mm from the needle tips. The plasma was driven by an RF power supply with a frequency of 320 kHz. Argon gas with a total flow rate of 2 slm was used as a feed gas throughout the experiments.



A dielectric vessel holding a water sample was placed under the plasma source. Water in the vessel could be stationary (in case of smaller sample volumes) or it could be connected in a recirculation loop with an additional vessel, increasing the total volume of the water sample up to 600 ml. The idea was to test if we could increase the volume of obtained PAW while keeping similar properties, i.e. pH and RONS concentrations. In case of sample recirculation, a water pump (Atman AT-300) in the external vessel was used to run the water sample under the plasma jet and to adjust the flow of the sample in the system. Due to the capacity of the pump in some cases we had to use additional chokers to additionally reduce the water flow. In our experiments we have used volumes from 100 ml up to 600 ml of water.

Figure 1. Experimental setup of the 3-pin APPJ for water treatments

The treatment times were from 10 min up to 90 min depending on the volume and if the water recirculation was used. After treatments, we measured pH using a probe while the presence of RONS was measured by using strips and spectrophotometrically.

For stationary water sample case we treated volume of $V_0=100$ ml of distilled water. After the treatment time of 20 mins we measured pH of 3.5 while concentrations of H_2O_2 , NO_2^- , and NO_3^- were around 10 mg/l, 20 mg/l and 10 mg/l respectively. By addition of the recirculation system we were able to treat volumes of 400 ml and 600 ml of distilled water. For these measurements the input power to the pin-jet was kept the same as for the stationary case.

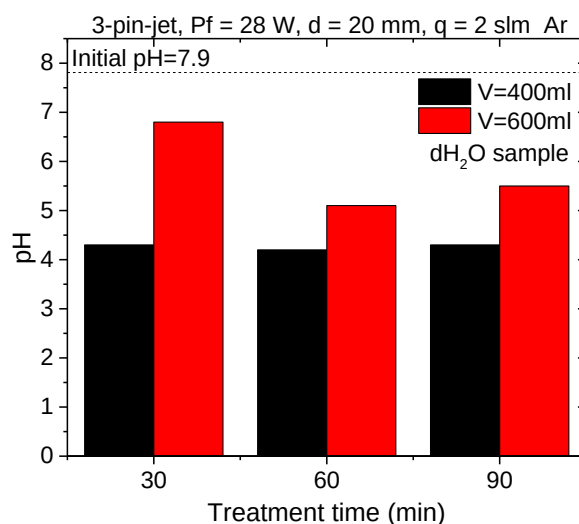


Figure 2. Measurements of pH of water samples treated for 30 mins, 60 mins and 90 mins using the recirculation system for two sample treatment volumes.

Since the volume sample was increased, we increased also the treatment time. In Figure 2 we show measurements of pH values after 30 mins, 60 mins and 90 mins of treatment. In case of $V_0=400$ ml pH values were almost constant despite the increased duration of treatment and stayed around 4. For $V_0=600$ ml of sample, pH was reducing more slowly in time with respect to 400 ml case. After 60 mins of treatment pH was further reduced and the final value stayed around 5. However, increased sample volume affected creation of reactive species. So even after 90 mins of treatment concentrations of H_2O_2 , NO_2^- , and NO_3^- were not reaching the values obtained with 100 ml of sample and were below 5 mg/l, 10 mg/l and 5 mg/l respectively for 400 ml volume and all below 5 mg/l for 600 ml of water sample.

So, on one side we obtained smaller reduction of pH by increasing the amount of the water sample treated. Generating PAW with less acidic conditions can be beneficiary for some applications where low pH can induce negative effects (e.g. application related to seeds and plants). Nevertheless, increasing the amount of the water sample reduces production of long-lived reactive species in PAW. This reduction cannot be compensated by just increasing the treatment time even if going to extreme duration such as 90 minutes. Therefore, when scaling up the system keeping target RONS concentration and their tuning has to involve changing more parameters so the optimization presents a complex and entangled task.

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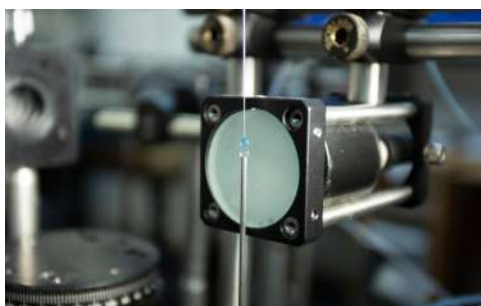
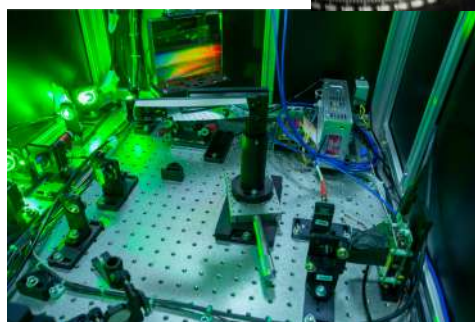


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Following the successful **1st edition in 2022**, we are pleased to announce the second edition of **The International Conference on Laser, Plasma and Radiation – Science and Technology (ICLPR-ST)**, which is organized by **INFLPR** in partnership with **Laser Plasma and Radiation – Science and Technology Association**, between June 16-21, 2024, in the magnificent scenery of the Danube Delta, Romania.

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Characterisation of atmospheric pressure plasma jets in contact with liquids, chemistry in liquids and applications in biomedicine and agriculture

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In the last two decades applications of cold plasmas in the fields of Plasma Medicine and Plasma Agriculture have driven the development of non-equilibrium plasma sources that operate at atmospheric pressure [1]. Their rich plasma chemistry, with ample amounts of Reactive Oxygen and Nitrogen Species (RONS), is responsible for triggering various mechanisms and effects in cells. The application of cold plasmas can be direct, when discharge is in the contact with the cells, and indirect, through application of Plasma Activated Liquids (PALs). In the latter case the main species interacting with cells are long-living RONS. When aqueous solutions (water, cell medium, saline solution etc.) are exposed to cold plasma, reactions occurring in the gaseous phase and at the gas-liquid interface, introduce short and long-living RONS, such as OH, O, O₃, H₂O₂, NO₂⁻, NO₃⁻, into the aqueous phase [2].

We have used several configurations of Atmospheric Pressure Plasma Jets (APPJ) that were powered by power supplies with frequencies ranging from kHz to GHz and with different electrode geometries. Each of these APPJs were chosen depending on the application and characterized in detail by optical emission spectroscopy, electrical characterization, ICCD imaging, mass spectrometry etc. The applications of these APPJs include direct and indirect treatment of plant cells, cancer cells, bacteria biofilm, suspension of bacteria, production of Plasma Activated Water/Medium for wound healing/anticancer vaccines etc. [3-5]. Also, in order to test the toxicity of PAW we have used a model plant *Lemna minor* and investigated the influence of the various concentrations of PAW on plant growth and amount of chlorophyll in the plant.

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Metabolic Response of Carrot Calli to PAW and Direct Plasma Treatment

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Plant callus tissue culture is a widely used tool in plant biotechnology for the genetic manipulation of plants, for studies of plant metabolism and cell development, and for the commercial production of valuable compounds as an alternative to direct tissue extraction of naturally occurring products that cannot be chemically synthesized. Recent advances in cold plasma or non-thermal atmospheric pressure plasma (NTAP) technology offer a wide range of applications for the treatment of plant cells/tissues. Here, we investigated alternations in plant metabolism induced by different plasma treatments using calli of three carrot varieties (*Daucus carota* subsp. *sativus*). The growth of black calli was significantly reduced but could be stimulated by using PAW rich in reactive oxygen and nitrogen species (RONS), while direct treatment improved the growth of white and orange carrot varieties. The phytochemical profile of the treated calli differed between varieties and changed after plasma treatment, especially with the respect to the total amount of dominant phenolic compounds. The oxidative stress in the plasma treated carrot calli was generally more pronounced after PAW treatment and led to an increase in the activity of antioxidant enzymes. CAT and SOD, followed by POX, are considered to be the front line of antioxidant defense of carrot calli against ROS generated by plasma treatment. To understand the effects of direct and PAW treatment on the antioxidant potential of calli, the expression of various genes related to the metabolism of carotenoids and anthocyanins was also investigated.

Scalability and Tailoring Reactive Species in Plasma-Activated Water Treatment Systems

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Non-thermal Plasmas (NTP) at atmospheric pressure create a rich gas phase chemistry of Reactive Oxygen and Nitrogen Species (RONS). When in contact with liquids, NTP induces the production of diverse short- and long-lived RONS species [1,2]. NTP is increasingly used to treat liquid samples, exploiting the chemistry of the active plasma volume to impact treated liquids properties. In plasma agriculture and biotechnology, Atmospheric Pressure Plasma Jets (APPJs) have emerged for water treatment, creating plasma activated water (PAW) or for destruction of pollutant molecules dissolved in water [3,4]. Despite successful PAW applications, complete understanding plasma-liquid interaction mechanisms and standardizing device performance for varied applications remain significant challenges.

In this work we applied two plasma sources for pure water treatment and tailored the RONS concentration in liquid by modifying the plasma chemistry in the gas phase, the type of water, and the parameters of the sample vessel. The first source was a plasma jet in solid pin electrode configuration (pin-jet) operating in the kHz regime with He or Ar as working gases. We demonstrated that in some cases the argon regime produces 10 times higher concentrations of reactive species (H_2O_2 , NO_3^- , NO_2^-) than when operating with He gas. Sample treatment volumes were from 4 ml up to 30 ml.

However, while this method is effective for producing highly reactive PAW, having small interaction surface limits its capacity to treat larger volumes of water needed for certain applications. As a first step, we scaled up the system by increasing the number of pin-jets – the multi-jet which is equipped with three needle electrodes [4]. Measuring the physico-chemical properties of the liquid after treatment revealed the effectiveness of increasing the treated volume and we obtained concentrations of RONS up to 100 mg/l. Apart from the operation against stationary sample with volumes of several tens of ml, this system can treat circulating water, resulting in a substantial increase of the treated volume, scaling up by tens of times. The maximum sample volumes treated with recirculation system were 600 ml.

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UNIVERZITET U BEOGRADU

FIZIČKI FAKULTET

Olivera J. Jovanović

**PLAZMA MLAZ SA ŠILJASTOM
ELEKTRODOM – KARAKTERIZACIJA I
PRIMENE U TRETMANIMA TEČNIH UZORAKA**

Doktorska disertacija

Beograd, 2026

UNIVERSITY OF BELGRADE

FACULTY OF PHYSICS

Olivera J. Jovanović

**PLASMA PIN-JET – CHARACTERIZATION
AND APPLICATION IN TREATMENTS OF
LIQUID SAMPLES**

Doctoral Dissertation

Belgrade, 2026

MENTOR:

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DATUM ODBRANE:

Zahvalnica

Doktorska disertacija pod nazivom „Plazma mlaz sa šiljastom elektrodom – karakterizacija i primene u tretmanima tečnih uzoraka“ je urađena pod mentorstvom dr Nikole Škora u Laboratoriji za neravnotežne procese i primenu plazme (raniji naziv: Laboratorija za gasnu elektroniku), Centra za neravnotežne procese, Centra izuzetnih vrednosti Instituta za fiziku u Beogradu, Instituta od nacionalnog značaja za Republiku Srbiju. Dr Nikola Škoro je rukovodio izradom disertacije, odabirom teme i metoda izučavanja, kao i fizičkim tumačenjem rezultata. Istraživanje je u velikom delu realizovano u okviru projekata Ministarstva prosvete, nauke i tehnološkog razvoja Republike Srbije - III41011 i Fonda za nauku Republike Srbije APPerTAinBIOM, broj 7739780, kojima je rukovodila dr Nevena Puač.

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Deo eksperimentalnih rezultata, u vezi sa optičkim merenjima brzom kamerom, urađen je u Laboratoriji za hemiju plazme Tehnološkog univerziteta u Brnu pod rukovodstvom prof. dr Františka Krčme (František Krčma, VUT, Brno, Czech Republic). Profesoru dr Františku Krčmi, koji nas je prerano napustio, veoma sam zahvalna na izuzetnoj saradnji, savetima i pomoći koju mi je pružio tokom boravka u Brnu. Želim da se zahvalim i kolegi dr Lukašu Dostalu (Lukáš Dostál, VUT), na nesebičnoj pomoći i zalaganju tokom zajedničkih merenja.

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pisanja disertacije, ali i na prijatnoj atmosferi koju ste stvorili, koja je ovaj deo rada učinila posebno lepim iskustvom.

Instrumentalnu metodu za određivanje produkata razgradnje u vodi tretiranoj plazmom, razvile su prof. dr Svetlana Grujić i doc. dr Ivana Matić Bujagić sa Tehnološko-metalurškog fakulteta Univerziteta u Beogradu i prof. dr Mira Petrović i dr Barbara Topolovec u Institutu ICRA u Ćironi (ICRA, Girone, Spain). Zahvalna sam im na saradnji iz oblasti primene plazme u tehnologijama prečišćavanja vode iz koje su dobijeni važni rezultati.

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Beograd, 2026.

Olivera Jovanović

Naslov disertacije: „Plazma mlaz sa šiljastom elektrodom – karakterizacija i primene u tretmanima tečnih uzoraka”

Sažetak:

Neravnotežne plazme na atmosferskom pritisku omogućavaju efikasno prenošenje električne energije na stvaranje i prenos hemijski aktivnih vrsta u tretiranu tečnost, bez korišćenja vakuumske tehnike i izazivanja termalnih efekata. U okviru ove disertacije je urađena dijagnostika niskotemperaturnog plazma mlaza za tretman tečnosti, konstruisanog u geometriji sa šiljastom elektrodom uz korišćenje He i Ar kao radnih gasova. Posebna pažnja posvećena je električnoj karakterizaciji, pri čemu je snaga pražnjenja određena kao ključni parametar koji je precizno meren i kontrolisan. Pražnjenje je proučavano pomoću tri optičke metode - emisijom spektroskopijom za detekciju pobuđenih vrsta, snimanjem profila pražnjenja iCCD kamerom uz upotrebu selektivnih optičkih filtera za praćenje relevantnih pobuđenih vrsta i snimanjem filamentarne strukture pri kontaktu sa površinom tečnosti brzom kamerom.

Promenom radnih parametara u sistemu, uočene su promene režima pražnjenja. Pokazano je da pri kontaktu sa tečnošću dolazi do širenja i račvanja strimera u filamente, pri čemu osobine mete značajno utiču na oblik strimera u delu gde je u kontaktu sa tečnošću i na dinamiku pražnjenja. Analizom promena u tretiranim tečnostima pokazano je da kontrolom parametara plazme može da se utiče na fizičko-hemijske karakteristike vode, čime je potvrđeno da interakcija nije jednosmerna – već da postoji uzajamni uticaj pražnjenja i mete. U okviru primena ove tehnologije je pokazano da plazmom aktivirana voda utiče na morfologiju biljaka, određen je njen stepen toksičnosti za biljke i pokazano je da se tretmanom zagađene vode uspešno razgrađuju odabrana organska jedinjenja.

Rezultati disertacije doprinose razumevanju procesa u višefaznim sistemima plazma-gas-tečnost i pružaju osnovu za kontrolisano stvaranje plazmom aktivirane vode na laboratorijskoj skali, što otvara prostor za dalje primene u biotehnologiji i novim multidisciplinarnim oblastima poput plazma poljoprivrede i tehnologije prečišćavanja vode.

Ključne reči: plazma mlaz na atmosferskom pritisku, neravnotežna plazma, snaga pražnjenja, strimersko pražnjenje, optička emisijna spektroskopija, tretman plazmom, interakcija plazme i tečnosti, reaktivne vrste, PAV, plazma poljoprivreda

Naučna oblast: Fizika

Uža naučna oblast: Fizika jonizovanog gasa i plazme

Title: " Plasma pin-jet – characterization and application in treatments of liquid samples"

Abstract:

Non-equilibrium atmospheric pressure plasmas enable efficient conversion of electrical energy into chemically active species that can be transferred into liquids, without the need for vacuum systems or the generation of significant thermal effects. In this dissertation, a low-temperature plasma jet designed for liquid treatment, based on a pin electrode geometry and operated with He and Ar as working gases, was comprehensively diagnosed. Special attention was given to the electrical characterization, where the discharge power was precisely measured and controlled as a key operational parameter. The discharge was investigated using three optical diagnostic methods: optical emission spectroscopy for identifying excited species, iCCD imaging with selective optical filters for tracking specific excited states, and high-speed imaging to capture the filamentary structure at the plasma–liquid interface.

By varying the operating parameters, transitions between different discharge modes were observed. It was demonstrated that upon contact with the liquid surface, the streamer expands and branches into filaments, with the properties of the target liquid significantly influencing the streamer morphology and discharge dynamics. Analysis of the treated liquids revealed that the physical and chemical characteristics of water can be adjusted by varying plasma parameters, confirming a bidirectional interaction between the plasma and the target. Within the applied aspect of this work, plasma-activated water was shown to affect plant morphology, while its phytotoxicity was assessed. At the same time, treatment of polluted water proved effective in decomposing selected organic compounds.

The results of this dissertation contribute to the understanding of processes in multiphase plasma–gas–liquid systems and provide a foundation for the controlled generation of plasma-activated water on a laboratory scale. This opens new opportunities for applications in biotechnology and emerging multidisciplinary fields such as plasma agriculture and water purification technologies.

Keywords: atmospheric pressure plasma jet, non-equilibrium plasma, discharge power, streamer discharge, optical emission spectroscopy, plasma treatment, plasma–liquid interaction, reactive species, PAW, plasma agriculture

Scientific field: Physics

Scientific subfield: Physics of ionized gas and plasma,

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1. UVOD

1.1 NERAVNOTEŽNA PRAŽNJENJA NA ATMOSFERSKOM PRITISKU

Neravnotežne (netermalne) plazme karakteriše uspostavljanje efikasnog mehanizma prenošenja energije na elektrone što ima za posledicu visoke temperature elektrona (reda veličine 10^4 K), dok druge, teže čestice (atomi, molekuli, joni) imaju temperature bliske ambijentalnoj temperaturi. Velika razlika između temperatura čestica u sistemu ostaje takva tokom vremena, tj. sistem se održava u neravnotežnom stanju pa odatle potiče i naziv za ovaj tip plazme. Ovakve osobine neravnotežnog pražnjenja dovode do dve osnovne prednosti u odnosu na termalne plazme i reaktore. Prva je da omogućavaju započinjanje hemijskih reakcija koje imaju pragove na visokim energijama, odnosno temperaturama, u sredinama koje se nalaze na sobnoj temperaturi. Druga je da se energija efikasno troši na stvaranje željenih hemijskih vrsta i ne gubi se na zagrevanje gasa.

Plazma se u laboratorijskim uslovima može uspostaviti pri različitim pritiscima. Da bi se plazma dobila na niskom pritisku, potrebni su vakumski sistemi koji su skupi i tehnološki zahtevni za održavanje. Kako bi se izbegli vakumski sistemi kao i da bi se omogućila primena u domenu uzoraka koji se ne mogu izlagati niskim pritiscima plazme na atmosferskom pritisku su izučavane u poslednjih nekoliko decenija (Lu et al., 2019).

U vazduhu na atmosferskom pritisku se pored neutralnih atoma i molekula uvek nalazi i određena koncentracija naelektrisanih čestica – jona i slobodnih elektrona. Naelektrisane čestice nastaju pod dejstvom kosmičkih zraka i njihova koncentracija je oko 10^9 m^{-3} na nivou mora (Hulburt, 1931). Iako se pod ovakvim uslovima gas ponaša kao dielektrik, ukoliko se primenom električnog polja dovede značajna količina električne energije, elektroni napuštaju atome i molekule čime se povećava broj slobodnih naelektrisanja. Elektroni ubrzavaju u električnom polju, dok slobodna pokretljivost jona ostaje mala, ali se time povećava broj sudara u kojima dolazi do multiplikacije elektrona. Na kraju, veći broj elektrona i jona u gasu menja njegove električne osobine i on postaje jonizovani gas ili plazma. Prelaz iz gasa u plazmu nije fazni prelaz u termodinamičkom smislu jer se dešava postepeno sa povećanjem temperature (Lu et al., 2019; Turner, 2016).

TOWNSEND-OVA TEORIJA PROBOJA U GASU

Postoje dve osnovne teorije koja opisuju proboj u gasu i uspostavljanje gasnog pražnjenja: Townsend-ova teorija i Teorija strmerskog proboja (Becker et al., 2004a; Raizer, 1991a; Turner, 2016). Townsend-ov proboj se obično razmatra u sistemu dve planparalelne elektrode koje su povezane u kolo sa DC napajanjem, kada se povećava primenjeni napon V . Pretpostavlja se da je primenjeno električno polje homogeno, odnosno $E=V/d$, gde je d međuelektrodno rastojanje. Povećavanje električnog polja prouzrokuje porast električne struje u kolu. Nakon što se dostigne određena vrednost napona V_p , dalje, povećanje električnog polja, proizvešće nagli porast struje i prelazak iz nesamostalnog u samostalno pražnjenje. Ako u trenutku proboja struja naglo raste, to znači da gustina struje j teži beskonačnosti ($j \rightarrow \infty$):

$$j = j_0 \frac{e^{\alpha d}}{1 - \gamma(e^{\alpha d} - 1)} \quad (1.1)$$

- j_0 – gustina struje elektrona,
- α - koeficijent jonizacije gasa, definiše broj elektrona koji jedan elektron proizvede na dužini puta od 1 cm krećući se u pravcu spoljašnjeg električnog polja E
- γ - koeficijent sekundarne emisije, zavisi od materijala katode i vrste gasa i uključuje doprinos teških čestica i fotona na izbijanje elektrona iz katode
- $e^{\alpha d}$ – faktor multiplikacije elektrona u međuelektrodnom prostoru.

Odatle sledi uslov za proboj:

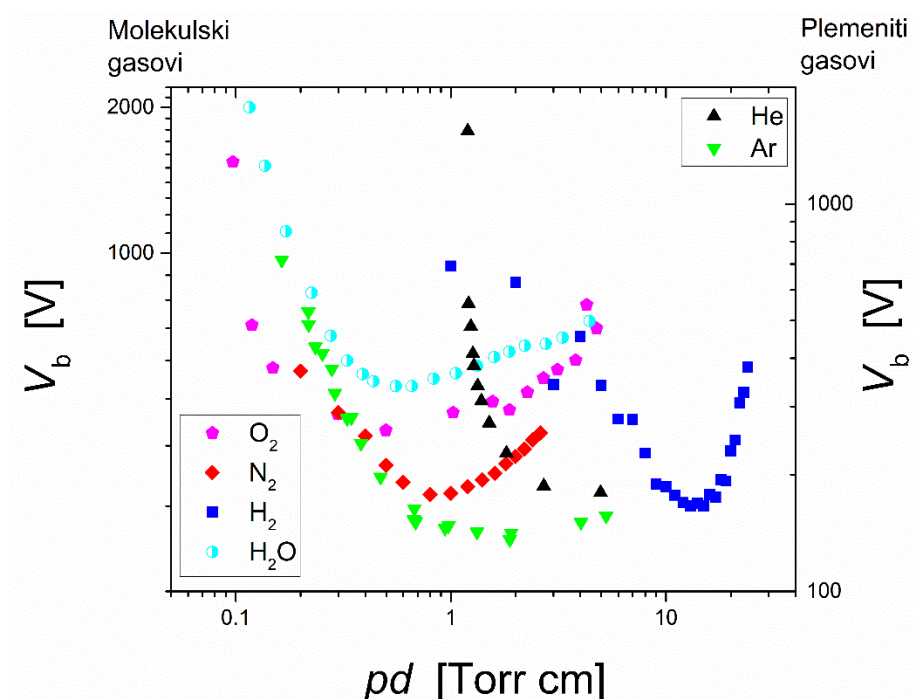
$$1 - \gamma(e^{\alpha d} - 1) = 0, \quad \alpha d = \ln \frac{1+\gamma}{\gamma} \quad (1.2)$$

Empirijsku relaciju koja povezuje napon proboja V_p za dati gas (i materijal katode), pritisak gasa p i rastojanje između elektroda d , $V_p = f(pd)$, pronašao je naučnik Pašen (puno ime Louis Carl Heinrich Friedrich Paschen) 1889. godine (Paschen, 1889). Relacija važi samo na datoj temperaturi, ali može postati univerzalnija ukoliko se umesto pritiska uzme koncentracija čestica gasa N , $V_p = f(Nd)$. Pašenove krive za različite gasove prikazane su na Slici 1.1.

Ove krive opisuju se Pašenovim zakonom:

$$V_p = \frac{Bpd}{\ln(Apd) - \ln\left[\ln\left(\frac{1+\gamma}{\gamma}\right)\right]} \quad (1.3)$$

i mogu se dobiti ukoliko su poznati koeficijent jonizacije gasa α i γ Konstante A i B su karakteristige gasa koji se ispituje. Minimalna vrednost napona proboja i odgovarajuća vrednost proizvoda pd može se naći kada se diferencira izraz (1.3) i izjednači sa nulom.



Slika 1.1.: Pašenove krive (Petrović et al., 2014) – zavisnost probojnog napona od proizvoda pritiska gasa i međuelektrodnog rastojanja za dvoatomske gasove (N_2 , H_2 , O_2), vodenu paru i retke gasove (He, Ar).

Električni proboj u gasu na atmosferskom pritisku predmet je proučavanja kako u fundamentalnim istraživanjima tako i u različitim oblastima primene. Fizički fenomeni koji se odvijaju u ranoj fazi proboja na atmosferskom pritisku imaju dosta sličnosti sa onima u inicijalnoj fazi pražnjenja na niskom pritisku. Počinju kada jedan inicijalni elektron pod uticajem električnog polja stvara rastuću lavinu elektrona.

Pašenove krive u vazduhu na atmosferskom pritisku posebno su proučavane u oblasti visokonaponskog inženjerstva, zbog izolacionih osobina vazduha (Billen et al., 2020; Chachereau & Franck, 2017). Pored toga i druge osobine vazduha utiču na probojni napon. Pokazano je da vlažnost vazduha smanjuje probojni napon koji dostiže minimum na vrednosti oko 1%, a zatim ponovo raste (Becker et al., 2004; Lowke, 1992), kao i da je izražena frekventna zavisnost probojnog napona sa minimumom na oko 1 MHz (Kunhardt, 2000).

STRIMERSKI MEHANIZAM PROBOJA, STRIMERSKO PRAŽNJENJE

Strimeri su jedan od osnovnih vidova električnog pražnjenja na atmosferskom pritisku. Spadaju u grupu hladnih atmosferskih pražnjenja koja se često generišu plazma mlazovima. Teorija strimerskog proboja koju su predložili Loeb (1954), Raether (1964) i Meek & Craggs (1978), objašnjava fenomen pražnjenja na visokom pritisku (kao što je atmosferski pritisak) i uzima u obzir pojave koje Townsend - ova teorija ne razmatra:

- vremensko kašnjenje električnog proboja i
- efekat prostornog naelektrisanja

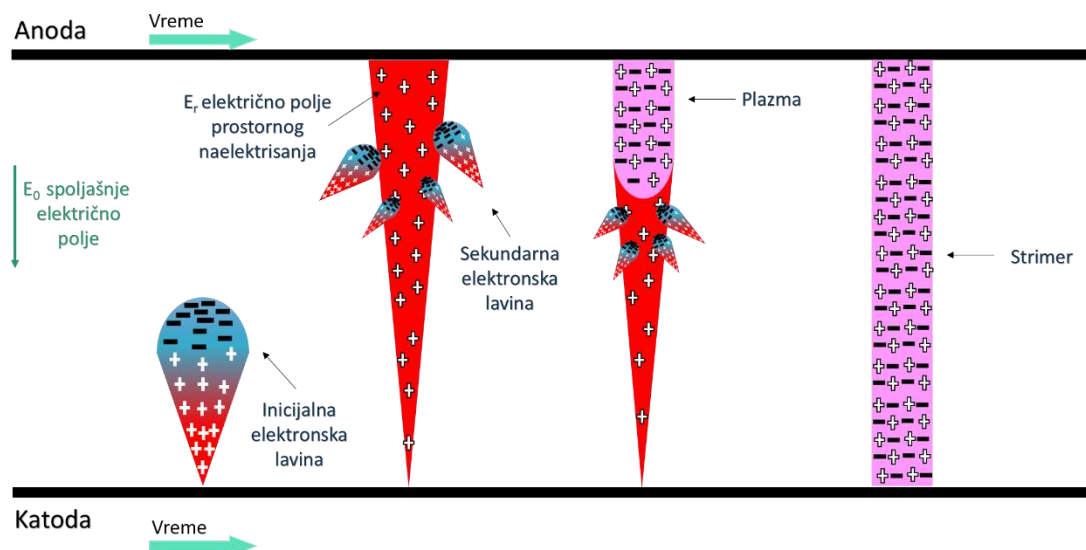
Strimeri su prisutni i kod drugih vrsta plazma izvora kao i u prirodi, gde prethode ostalim vidovima pražnjenja kao što su varnica i munja, ukoliko se ne ograniči struja pražnjenja (Nijdam et al., 2020).

Strimer je slabo do umereno jonizovan uzani kanal koji nastaje na atmosferskom pritisku u gasu koji je izložen dejstvu jakog električnog polja. Nakon formiranja se širi ka elektrodama, u jednom ili oba smera istovremeno. U zavisnosti od uslova u sistemu, električnog polja i sredina u kojoj pražnjenje nastaje, formiraju se složene razgranate strukture u vidu radijalnih filamenata. (Bruggeman et al., 2017; Foster, 2017; Nijdam et al., 2020; Raizer, 1991a). Energija brzo-napredujućeg strimera se uglavnom troši na ekscitaciju radnog gasa (Reuter et al., 2018; Sobota et al., 2014). U slučaju plazma mlazova ovo je demonstrirano kroz prostiranje pulsnih strimera na atmosferskom pritisku (engleski Pulsed Atmospheric pressure Plasma Streamer- PAPS), takozvanih „plazma metaka“ (Maletić et al., 2017; Puač et al., 2012; Robert et al., 2012; Winter et al., 2015).

Prema mestu nastajanja strimeri se dele na pozitivne i negativne. Pozitivni strimeri (anodni) se razvijaju od anode ka katodi (eng. cathode-directed), a negativni (katodni) od katode ka anodi (eng. anode-directed).

Mehanizam razvoja pozitivnog strimera (Leob i Meek)

U sistemu sa dve ravne paralelne elektrode između kojih je gas na atmosferskom pritisku, primenjeno je spoljašnje električno polje E_0 (videti sliku 1.2). Kada je napon primenjen na međuelektrodnu oblast dovoljno visok, tada elektroni napuštaju katodu, formirano električno polje ih ubrzava i dolazi do sudara sa molekulima gasa koji se pri tome ekscituju i jonizuju. Stvorena primarna lavina nije homogena u prostoru zbog smanjenog slobodnog puta čestica na atmosferskom pritisku. To se može predstaviti kao elektronski oblak između elektroda, iza koga ostaje prostorno naelektrisanje pozitivnih jona. Pošto elektronska lavina rezultira tako velikom promenom prostornog naelektrisanja zahvaljujući razlici u pokretljivosti elektrona i jona, lokalno električno polje unutar elektronskog oblaka je veoma izobličeno.



Slika 1.2: Mehanizam nastajanja strimerskog pražnjenja.

U probojnom električnom polju, elektroni mogu dostići brzinu od 2×10^7 cm/s, dok je brzina kretanja pozitivnih jona između elektroda oko 2×10^5 cm/s, što znači da su pozitivni joni stacionarni u poređenju sa elektronima (Raizer, 1991). Pobuđeni molekuli iz lavine emituju fotone i dolazi do efekta fotojonizacije u gasu, pri čemu elektroni nastali ovim putem, iniciraju sekundarne lavine. One privučene pozitivnim jonima iz primarne lavine prave kvazineutralnu plazmu, formirajući pozitivan strimer kada front jonizacionog talasa inicijalne lavine elektrona stigne na anodu. Lokalno električno polje je veoma povećano na glavi strimera usled formiranja prostornog naelektrisanja. Strimersko pražnjenje se prostire u vidu nelinearnih jonizacionih talasa koji se brzo kreću i čiji se frontovi šire, što dovodi do samoproširujućeg pražnjenja.

Mehanizam nastajanja negativnog strimera

Situacija kada primarna lavina mora da pređe čitavu međuelektrodnou oblast da bi došlo do formiranja strimera nastaje kada je primenjeni napon jednak probojnom naponu. U slučaju kada je primenjeni napon veći od probojnog napona, jonizacioni stepen na frontu elektronske lavine je dovoljan da bi nastao strimer. Nakon formiranja, strimer se samostalno razvija ka anodi. Usled zračenja pobuđenih molekula, dolazi do fotojonizacije i stvaranja sekundarnih lavina. Drift elektrona je u ovom slučaju je istog smera kao smer kretanja glave strimera. Elektroni iz glave strimera dolaze u oblast pozitivnog naelektrisanja koji ostaje iza sekundarnih lavina, što dovodi do formiranja plazme. Zbog toga je brzina širenja strimera u ovom slučaju mnogo veća od brzine širenja elektronske lavine (Raizer, 1991).

Meek-ov Kriterijum za stvaranje strimera

Prema Meek-ovoj teoriji razvoja strimera, glavni uzroci održavanja ovog tipa samostalnog pražnjenja su jonizacija elektronskim udarom i fotojonizacija u međuelektrodnom prostoru. Teorija ističe uticaj prostornog naelektrisanja na izobličenje električnog polja (Raether, 1964; Loeb, 1954; Meek & Craggs, 1978). Formiranje strimera direktno zavisi od naelektrisanja u frontu elektronske lavine. Kada se prostorno naelektrisanje poveća i izobličenje električnog polja se u određenoj meri pojača, u prostoru će se desiti dovoljna fotojonizacija koja će dovesti do formiranja strimera. Količina prostornog naelektrisanja i prostorne fotojonizacije su zavisni od gustine molekula gasa, odnosno pritiska gasa.

Meek-ov kriterijum za prelaz elektronske lavine u strimer je da se intenzitet radijalnog električnog polja prostornog naelektrisanja E_r izjednači sa primenjenim poljem E_0 :

$$E_r = KE_0, K \approx 1 \quad (1.4)$$

Kriterijum $E_r = E_0$ za formiranje strimera nakon što elektronska lavina pređe celo rastojanje između elektroda, može se iskoristiti za računanje minimalnog napona proboja i za vazduh on glasi:

$$\alpha d + \ln \frac{\alpha}{P} = 14,5 + \ln \frac{E}{P} + \frac{1}{2} \ln \frac{d}{P} \quad (1.5)$$

gde je:

- d – rastojanje između elektroda,
- P – pritisak gasa u mmHg,

NERAVNOTEŽNA PRAŽNENJA U KONTAKTU SA METOM

Kada neravnotežno pražnjenje na atmosferskom pritisku dolazi u kontakt sa okolnim vazduhom, može dovesti do stvaranja reaktivnih vrsta kiseonika i azota, emitovanja ultraljubičastog zračenja i u nekim slučajevima do razvijanja udarnih talasa, pri čemu je moguće ostvariti kontakt između aktivne zapremine formirane plazme i uzorka. Ovo između ostalog čini ove plazme pogodnim za kontakt sa tečnostima, odnosno tretmane tečnosti i prečišćavanje vode. Ovaj pravac istraživanja u oblasti fizike neravnotežnih pražnjenja vezan za osobine i primene neravnotežnih plazmi u tretmanima tečnih uzoraka postao je vrlo aktuelan u poslednjoj deceniji (Barjasteh et al., 2021; P J Bruggeman et al., 2016; P Lukes et al., 2014).

Jedna vrsta izvora neravnotežne plazme su plazma mlazevi koji rade na atmosferskom pritisku. Koncentracije elektrona u neravnotežnim plazma mlazevima su tipično ispod 10^{19} m^{-3} (Z. Machala et al., 2009). Plazma mlazevi na atmosferskom pritisku imaju dugu istoriju od preko pedeset godina, a istraživanja vezana za ove plazma izvore su intenzivirana u poslednjih dvadeset godina. Tokom ovog perioda, razvijen je njihov dizajn i mehanizam generacije plazme, a jedan od glavnih pokretača istraživanja bilo je njihovo prilagođavanje za primenu u različitim oblastima (Winter et al., 2015) kao što su biomedicina, tretmani vode i poljoprivreda (Foster, 2017; Puač et al., 2018; Weltmann et al., 2010).

Postoji mnogo različitih klasifikacija plazma mlazeva na atmosferskom pritisku. One su najčešće zasnovane na:

- geometriji pražnjenja, raspodeli elektroda ili konfiguraciji polja,
- vrsti generisane plazme (tinjavo pražnjenje, korona, strimer, luk, varnica itd.),
- frekvenciji pobude (AC, DC, RF, MW) ili načinu pobude (kontinualni signal, impulsna pobuda),
- tipu radnog gasa ili smeši gasova koji se puštaju (produvavaju) kroz pražnjenje.

Plazma mlazevi na atmosferskom pritisku se konstruišu u različitim geometrijama i konfiguraciji elektroda. Konstruišu se kao dielektrična barijerna pražnjenja u dielektričnoj cevčici sa spoljašnjim prstenastim elektrodama i uzemljenom elektrodom. Takođe se prave i u konfiguraciji sa centralnom elektrodom, koja može biti i zašiljena na vrhu ili šuplja, sa ili bez dielektrične cevi oko nje. U tom slučaju druga elektroda može biti uzorak koji se tretira, koji je dielektrik ili je od provodnog materijala. Glavna razlika između ovih plazma mlazeva je orijentacija električnog polja u odnosu na pravac protoka gasa. Ovo između ostalog utiče na to koliko će se plazma prostirati izvan cevi plazma mlaza kao i na generisanje reaktivnih vrsta. U zavisnosti od geometrije elektroda, frekvencije pobude, napajanja i radnog gasa razlikovaće se tip generisane plazme i mod pražnjenja, a kod plazma mlazeva najčešće su realizovana strimerska pražnjenja i difuzno (tinjavo) pražnjenje na atmosferskom pritisku.

Uticaj konfiguracije plazma mlazma na osobine pražnjeja prikazan je u mnogobrojnim istraživanjima. Pokazano je da efikasnost procesa prenosa snage iz napajanja u sistem plazma mlaza određuje mod pražnjenja (P. Lu et al., 2017; Sobota et al., 2014; Walsh et al., 2010). Na primer, prikazano je poređenje dva režima pražnjenja stvorena primenom različitih izvora pobude (impulsnog izvora direktne struje i naizmeničnog izvora sinusne pobude) kod plazma mlaza sa jednom elektrodom. Pokazano je da korišćeni izvor pobude određuje struju pražnjenja, dužinu plazma mlaza i temperaturu plazme (Xiong et al., 2010). Dodavanje uzemljene elektrode uzrokuje gubitak snage, ali istovremeno sprečava formiranje pražnjenja unutar uređaja (Sobota et al., 2014).

Dodatna kompleksnost sistema nastaje kada se u sistem plazma mlaza uvede meta koja je u kontaktu sa plazmom, kako bi se plazma mlaz koristio u tretmanima različitih površina. Električne karakteristike plazme i njena reaktivnost utiču na metu, ali istovremeno meta utiče na dinamičke osobine pražnjenja. Mete koje interaguju sa plazma mlazevima mogu se okarakterisati prema njihovim električnim osobinama – električnoj provodljivosti, dielektričnoj propustljivosti i tome da li je njihov električni potencijal plivajući ili pod spoljnom kontrolom napona. Drugi važan parametar je debljina mete, koja je u većini slučajeva reda veličine 1 mm (Viegas et al., 2022).

Pored meta koje su u čvrstom stanju, i tečnosti mogu biti izložene delovanju plazmi na atmosferskom pritisku. Izlaganje vodenih uzoraka pražnjenju na atmosferskom pritisku dovodi do promena u osobinama tečnosti, u nekim slučajevima do smanjenja pH vrednosti, promene električne provodljivosti i redoks potencijala i stvaranja reaktivnih vrsta kiseonika i azota (Bruggeman & Brandenburg, 2013; Machala et al., 2019). U zavisnosti od konfiguracije plazma mlaza, tipa pražnjenja i energije oslobođene u pražnjenju, različite reakcije će se odvijati u gasnoj fazi (plazmi) i u tečnosti (Ng et al., 2021a). Kako bi se bolje razumeo složen odnos između karakteristika izvora plazme, osobina pražnjenja i karakteristika tretirane tečnosti, potrebno je izvršiti detaljnu dijagnostiku plazma mlaza.

1.2 KARAKTERIZACIJA GASNOG PRAŽNJENJA NA ATMOSFERSKOM PRITISKU

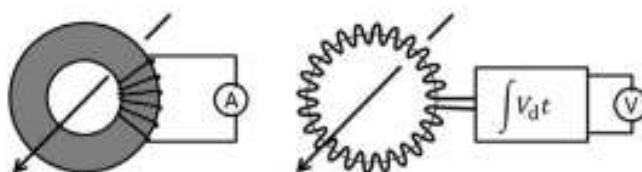
1.2.1 VREMENSKI PROMENLJIVI SIGNALI STRUJE I NAPONA, MERENJE SNAGE

Nakon što multiplikacioni procesi u gasu dovedu do prelaska nesamostalnog električnog pražnjenja u samostalno, mogu se uspostaviti različiti oblici pražnjenja. Kada je električno polje između elektroda homogeno (slučaj planparalelnih elektroda), može nastati tinjavo ili lučno pražnjenje. U sistemima kod kojih je električno polje izrazito nehomogeno (slučaj gde je jedna elektroda u obliku šiljka, a druga je ravna) može se javiti korona, strimer, varnica ili lučno pražnjenje. U zavisnosti od same eksperimentalne postavke i geometrije elektroda mogu se koristiti različiti napajачi. Izvori plazme na atmosferskom pritisku napajaju se korišćenjem visokonaponskog izvora kontinualnog signala visoke frekvencije ili impulsnog signala u opsegu frekvencija od DC do gigaherca. Primenjeni naponi su reda veličine desetina kilovolta, a struje reda veličine miliampera.

Svaki od gore pomenutih tipova pražnjenja karakteriše različita kriva zavisnosti struje pražnjenja od primenjenog napona, tzv. strujno-naponska karakteristika – I - V . Za dobijanje I - V karakteristika potrebno je odrediti vrednosti napona i struje pražnjenja u širokom opsegu radnih parametara za šta se koriste električne sonde.

Dijagnostika brzopromenljivih električnih signala zahteva adekvatnu tehniku merenja. Za merenje vremenski-promenljivih signala čije su frekvencije u opsegu od par stotina kiloherca i dalje se mogu koristiti visokonaponske i strujne sonde.

Vremenski promenljivi naponski signali se snimaju visokonaponskim sondama koja je povezana za osciloskop na kome se beleži vremenska varijacija napona. Vremenski promenljivi strujni signali se mogu dobiti merenjem napona na krajevima elementa sa poznatom impedansom. Za merenje impulsnih i naizmeničnih struja, mogu se koristiti strujni transformatori ili Rogovski navoj, koji su prikazani na slici 1.3.



Slika 1.3: Metode merenja naizmenične struje: strujni transformator (levo) i Rogovski navoj (desno) (Ishikawa, 2016).

Strujna sonda koja je korišćena u okviru ove disertacije koristi hibridnu tehnologiju koja uključuje senzor zasnovan na Holovom efektu (eng. Hall effect) koji meri jednosmernu struju i strujni transformator za merenje naizmenične struje. Time je omogućeno nesmetano merenje struje, bez ubacivanja električnog priključka u kolo plazma mlaza.

METODE MERENJA SNAGE

Parametar koji značajno utiče na interakcije koje će se odvijati u gasoj fazi, ali i samom uzorku koji se tretira, kao i na proizvodnju reaktivnih vrsti jeste snaga koja se oslobodi u pražnjenju. Pored toga, u primenama neravnotežnih plazmi u tretmanima različitih uzoraka utrošena energija (oslobođna snaga u jedinici vremena) je glavni pokazatelj efikasnosti tretmana. Zbog toga mnoge primene zahtevaju da oslobođena snaga bude strogo kontrolisana kako bi se ostvario željeni uticaj tretmana i ne bi došlo do uništavanja mete koja se tretira.

RMS vrednosti i V-I metod

Jedan od najčešće korišćenih metoda za indirektno merenje snage oslobođene u električnom kolu, zasnovan je na snimanju vremenski promenljivih signala napona i struje, $v(t)$ i $i(t)$. Za predstavljanje vrednosti električnih parametara, umesto trenutne snage, uglavnom se koriste koreni kvadrata srednje vrednosti (eng. root mean square - RMS), koji se računaju na sledeći način:

$$V_{rms} = \sqrt{\frac{1}{nT} \int_0^{nT} v^2(t) dt} \quad (1.6)$$

$$I_{rms} = \sqrt{\frac{1}{nT} \int_0^{nT} i^2(t) dt} \quad (1.7)$$

gde je:

- T period oscilovanja signala,
- $\frac{1}{nT} \int_0^{nT} v^2(t) dt$ je srednja vrednost kvadrata napona za n perioda. Dobija se računanjem površine ispod grafika zavisnosti kvadrata napona od vremena, u intervalu od n perioda oscilovanja signala. Analogno važi i za struju $i(t)$.

U V-I metodu, prvo se računaju trenutne snage kao proizvod $v(t)$ i $i(t)$, a zatim se srednje snage dobijaju korišćenjem sledeće formule:

$$P_{mean} = \frac{1}{nT} \int_0^{nT} v(t) \cdot i(t) dt \quad (1.8)$$

Q-V metod

Drugi metod koji se koristi za električnu karakterizaciju i merenje snage zasnovan je na grafiku zavisnosti količine naelektrisanja $q(t)$ od primenjenog visokog napona $v(t)$ odnosno Lissajous-ovim figurama. Za ovaj tip merenja potrebno je u električno kolo ubaciti kondenzator serijski sa plazma mlazom i uzemljenom granom kola, tako da ista količina naelektrisanja koje je formirano u pražnjenju, protiče i kroz uzemljenu granu kola i kondenzator. Merenjem napona na kondenzatoru, poznavajući električni kapacitet uz pretpostavku da je kondenzator idealan (nema parazitsku otpornost), može se izračunati protok naelektrisanja kroz kondenzator. Crtanjem grafika zavisnosti količine naelektrisanja od primenjenog visokog napona, za jedan period signala, dobija se zatvorena kriva linija – Lissajous-ova figura. Površina koja je uokvirena figurom jednaka je energiji koja se oslobodi za jedan period primenjenog naponskog signala (Hoľub, 2012; Lu et al., 2019; Weltmann et al., 2010):

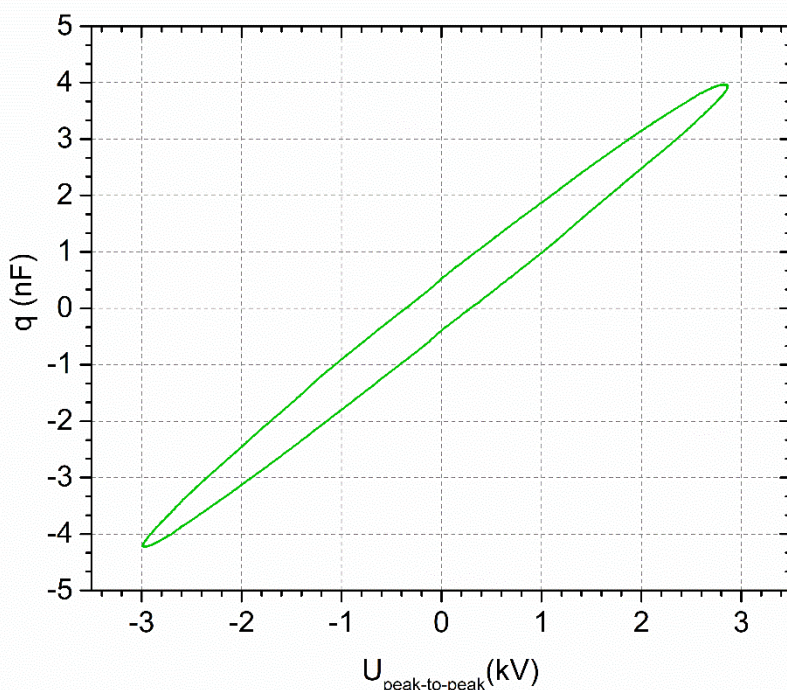
$$E = C \oint v(t) dv_G = \oint v(t) \frac{dQ}{dt} dt = \frac{P_{mean}}{f} \quad (1.9)$$

gde je:

- C kapacitet kondenzatora,
- $v_G(t)$ napon na krajevima kondenzatora,
- $v(t)$ primenjeni visokonaponski signal,
- $f = 1/T$ odgovarajuća frekvencija i
- P_{mean} srednja snaga oslobođena tokom jednog perioda primenjenog naponskog signala.

Lissajous-ova figura se može dobiti direktno na osciloskopu korišćenjem moda XY prikaza (XY display mode), koji omogućava prikaz jedne fizičke veličine (Y signal) koja se meri na jednom kanalu osciloscopa u funkciji druge fizičke veličine (X signal) koja se istovremeno meri na drugom kanalu. U slučaju merenja snage korišćenjem Q-V metoda, potrebno je da visoki napon bude na x osi, a napon na krajevima mernog kondenzatora na y osi. Grafik zavisnosti naelektrisanja koje prođe kroz plazmu i merni kondenzator prikazan je na Slici 1.4

Da bi se osiguralo da je uticaj kondenzatora na ceo sistem minimalan, kapacitivnost kondenzatora se bira tako da bude velika u odnosu na ekvivalentnu kapacitivnost plazma mlaza, međuelektrodnog prostora koji je ispunjen gasom i mete koja se tretira. Razlika treba da bude barem jedan red veličine. Drugo važno svojstvo jeste vrsta odabranog kondenzatora. Poželjno je izabrati keramički kondenzator koji ima male parazitne gubitke i tačnost u merenju naelektrisanja (Peeters & Butterworth, 2019). Dodatna prednost je što keramički kondenzatori nisu polarizovani i mogu se bezbedno spojiti u kolo naizmenične struje.



Slika 1.4: Grafik zavisnosti količine naelektrisanja koje prođe kroz merni kondenzator - q od primenjenog visokog napona U .

1.2.2 OPTIČKA EMISIONA SPEKTROSKOPIJA I SNIMANJE KAMEROM

Optička emisiona spektroskopija je neinvazivna pasivna dijagnostička metoda koja se često koristi zbog jednostavne tehnike merenja i zasnovana je na detekciji fotona koje emituju ekscitovani atomi i molekuli pri elektronskom prelazu u niže vezano stanje. Dobijeni emisioni spektar osim informacije koje su vrste detektovane u plazmi, daje podatke i o opsegu talasnih dužina iz kog potiče zračenje, apsolutne i relativne intenzitete emitovanih linija, dok je širina spektralne linije povezana sa različitim mehanizmima širenja. Zbog toga je ovaj metod pogodan za dijagnostiku različitih fizičkih osobina plazme kao što su koncentracije i temperature čestica u gasu, proučavanje međusobnih interakcija između čestica, ili interakcija čestica i električnog polja plazme. Moguće je proučavanje i dinamičkih procesa u plazmi ukoliko se koriste spoljašnje jedinice za sinhronizaciju signala.

Procesi emisije i apsorpcije zračenja su prelazi između kvantnih stanja; odnosno ponašanje čestica je kontrolisano pravilima kvantne mehanike. Radijativni procesi koji se odnose na prelaze elektrona dele se na:

- prelaze između gornjih i donjih vezanih stanja (vezani prelazi),
- prelaze između vezanih i slobodnih stanja (slobodno-vezani prelazi) i
- slobodni elektroni dobijaju energiju koju gube pozitivno naelektrisane čestice koje emituju fotone (slobodni prelazi, koji se nazivaju zakočno zračenje - Bremsstrahlung).

Obično su za dijagnostiku neravnotežnih tipova pražnjenja najvažniji vezani prelazi. Svi elektroni u višim vezanim stanjima spontano gube energiju emisijom zračenja i dostižu ravnotežno stanje. Širina spektralne linije je obrnuto proporcionalna životnom veku zračenja. Spektralno širenje daje različite vrste informacija o emiterima svetlosti u plazmi. Spontani prelazak iz gornjeg vezanog stanja određen je najužom širinom spektra, koja se naziva prirodno širenje.

Čestice plazme koje emituju zračenje imaju termalnu komponentu kretanja, čija je brzina obično data Maksvelovom raspodelom. Usled termalnog kretanja dolazi do Doplerovog širenja linije, čija je širina na polovini maksimuma (eng. full width half maximum - FWHM) data analitičkim izrazom:

$$\Delta\lambda = 7,16 \cdot 10^{-7} \lambda_0 \sqrt{\frac{T}{m}} \quad (1.10)$$

koji predstavlja Voigt-ov profil. Iz širine linije se može izračunati temperatura T gasa molekulske mase M (Ding et al., 2022; Ishikawa, 2016; Thorne, 1988).

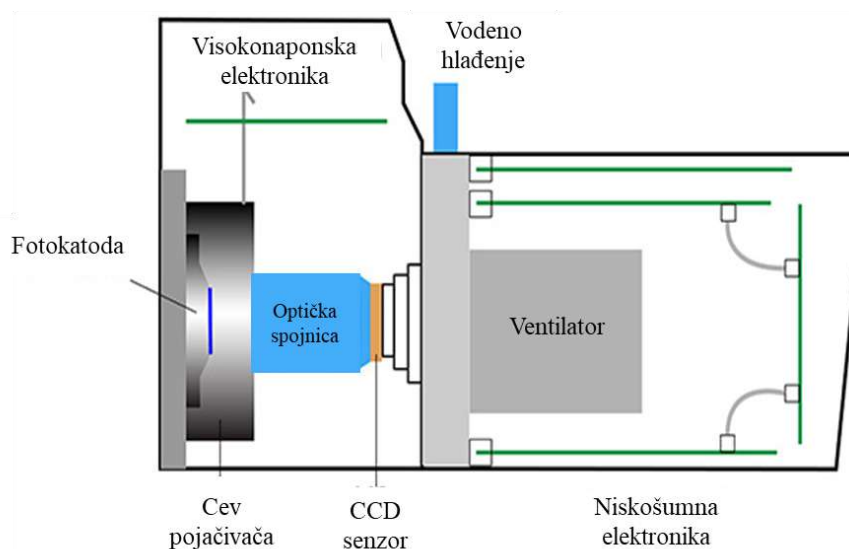
Kod plazmi koje se generišu u ambijentalnim uslovima u vazduhu, populacija unutrašnjih energetskih stanja odstupa od Bolcmanove raspodele. Ovo se odnosi na raspodele elektronskih i vibracionih nivoa, dok se za rotacione nivoe može uzeti da prate Bolcmanovu raspodelu. Njihova vremena života na atmosferskom pritisku su zbog sudarnih procesa značajno kraća od tipičnog vremena prelaska rotacione u translacionu energiju (brzi su procesi relaksacije ovih stanja). Stoga se temperatura gasa može aproksimirati temperaturom rotacionih stanja (Zhu et al., 2008) koja se određuju iz odnosa intenziteta rotacionih spektralnih linija. Mogu se koristiti prelazi O_2 , N_2 , N_2^+ , NO i OH , a najčešće se koriste spektri OH radikala, prelaz $A^2\Sigma-X^2\Pi$ 0,0, i N_2 drugi pozitivni sistem, prelazi ($C^3\Pi_u-X^3\Sigma_g$), kod kojih je pronađeno najbolje slaganje između eksperimentalnih podataka i simulacija (Becker et al., 2004).

Postoje dva osnovna ograničenja ovog metoda (Qing Xiong, 2019). Prvi je što zavisi od mehanizama nastajanja pobuđenih rotacionih stanja molekula. Ukoliko procesi prelaska rotacione u translacionu energiju nisu dovoljno brzi da termalizuju sve rotacione nivoe, dobijena temperatura gasa će biti veća od stvarne temperature zbog prenaseljenosti viših rotacionih stanja. Ovakav slučaj se javlja kod pražnjenja u kontaktu sa vodom kada se u metodi koriste prelazi sa OH ($A^2\Sigma^+$) ili u argonskom pražnjenju ako se koriste prelazi iz stanja N_2 ($C^3\Pi_u$).

1.2.3. PROSTORNI PROFILI EMISIJE ZRAČENJA IZ PLAZME

Jedna od najčešće korišćenih neinvazivnih optičkih metoda koja služi za proučavanje morfologije plazme i dinamičkih procesa, kao što su prostorno i vremenski razloženi razvoj pražnjenja, zasnovana je na korišćenju iCCD kamera. Karakteristike ovih detekcionih uređaja, osetljivost i ekspozicija, moraju da omoguće snimanje veoma slabih svetlosnih signala i snimanje brzih procesa u plazmi. Zbog toga oni osim sistema za detekciju sadrže i pojačavač signala i čip za skladištenje i integraciju signala na samom uređaju.

ICCD kamera (engl. – Intensified Charge Coupled Device) je optički detekcioni uređaj čiji su osnovni delovi - pojačavač signala i CCD senzor, prikazani na Slici 1.5. Pored toga, uređaj sadrži sistem za hlađenje (Peltijev hladnjak) radi smanjenja šuma i elektroniku za konverziju snimljenog signala u digitalni oblik.

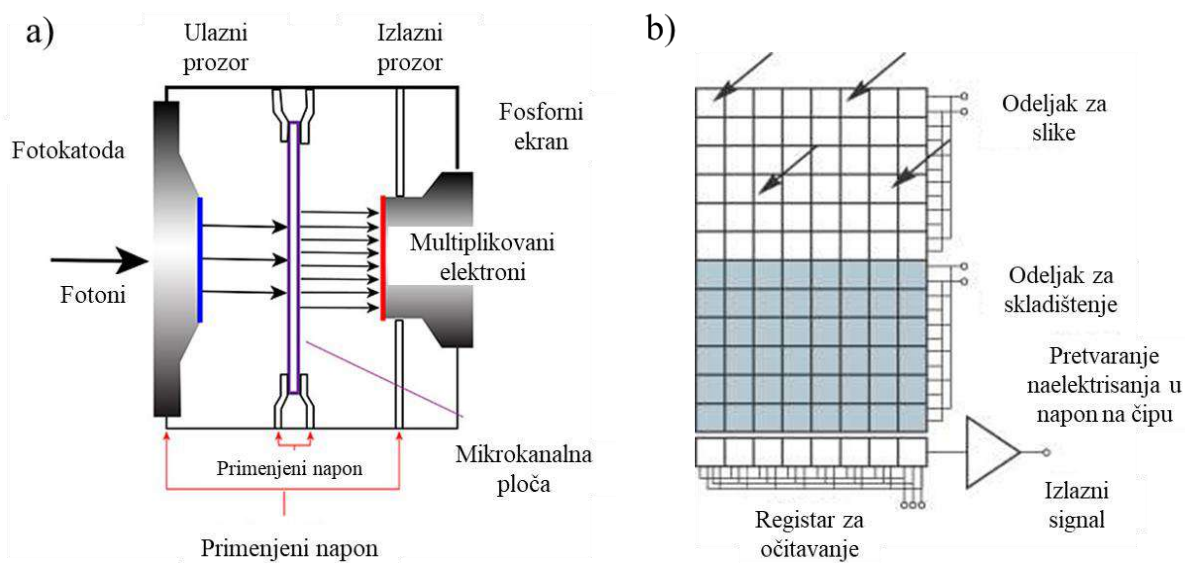


Slika 1.5: Shematski prikaz komponentata kamere, bočni presek kamere.

Pojačavač signala (Slika 1.5) ima dvostruku ulogu u sistemu. Njegova osnovna funkcija je da ulazni signal fotona koji padaju na fotokatodu pretvori u elektrone i primenom električnog polja ih usmeri ka mikrokanalnoj ploči u kojoj dolazi do njihove multiplikacije. Nakon procesa multiplikacije, elektroni se električnim poljem usmeravaju do fosfnog ekrana, gde se elektronski signal ponovo pretvara u fotone. Druga uloga pojačavača je obezbeđivanje vrlo kratkih ekspozicija, pri čemu se primenjenim naponom kontroliše otvaranje i zatvaranje prolaza elektrona ka mikrokanalnoj ploči.

Fotoni nastali na fosornoj ploči zatim dovode na CCD senzor, koji se sastoji od matrice fotoosetljivih dioda. Svaka ćelija matrice predstavlja jedan piksel. U diodama nastaju fotoelektroni čiji je signal srazmeran intenzitetu upadne svetlosti za dati piksel. Ispod redova osvetljenih piksela (Slika 1.6) nalaze se redovi zaklonjenih ćelija u koje se signal skladišti. Signal potom prolazi kroz pojačavač i analogno-digitalni pretvarač, a na izlazu se dobija digitalni broj.

Optička metoda slikanja plazma mlaza u kontaktu sa dielektričnom ili tečnom metom do sada je korišćena za snimanje formiranja pražnjenja i stvaranje reaktivnih vrsta u gasnoj fazi u različitim elektrodnim konfiguracijama. Kod plazma mlaza u konfiguraciji sa centralnom napajanom elektrodom i dielektričnom ravnom elektrodom, proučavan je uticaj polariteta primenjenog električnog polja na formiranje pražnjeja u kontaktu sa staklenom metom (Guaitella & Sobota, 2015) i ispitivan je uticaj vrste dielektrične elektrode (staklo ili voda) na temperaturu gasa (Hofmann et al., 2014). Dielektrična meta korišćena je i kod snimanja jonizacionih talasa proizvedenih DBD plazma mlazom sa koaksijalnim prstenastim elektrodama (Cosimi et al., 2022), gde je ispitivan uticaj tipa dielektrične mete (staklo ili voda) na prostorno-vremensku raspodelu reaktivnih vrsta (He^* , O^* , OH^* , N_2^*) (Gerling et al., 2015).



Slika 1.6: a) Pojačavač signala u iCCD kameri i b) CCD senzor.

U slučaju konfiguracije sa tečnom metom, izučavan je uticaj provodnosti mete, protoka radnog gasa i jačine primenjenog električnog polja na interakciju plazme i tečnosti (Kovačević et al., 2018), uticaj polariteta električnog polja na formiranje OH radikala i drugih reaktivnih vrsta (O , N_2 , N_2^+) (Ng et al., 2021b), kao i uticaj potencijala tečne mete (plivajući potencijal, sa spoljnom kontrolom napona ili uzemljena) na dinamiku pražnjenja (Slikboer & Walsh, 2021). Osim za snimanje pražnjenja u gasnoj fazi, ova metoda je korišćena i u slučaju korona pražnjenja u vodi gde je praćeno stvaranje H_α linije i formiranje mehurića u vodi (Grosse et al., 2021; Pongrač et al., 2018).

1.2.4 VREMENSKA EVOLUCIJA STRIMERA U KONTAKTU SA TEČNOŠĆU

Brze kamere (eng. High-Speed cameras, Fast framing cameras) su u mogućnosti da snimaju video zapise veoma velikom brzinom, tj. sa velikim brojem kadrova (eng. frame) u jednoj sekundi usled čega se dobijaju izuzetno detaljni snimci događaja koji se dešavaju u vremenskom okviru od reda veličine μ s do nekoliko ns. Dizajnirane su tako da snimaju niz uzastopnih slika posmatranog događaja koristeći pri tome napredne senzore sa velikom brzinom očitavanja i brzom obradom podataka. Ovakve kamere u osnovi rade isto što i iCCD kamere, ali umesto jednog pojačavača i jednog CCD senzora sadrže veći broj ovih elemenata, tako da su u mogućnosti da obrađuju i skladište više slika istovremeno.

Kada ulazni snop svetlosti prođe kroz sočivo kamere, prolazi kroz optički razdelnik snopa (eng. beam splitter) i deli se na onoliko delova koliko ima CCD senzora u kameri. Na taj način na svaki senzor dolazi ista količina početnog svetlosnog snopa i na svaki senzor se učitava ista slika. Pošto na svaki CCD senzor dolazi samo deo ulaznog snopa, a sa druge strane, da bi se snimili brzi događaji ekspozicija mora da bude veoma kratka - reda veličine nekoliko nanosekundi, ispred svakog CCD senzora postoji po jedan pojačavač. Da se ne bi snimalo više identičnih slika istovremeno (ista slika na svakom CCD senzoru), pojedinačni pojačavači u datom trenutku mogu biti uključeni ili isključeni. Osim toga, može se podesiti vremenski pomak između uključivanja/isključivanja jednog pojačavača u odnosu na drugi (to jest vreme između snimanja dva uzastopna kadra) sa tačnošću reda veličine 1 ps. Takođe, kamera može prilagođavati osveljenost za svaki pojedinačni kadar.

Ograničenje kod ovih kamera je u tome što CCD senzori mogu da snimaju samo jednu ili dve slike istovremeno (u zavisnosti od tipa senzora), a nakon toga moraju da ih prenesu u memoriju. Vreme prenosa traje nekoliko mikrosekundi.

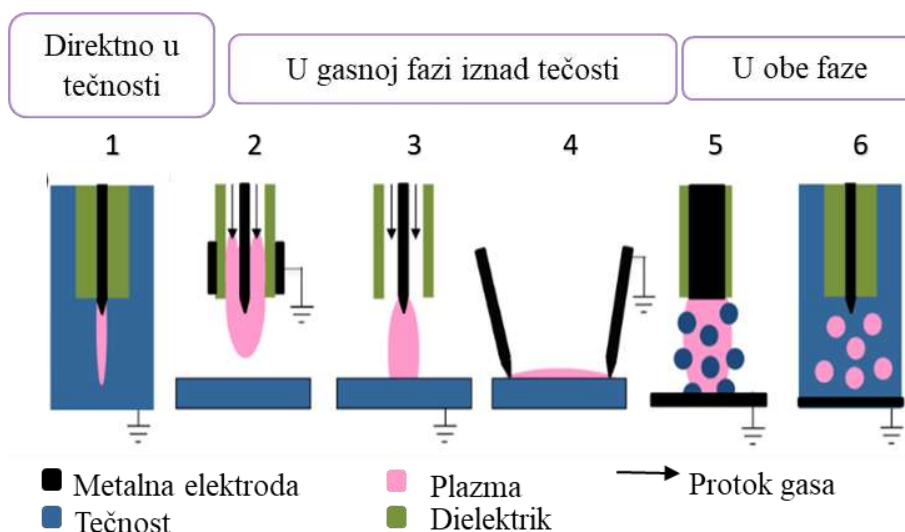
Metoda snimanja formiranja neravnotežnih pražnjenja brzim kamerama, u poslednjih dvadeset godina uglavnom je korišćena za snimanje plazma mlazeva u slobodnom režimu u vazduhu, kada nisu u kontaktu sa metom. U sistemima u kojima plazma interaguje sa vodom proučavana su uglavnom pražnjenja direktno u vodi ili u mehurićima u vodi (Asimakoulas et al., 2020; Kozakova et al., 2017; P. Xiao & Staack, 2014). Mali je broj istraživanja koja proučavaju pražnjenje u gasnoj fazi iznad vode u samom vazduhu (Marinova et al., 2023) ili u aerosolima (Janda et al., 2021).

1.3 PRIMENE PRAŽNJENJA U TRETMANIMA TEČNIH UZORAKA

1.3.1 REGIJE INTERAKCIJE PLAZME I TEČNOSTI

Dovođenje plazme u kontakt sa tečnošću može se ostvariti kroz različite geometrije reaktora zasnovanih na različitim principima rada i vrstama pražnjenja koja se formiraju (videti Sliku 1.10). Na osnovu međusobnog odnosa između faza (gasa, plazme i tečnosti) električna pražnjenja u kontaktu sa tečnostima se mogu podeliti u tri glavne grupe (P. Bruggeman & Leys, 2009):

- 1) električna pražnjenja u tečnosti,
- 2) električna pražnjenja uspostavljena u gasu iznad tečnosti - sa tečnošću kao elektrodom i
- 3) pražnjenja u obe faze:
 - a. u mehurićima u tečnosti
 - b. u aerosolima

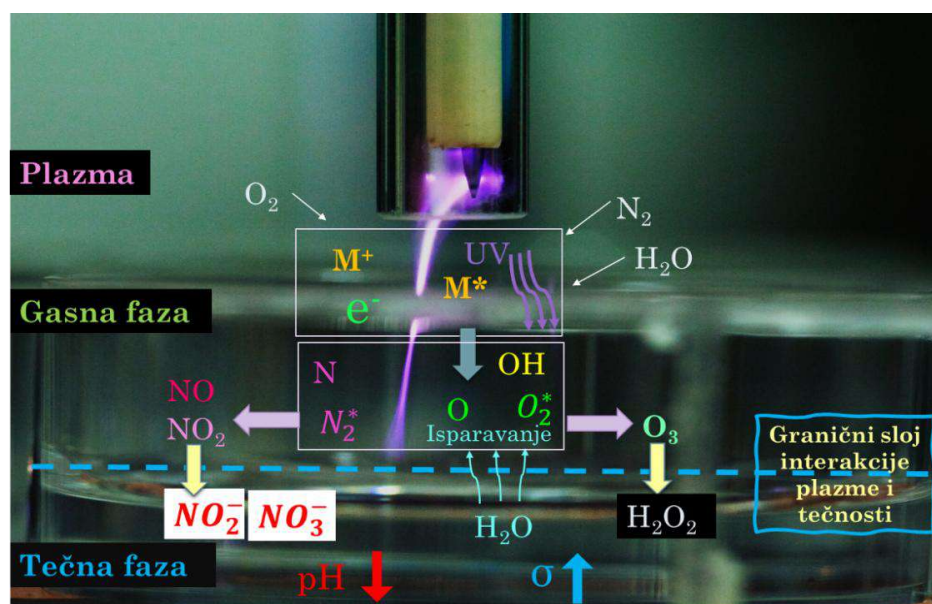


Slika 1.10: Električna pražnjenja u sistemima gde plazma interaguje sa tečnošću (prema (P J Bruggeman et al., 2016; X Lu et al., 2012): 1- pražnjenje direktno u tečnosti, pražnjenje u gasnoj fazi 2- iznad tečnosti i 3- u kontaktu sa tečnošću, 4- pražnjenje u konfiguraciji dve šiljaste elektrode, na površini tečnosti, 5- pražnjenje u aerosolima u gasnoj fazi i 6- pražnjenje u mehurićima u tečnosti.

Prednost geometrije gde se pražnjenje inicira u gasnoj fazi iznad tečnosti je u nižem naponu potrebnom za ostvarivanje proboja u poređenju sa pražnjenjem koje se formira direktno u tečnosti. Osobine ovih pražnjenja mogu biti različite u zavisnosti od geometrije elektroda. Jedna od češćih konfiguracija izvora koji se koriste za generisanje pražnjenja u gasu iznad tečnosti sastoji se od jednog ili više metalnih šiljaka na koje se dovodi signal visokog napona. Ovo je u tom slučaju napajana elektroda dok je pražnjenje obično uzemljeno preko tečnosti koja predstavlja uzemljenu (tečnu) elektrodu. Otpornost tečne elektrode je obično velika pa predstavlja jedan od mehanizama za ograničavanje struje pražnjenja i ima ulogu da stabilise pražnjenje. Struja pražnjenja se transportuje kroz tečnu elektrodu jonima koji imaju mnogo manju pokretljivost od elektrona u metalnim elektrodama. Pored toga voda i druge tečnosti imaju značajno manji sekundarni koeficijent emisije elektrona nego većina metala pa pražnjenje funkcioniše dominatno zahvaljujući zapremiskim procesima.

Na graničnom sloju između gasne i tečne faze dolazi do hemijskih reakcija u kojima učestvuju elektroni iz plazme (videti Sliku 1.11), pri čemu dolazi do formiranja reaktivnih hemijskih vrsta (npr. $\bullet\text{OH}$, O , N_2 , N_2^+ , NO) kao i difuzije vrsta proizvedenih u gasnoj fazi, u tečnu fazu (npr. O_2^- , O^- , O^+ , $\text{NO}_2\bullet$) (Bogaerts et al., 2010; Lukes et al., 2014; Norberg et al., 2014). Istraživanja su pokazala da plazma mlaz ima veliki potencijal u prenosu visoko reaktivnih vrsta iz gasne faze u tečnu fazu zahvaljujući protoku gasa u mlazu (P J Bruggeman et al., 2016; Du et al., 2019). Svaka od ovih vrsti može da pokrene druge hemijske reakcije na graničnom sloju između dve faze ili čak u površinskim slojevima tečnosti sa kojom je plazma u kontaktu, gde reaktivne vrste prežive dovoljno dugo i lako mogu biti apsorbovane.

Uzajamni uticaj između plazma mlaza i površine mete je veoma značajan jer parametri plazme zavise od udaljenosti i materijala mete. Primećeno je da udaljenost mete od vrha napajane elektrode utiče na oblik, dužinu i boju plazme kao i na električne parametre pražnjenja. Pored toga, pokazano je da se provodnost i način uzemljenja mete odražavaju na sastav gasne faze i na koncentraciju merenih reaktivnih vrsta u tečnim uzorcima (Kovačević et al., 2018; Sobota et al., 2019; Zaplotnik et al., 2015).



Slika 1.11: Plazma mlaz sa šiljastom elektrodom u kontaktu sa vodom - oblasti interakcije plazme i tečnosti. Legenda: M^+ i M^* - jon i metastabil čestica radnog gasa, e^- elektroni, UV – fotoni iz plazme, σ – električna provodljivost tečnosti, O_2 , N_2 i H_2O su u sastavu okolnog vazduha.

Druga grupa električnih pražnjenja je ona koja se generiše direktno u tečnostima. Za uspostavljanje ovog tipa pražnjenja potrebno je primeniti jako električno polje reda veličine 10^9 Vm^{-1} između elektroda koje su potopljene u tečnosti. Pražnjenje se generiše impulsnom pobudom i dolazi do formiranja strimera u tečnosti (Bruggeman & Leys, 2009; Bruggeman et al., 2017). Veliki deo snage oslobođene u pražnjenju je u vidu ultraljubičastog zračenja koje je značajno u procesima koji dovode do razgradnje i uništavanja organskih zagađivača rastvorenih u vodi.

Stvaranjem mehurića u tečnosti omogućava se električni proboj na nižim primenjenim naponima u poređnju sa pražnjenjem direktno u tečnosti. Pored toga, prilagođavanjem sastva radnog gasa kontroliše se stvaranje različitih reaktivnih vrsti koje u ovom tipu pražnjenja mogu lakše da difunduju iz gasne u tečnu fazu (Cao et al., 2018).

1.3.2 HEMIJA STRIMERSKOG PRAŽNJENJA U HE I AR U KONTAKTU SA TEČNOŠĆU - STVARANJE REAKTIVNIH VRSTA U GASU I TEČNOSTI

Sastav radnog gasa koji se koristi za uspostavljanje pražnjenja u gasu iznad vode je važan pri razmatranju u kojem se određuje koje će krajnje reaktivne vrste nastati u tretiranoj tečnosti. Prisustvo gasova koji sadrže kisonik i azot (vazduh, O₂, N₂) doprinosi stvaranju RONS. Ukoliko u okolnoj atmosferi postoji vodena para, ona će takođe uticati na sastav i dinamiku plazme, a time na stvaranje reaktivnih vrsta. Stvaranju vodene pare doprinosi i to što je plazma u kontaktu sa tečnom metom.

U reakciji gasne plazme i tečnosti reaktivne vrste uglavnom nastaju ekscitacijom elektronskim udarom ili disocijacijom molekula vazduha i molekula vode. Prvi korak u istraživanju interakcija u gasnoj fazi jeste identifikacija vrsta koje nastaju u plazmi, u mešavini radnog gasa i okolnog vazduha, iznad i u kontaktu sa tečnom fazom. Među brojnim azotnim i kiseoničnim vrstama koje se formiraju u ovoj zoni, dva za primene izuzetno važna radikala su - hidroksil OH i azot monoksid NO.

U gasnoj fazi OH radikal nastaje disocijacijom molekula vode u reakciji sa slobodnim elektronima ili metastabilom Ar^{*}, He^{*}, N₂^{*} (u jednačini 1.12 označeni sa M^{*}) (Nikiforov et al., 2011):



Naelektrisane čestice iz plazme mogu dovesti do jonizacije molekula vode,



koji u reakciji sa drugim molekulom vode stvara OH i hidronijum jona H₃O⁺:



Kada se u gasnoj smeši koristi kiseonik (ili vazduh koji ga sadrži), disocijacijom molekula kiseonika elektronskim udarom nastaje atom kiseonika koji kroz reakciju sa molekulom vode (Petr Lukes & Locke, 2005), takođe doprinosi stvaranju OH radikala:



Prisustvo azotnih jedinjenja u oblasti interakcije plazme i tečnosti pokreće kompleksne hemijske procese u kojima oni reaguju sa primarnim vrstama nastalim u gasnoj fazi. U međusobnoj reakciji između atoma i molekula azota i kiseonika i u reakciji sa OH radikalom, dolazi do nastanka drugog važnog radikala – azot-monoksida NO (Turner, 2016):



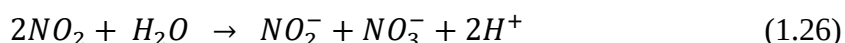
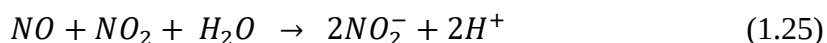
Stvorene sekundarne reaktivne vrse nastavljaju dalje učešće u reakcijama koje nisu ograničene na usku oblast interakcije plazme i tečnosti, već nastavljaju da se odvijaju u celokupnoj zapremini tečne faze i direktno zavisi od rastvorljivosti reaktivnih vrsta u vodi. Rastvorljivost gasovitih reaktivnih vrsta u tečnostima u uslovima ravnoteže data je konstantom Henrijevog zakona (Henry, 1803; Sander, 2015):

$$H = \frac{c_t}{p} \quad (1.23)$$

gde je:

- c_t – koncentracija gasa koji je rastvoren u tečnosti
- p – parcijalni pritisak tog gasa u gasnoj fazi iznad površine tečnosti.

Izlaganje vodenih uzoraka netermalnoj plazmi dovodi do izmena osobina vode, stvarajući pri određenim uslovima tretmana kiselu sredinu, menjajući provodljivost i redoks potencijal i stvarajući sekundarne reaktivne vrste kiseonika i azota u vodi. Kada gasovite vrste dospeju u tečnost, one nastavljaju da učestvuju u reakcijama sa vodom, ali i sa jedinjenjima rastvorenim u vodi. Reaktivne vrste azota nastale u gasnoj fazi dovode do zakišeljavanja tečne faze i smanjenja pH vrednosti kroz reakcije stvaranja nitrita, nitrata, njihovih jona i kiselina (Machala et al., 2013):



Azot-monoksid je jedinjenje čije je nastajanje u endotermnim reakcijama favorizovano u impulsnom korona pražnjenju i strimerskom pražnjenju. Njegovo prisustvo u gasnoj fazi objašnjava smanjenje pH vrednosti tečnosti kroz učestvovanje u reakcijama koje dovode do stvaranja azotne kiseline. Doprinosi oksidacionim efektima i degradaciji zagađivača kroz reakcije formiranja azotnih derivata aromatičnih jedinjenja (Brisset & Pawlat, 2016).

Kroz reakcije u kojima učestvuje azot-monoksid može nastati kratkoživeći radikal peroksinitrit $ONOO^-$:



jedinjenje koje se zbog svojih elektrohemijskih osobina smatra ključnim u rastvorima organskih jedinjenja. Pošto je jako oksidativno sredstvo može interagovati sa biološkim molekulima kada doovodi do inaktivacije bakterija.

Vodonik peroksid H_2O_2 se formira u oblasti interakcije plazme i tečnosti. Ovo se dešava kod manjih izvora plazme, gde se tretiraju manje zapremine tečnosti, a postoji velika nehomogenost plazme (takvi su sistemi u kojima se formira strimerski kanal) (Gorbanev et al., 2016; Locke, 2012). Može nastati u vodi rekombinacijom OH radikala:



To je stabilno jedinjenje koje slabo interaguje sa organskim zagađivačima u tečnosti u poređenju sa OH radikalom. Vodonik peroksid se može rastvoriti u vodi, gde predstavlja izvor nastajanja OH radikala koji dalje reguje sa organskim zagađivačima.

U brojnim primenama je iskorišćen potencijal niskotemperaturne plazme na atmosferskom pritisku za formiranje hemijski reaktivne sredine pri čemu se ne koriste dodatni hemijski reagensi. Dosadašnja istraživanja u ovoj oblasti su se fokusirala na dizajniranje izvora plazme od kojih se veliki broj pokazao uspešnim u primenama u oblastima medicine i biologije, kod tretmana termički osetljivih i nestabilnih materijala, žive materije i stvaranju specifičnih čestica i hemijski aktivnih vrsta (Graves et al., 2019; Magureanu et al., 2021; Puač et al., 2018). Svaki od ovih izvora se pokazao uspešnim u konkretnoj oblasti primene. Međutim, glavni nedostatak postojećih interdisciplinarnih istraživanja je nedovoljno poznavanje procesa i reakcija na osnovu kojih bi se napravili opšti zaključci o ključnim parametrima plazme koji su doveli do željenih rezultata tretmana. Cilj ovog dela disertacije bio je da se dođe do novih saznanja o procesima koji se odvijaju u gasnom pražnjenju na atmosferskom pritisku, o uticaju osobina plazme na reakcije nastalih reaktivnih vrsta sa tečnošću i o efektima plazme na karakteristike tretirane tečnosti.

1.3.3 TRETMANI ČISTE VODE I PRIMENA NA BIOLOŠKE SISTEME

Najnoviji pravac istraživanja u okviru ove oblasti je u polju tretmana vode plazmom pri čemu se zatim tretirana voda naknadno koristi za tretman biljnog materijala (Puač et al., 2018; Thirumdas et al., 2018). Zbog načina primene ovakvi tretmani se često nazivaju indirektnim tretmanima plazmom, jer su promenjene osobine i reaktivnost tretirane vode neposredna posledica izlaganja hladnoj plazmi (Bradu et al., 2020).

Plazmom aktivirana voda (eng. plasma activated water) - PAV se dobija izlaganjem destilovane/dejonizovane vode ili fiziološkog rastvora električnom pražnjenju u (vlažnom) vazduhu na atmosferskom pritisku u toku određenog vremenskog perioda. Tečnost ostaje aktivirana dug vremenski period (koji se meri mesecima (Tsoukou et al., 2020)) i nakon što se plazma isključi. Mnoga istraživanja zbog jednostavnosti koriste čistu (destilovanu/dejonizovanu vodu), kako bi se izbeglo da materije rastvorene u vodi budu oksidovane i da dođe do stvaranja jona (na primer hloridnih jona koji potiču od NaCl u fiziološkom rastvoru), a koji bi mogli da utiču na hemijske efekte gasnog pražnjenja. Dalja istraživanja su otkrila da prisustvo malih koncentracija unapred rastvorenih supstanci u vodi nisu značajno promenili rezultate dobijene korišćenjem destilovane vode (Brisset & Pawlat, 2016).

Reaktivne vrste kao što su: hidroksilni radikal ($\cdot\text{OH}$), atomski kiseonik (O), ozon (O_3), vodonik peroksid (H_2O_2), nitritni jon (NO_2^-), nitratni jon (NO_3^-) i peroksinitrit (ONOO^-) su biološki relevantni molekuli koji imaju važnu ulogu u metaboličkim procesima u ćelijama biljaka. Zahvaljujući svom hemijskom sastavu plazmom tretirana voda može poslužiti kao složeni hemijski reagens u tretmanima različitih vrsta bioloških sistema. Jedinstvenost plazmom tretirane tečnosti je pokazana u eksperimentima gde je dodavanjem čistih hemikalija u vodu napravljen reagens koji nije doveo do istih rezultata i efekata kao primena vode tretirane plazmom (Zhang et al., 2017).

Ipak, stvaranje plazmom aktivirane tečnosti sa specifičnim, unepred zadatim osobinama i određenim sadržajem reaktivnih čestica je izuzetno zahtevan zadatak. U zavisnosti od tipa primenjenog pražnjenja i oslobođene energije u plazmi, kao i gasne smeše i hemijskog sastava tečne faze, različiti tipovi reakcija će se odvijati u gasnoj fazi (plazmi) i tečnosti. Određene reaktivne vrste stvorene u gasnoj fazi i na granici gasne i tečne faze, mogu da ulaze i rastvaraju se u vodi a pokreću naredne reakcije u tečnoj fazi. Sa druge strane, interakcija na granici gasne i tečne faze utiče na osobine same plazme. Zbog toga međusobna interakcija između oba sistema, plazme i tečnog uzorka, zapravo na kraju određuje osobine aktivirane tečnosti.

U poslednje vreme, inovativnost upotrebe hladne plazme na atmosferskom pritisku se ogleda u raznolikim primenama u medicini i biologiji. Oblast primene plazme u biologiji se brzo razvija i proširuje poslednjih godina zahvaljujući brojnim eksperimentalnim istraživanjima gde je neravnotežna plazma korišćena u direktnim tretmanima biljnih materijala kao što su semena, koren, list, kalus (skup biljnih ćelija) itd. (Adhikari et al., 2020; Puač et al., 2014).

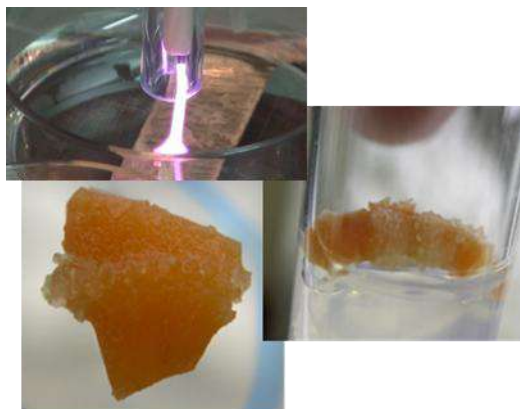
PAV I PRODUKCIJA SEKUNDARNIH METABOLITA U BIOLOŠKIM SISTEMIMA

Biljke u metaboličkim procesima proizvode različita hemijska jedinjenja. Sekundarni metaboliti koji igraju važnu ulogu u samom životu biljaka, u farmaceutskoj, prehrambenoj i kozmetičkoj industriji su iskorišćeni kao lekovi, mirisi, ukusi, hranljive materije, repelenti, boje itd. Same biljke proizvode male količine sekundarnih metabolita i treba ih povećati da bi se ispunile komercijalne potrebe.

Kultura kalusnog tkiva (nediferenciranih biljnih ćelija) je *in vitro* tehnika gde se na tečnom ili čvrstom medijumu (hranljivoj podlozi) u kontrolisanim i sterilnim uslovima umnožava i gaji kalusno tkivo. Ono nastaje od uzorka biljnog tkiva, izolovanog iz gotovo bilo kog dela biljke, čijim postavljanjem na medijum se indukuje stvaranje nediferenciranih ćelija. Ukoliko se fragmenti ovako nastalog tkiva pre faze deobe i diferencijacije premeste na sveži medijum, može se održavati faza aktivnog rasta kalusa. Time se obezbeđuje biljni materijal koji se dalje koristi u istraživanju (Jakovljević, 2023).

Ukoliko se kalusno tkivo tokom kultivacije postavi u odgovarajuće kontrolisane uslove, može doći do uvećane sinteze biološki aktivnih jedinjenja – sekundarnih metabolita na koju ne utiču spoljašnji faktori. To se postiže dodavanjem prekursora -jedinjenje koje prethodi nekom drugom u metaboličkom procesu, ili abiotičkih elicitora - jedinjenja ili fizičke i hemijske pojave koje se primenjuju u cilju indukovanja biljaka za odbranu od stresnih faktora (Maffei et al., 2012). Pored toga, sekundarni metaboliti se u ovom slučaju dobijaju u kraćem vremenskom periodu u odnosu na vreme potrebno za kultivaciju cele biljke.

Primena PAV-a kao novog elicitora za tretman biljnih kultura u *in vitro* sistemima može da se ponudi kao novi pristup za proizvodnju biljnih metabolita, sa obzirom da predstavlja novu metodu koja je energetski efikasna i ekološki prihvatljiva. U ovoj disertaciji ideja je bila da se PAV koristi kao reaktivni medijum za tretman kalusa šargarepe (*Daucus carota* subsp *sativus*, prikazani na Slici 1.12). Šargarepa je veoma značajna povrtarska kultura sa dobro poznatom fiziologijom i sekvenciranim genomom. Predstavlja jednu od prvih biljnih vrsta korišćenih u razvoju tehnika kulture biljnog tkiva, uključujući indukciju i održavanje kalusa u *in vitro* uslovima. Danas se u velikoj meri proizvodi i široko koristi u biologiji i biotehnologiji. Između ostalog, šargarepa je bogata pigmentima kao što su karotenoidi, antocijanini i betacijanini koji deluju kao moćni antioksidansi, imobilizujući štetne slobodne radikale.



Slika 1.12: Tretman vode plazma mlazom i kalusi šargarepe u dobijenoj PAV.

Do sada je u nekoliko istraživanja korišćen direktan tretman kalusa različitih biljnih vrsta sa korona pražnjenjem (Darigh et al., 2023; Tardast et al., 2023), plazma iglom (Puač et al., 2014, Jevremović et al., 2025) i drugim izvorima (Rahnama et al., 2025). Međutim i pored sve većeg interesovanja za primenom plazme u biotehnologiji, u dliteraturi se i dalje se ne može naći više istraživačkih radova na ovu temu. U dostupnoj literaturi pronađen je tek jedan rad u kojem se PAW koristi kao elicitor u kalusnoj kulturi (Fathali Tehrani et al., 2025).

1.3.4 PREČIŠĆAVANJE ZAGAĐENE VODE – DEKONTAMINACIJA PLAZMOM

Dobar kvalitet sveže (slatke) vode (eng. fresh water) je neophodan za dobrobit i zdravlje ljudi, za podršku zdravih vodenih ekosistema i biodiverziteta, i ključan za poljoprivredu i proizvodnju hrane, tri glavne komponente Evropskog zelenog dogovora (eng. European Green Deal) (Evropska komisija, 2019). Uprkos značajnom napretku u smanjenju konvencionalnih zagađivača vode i poboljšanju kvaliteta sveže vode, obim zagađenja iz urbanih tačkastih i netačkastih izvora (Slika 1.13) ostaje važan izazov, posebno u područjima sa nedostatkom vode. Prethodno kontaminirane lokacije i industrijska postrojenja koje upravljaju opasnim hemikalijama, kao što su veoma postojana jedinjenja, zajedno sa poljoprivredom i proizvodnjom hrane (pesticidi, herbicidi, antibiotici itd.) i aktivnosti u domaćinstvu oslobađaju brojne supstance koje pojedinačno ili kombinovano predstavljaju zabrinutost za bezbednost zaliha vode za piće.



Slika 1.13: Izvori zagađenja (industrija poljoprivreda i farmacija) prirodnih voda i plazma kao napredni oksidacioni proces. Delovi slike su fotografije preuzete sa platforme *Unsplash* (autori: Wooldridge, Zilles, Kanesaki, Wicaksono).

Obrada industrijskih otpadnih voda obično se sastoji od tri faze. Primarna je ona u kojoj se mehanički otklanjaju čvrsti materijali i koriste se hemijski aditivi, dok sekundarna faza podrazumeva biološke procese u kojima se biorazgradive organske materije uklanjaju iz vode delovanjem mikroorganizama. U tercijarnim procesima se supstance koje ostanu nakon prve dve faze uklanjaju taloženjem, inverznom osmozom ili dezinfekcijom putem naprednih oksidacionih procesa (NOP) (eng. advanced oxidation processes - AOP). Napredni oksidacioni procesi su se pokazali uspešnim u uklanjanju „trajnih” organskih mikrozagađivača (OMZ) (eng. organic micro pollutant OMP) čije su koncentracije u otpadnim vodama reda veličine od 1 ng/l do 1 µg/l. Kao organski mikrozagađivači su prepoznate različite industrijske organske supstance, farmaceutici, organske boje, pesticidi itd. (Arslan et al., 2017; Cuerda-Correa et al., 2019; Ebele et al., 2017; Garrido-Cardenas et al., 2019; Patel et al., 2019).

Plazme koje stupaju u interakciju sa tečnostima, uključujući čistu vodu i tečnosti koje sadrže rastvorene organske supstance, predmet su aktuelnih istraživanja zato što kontrolom parametara pražnjenja može da se usmerava tok hemijskih reakcija, uz efikasan prenos električne energije u formiranje ciljanih produkata. Iako upotreba ovih sistema postavlja pred istraživače velike naučne i inženjerske izazove, sa druge strane omogućava niz praktičnih primena. Izazovi vezani za primenu ovakve tehnologije postoje jer ona obuhvata složene probleme u oblasti fizike i hemije plazme, ali i hemijskog i elektro inženjersva. U oblasti životne sredine postoje dve osnovne primene: u tretmanu (vlažnog) vazduha i tretmanu tečnosti. Tretmanu tečnosti gasnom plazmom se obavljaju samo na atmosferskom pritisku jer bi na manjim pritiscima došlo do potpunog isparavanja uzorka. Pored toga važna primena netermalnih plazmi je i u tretmanu zemljišta.

Razgradnja organskih zagađivača u otpadnim vodama tretmanom plazmom, ali pri čemu ovaj postupak nije zasnovan na termalnom dejstvu predstavlja novi trend u istraživanjima u ovoj oblasti. Pokazalo se da hladna atmosferska plazma efikasno razgrađuje OMZ i predstavlja obećavajući inovativni NOP, čemu svedoči sve veći broj objavljenih studija u kojima su predloženi različiti uređaji za razgradnju molekula organskog otpada rastvorenog u vodi (Hijosa-Valsero et al., 2014; Kumar et al., 2022; S. Li et al., 2014; Magureanu et al., 2015; Malik, 2010; Topolovec et al., 2022).

Utrošak energije potreban za tretmane umerene ili velike količine tečnosti je jedan od najvažnijih faktora koji utiče na praktičnost upotrebe ove tehnologije. Pored efikasnosti stvaranja aktivnih hemijskih vrsta koje su ključne za uspešnost primena, pre svega reaktivnih vrsta kiseonika ($\bullet\text{OH}$, H_2O_2 , O_3 , O) i azota, važna je i cena napajanja celog sistema i efikasnost pretvaranja ulazne energije u enrgiju gasnog pražnjenja. Proizvodi reakcija koji se stvaraju u sistemu zavise od vrste električnog pražnjenja, energije koja je uneta u sistem i obrazaca po kojima se odvijaju reakcije pri interakciji gasa i tečnosti (Nau-Hix et al., 2021; Vasilev et al., 2024).

UKLANJANJE PESTICIDA IZ VODE TRETMANOM PLAZMOM

Raste zabrinutost zbog efekata pesticida na životnu sredinu, organizme koji nisu ciljani za suzbijanje i zdravlje ljudi. Procenjuje se da se u globalnoj poljoprivrednoj proizvodnji godišnje koristi oko 3,5 miliona tona pesticida. Istovremeno, istraživanja pokazuju da iako upotreba pesticida značajno utiče na prinos poljoprivredne proizvodnje i gotovo su nezamenljivi u savremenoj poljoprivredi, manje od 40% upotrebljene količine dolazi do ciljanih organizama, dok ostatak završava u životnoj sredini (Ni et al., 2024).

Konvencionalne metode tretiranja pesticida, uglavnom su zasnovane na faznom prelazu zagađivača i osim što podrazumevaju relativno visoke troškove primene, mogu da izazovu sekundarno zagađenje kao što je stvaranje mulja (López-Loveira et al., 2017). Pored toga, uklanjanje pesticida iz vode otežava neselektivna upotreba i prisustvo širokog spektra pesticida, zbog čega su potrebne alternativne metode kao što su NOP.

U konkretnom slučaju uklanjanja pesticida rastvorenih u vodi, uloga kombinovanih NOP je da neselektivno oksiduju širok spektar otpornih organskih zagađivača do njihove potpune mineralizacije u ugljen-dioksid, vodu i mineralne soli. Na taj način se u vodi stvaraju vrste koje su sada pogodne za dalju biorazgradnju konvencionalnim biološkim procesima koji dovode do stvaranja biomase, hlorovodonične kiseline, biogasa i vode (Lafi & Al-Qodah, 2006; Quiroz et al., 2011; Ribeiro et al., 2015).

Države članice i politike Evropske unije nastoje da smanje zavisnost od hemijskih pesticida za zaštitu useva kroz dizajn i primenu pristupa koji uključuju ograničenja upotrebe različitih aktivnih supstanci. Kako bi se osigurao najmanji rizik po ljudsko zdravlje i životnu sredinu, podstiče se razvoj takozvanih supstanci niskog rizika kroz nekoliko regulatornih podsticaja u EU (Direktive o vodi za piće, vodi za kupanje i poplavama (EU, 1998, 2000, 2006, 2007)). Međutim, promena prirode niskorizičnih sredstava za zaštitu bilja zahteva povećanje kapaciteta za procenu rizika. Proces odobravanja i autorizacije sredstava za zaštitu bilja mora da ide u korak sa naučnim i tehnološkim razvojem u cilju unapređenja metoda procene novih niskorizičnih sredstava za zaštitu bilja. Identifikacija proizvoda razgradnje pesticida i njihovih puteva raspada je izuzetno zahtevna, a evolucija toksičnosti ovih nusproizvoda je još uvek ograničena na teorijsku analizu i nedostaje dalja eksperimentalna provera (Ni et al., 2024).

U okviru ove disertacije tretman plazmom je korićen za prečišćavanje vode zagađene pesticidima malationom (eng. malathion) i acetamipridom (eng. acetamiprid) - sintetičkim organskim insekticidima koji spadaju u grupu organofosfata, odnosno neonikotinoida, respektivno.

Organofosfati su supstance koje su stvorene da bi zamenile grupu mnogo otpornijih pesticida - hlorisanih ugljovodonika. Malation utiče na centralni nervni sistem i veoma je toksičan za čoveka. To je bezbojna supstanca rastvorljiva u organskim rastvaračima, ali i u vodi, zbog čega ima tendenciju da se infiltrira u izdane (eng. aquifer) i podzemne vode. Ipak zbog sposobnosti da se zadrži u životnoj okolini, smanjuje mogućnost da dospe u podzemne vode (Syafudin et al., 2021).

Neonikotinoidi su insekticidi koji su sa 25% zastupljeni na svetskom tržištu. Ova jedinjenja deluju na centralni nervni sistem insekata i najveći nedostatak je što nisu selektivni, čime negativno utiču na životnu sredinu (Hladik et al., 2018).

Istraživanja upotrebe plazma tehnologije za prečišćavanje vode i biljnih kultura (Gavahian et al., 2021; Mousavi et al., 2016; Sarangapani et al., 2017) zagađenih pesticidima (Cong et al., 2021; Ni et al., 2024; Sarangapani et al., 2016), pokazala su da je uspešnost uklanjanja zagađivača vrlo uspešna i predloženi su njihovi putevi raspada. Ipak, mali je broj istraživanja koja ispituju toksičnost tretirane otpadne vode i njen uticaj ukoliko se nađe u životnoj sredini.

UKLANJANJE PFAS JEDINJENJA IZ VODE TRETMANOM PLAZMOM

Disperzija per – i polifluoroalkil supstanci (PFAS) u životnoj sredini i njihov uticaj na ljudsko zdravlje i ekosistem izazivaju zabrinutost u poslednjih nekoliko decenija (Ahrens & Bundschuh, 2014; Zhao et al., 2016). Ovi organski molekuli, takozvane „večne hemikalije“ deo su sintetičke proizvodnje od sredine dvadesetog veka i koriste se svakodnevno u komercijalne i potrošačke svrhe.

Nekoliko hiljada identifikovanih hemikalija spada u ovu grupu supstanci koje imaju različita svojstva, ali sve sadrže neverovatno jaku i stabilnu vezu ugljenik-fluor koju je veoma teško razbiti. Na jednom ili na oba kraja je vezana polarna ili jonizujuća „glavna“ grupa poput karboksilne i sulfonske kiseline (Buck et al., 2011). Takva struktura im pruža jedinstvena fizičko-hemijska svojstva, visoku termičku i hemijsku stabilnost, otpornost na vodu, ulja i masti. Raznolikost u strukturi i svojstvima pruža mogućnost za različite primene (Barzen-Hanson et al., 2017).

Pokazno je da je značajna (hronična) toksičnost nekih od ovih supstanci povezana sa njihovom bioakumulacijom u izvorima vode za piće i mogućnošću da se akumuliraju u dijetetskim proizvodima i ljudima izazivajući ozbiljne zdravstvene probleme (Hayman et al., 2021; X. Li et al., 2022; Sander, 2015). Najpoznatija potencijalno opasna jedinjenja su perfluorooktanski sulfonat (PFOS) i perfluorooktanska kiselina (PFOA) i njihovo korišćenje je regulisano širom Evrope i Sjedinjenih Država i (Evropska komisija, 2020; 2022; USEPA, 2016). Međutim zbog regulacija koje ograničavaju globalnu proizvodnju PFOS i PFOA supstanci, počeo je razvoj novih PFAS (Wang et al., 2013). Nedavne studije pokazuju da iako je mala mogućnost da se akumuliraju u vodenim organizmima, njihovo prisustvo u vodi (pijaćoj i podzemnoj vodi) ima uticaj na životinje i ljude koji je konzumiraju i izloženi su im (Xiao, 2017).

Pokazano je da je većina PFAS veoma otporna prema konvencionalnom tehnologijama za tretman otpadnih voda i slatkih voda kao što su adsorpcija na aktivnom uglju i metode jonske izmene (Chow et al., 2022). Nekoliko NOP se pokazalo efikasnim u njihovom uklanjanju iz vode, elektrohemijska oksidacija (Duinslaeger & Radjenovic, 2022), sonoliza (Gole et al., 2018) i fotokataliza (Z. Li et al., 2020). Među njima je i tehnologija tretmana plazmom, gde je korišćeno korana pražnjenje, impulsno strimersko pražnjenje i plazma u mehurićima u tečnosti (Biondo et al., 2023; Isowamwen et al., 2023; Saleem et al., 2020; Singh et al., 2019).

1.4 ORGANIZACIJA TEZE

Tema ove doktorske disertacije je proučavanje osobina neravnotežnog pražnjenja na atmosferskom pritisku u kontaktu sa tečnostima. Glavni cilj istraživanja bila je dijagnostika gasnog pražnjenja plazma mlaza sa šiljastom elektrodom, konstruisanog u *Laboratoriji za neravnotežne procese i primenu plazme*, u okviru *Centra za neravnotežne procese Instituta za Fiziku u Beogradu*. Ovaj izvor plazme proizvodi strimersko pražnjenje na atmosferskom pritisku koje je u kontaktu sa tečnošću i radi sa napajanjem u oblasti kHz frekvencija. Motivacija za ovo istraživanje bila je mogućnost novih primena takvog sistema u interdisciplinarnim oblastima koje uključuju biologiju, plazma poljoprivredu i tehnologije prečišćavanja vode.

Doktorska disertacija se sastoji od šest poglavlja. Prvo poglavlje je **Uvod** u okviru koga je dat teorijski opis metoda korišćenih u ovom istraživanju, kao i pregled aktuelnih tema istraživanja u oblasti primena koje su ovde izučavane. U okviru poglavlja **Eksperimentalna postavka i metodi** predstavljen je korišćeni izvor plazme, opisana je osnovna postavka eksperimenta i detaljno je prikazana postavka i aparatura korišćena u svakoj primenjenoj metodi. Na kraju ovog poglavlja dat je opis fizičko-hemijskih metoda korišćenih za karakterizaciju tečnih uzoraka tretiranih plazmom.

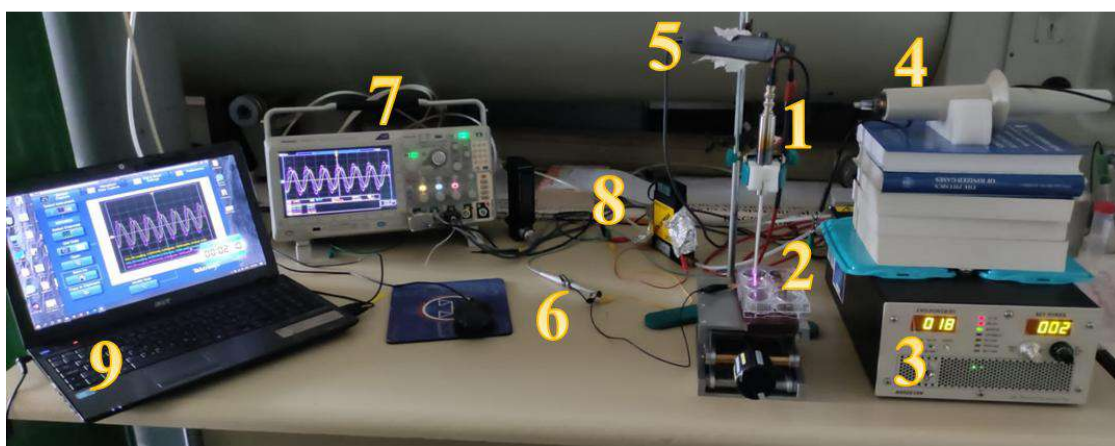
Rezultati istraživanja prikazani su u trećem i četvrtom poglavlju. Treće poglavlje **Dijagnostika gasnog pražnjenja u oblasti kHz frekvencija na atmosferskom pritisku u kontaktu sa tečnom metom** se sastoji iz dva potpoglavlja. U prvom su opisani rezultati **Električne karakterizacije strimerskog pražnjenja**, u okviru koje su merene električne karakteristike gasnog pražnjenja pri različitim parametrima u sistemu. Potpoglavljje **Detekcija emisije zračenja iz plazme** sadrži rezultate dobijene pomoću tri različite optičke metode. Optička emisiona spektroskopija, kao pasivna optička metoda služila je za snimanje spektra zračenja i identifikovanje pobuđenih vrste iz plazme i određivanje temperature gasa. Pored toga, urađeno je snimanje prostornih profila emisije zračenja pomoću iCCD kamere. Kako bi se dobile informacije o raspodeli emisije zračenja koja potiče od različitih reaktivnih vrsta iz plazme, ispred kamere su postavljani uskopojasni optički filteri – elementi za selekciju određene talasne dužine. Na taj način je snimljeno zračenje razloženo po talasnim dužinama. Snimanje razvoja i kretanja filamentarnog pražnjenja u oblasti interakcije plazme i tečnosti urađeno je brzom kamerom. Urađeno je snimanje strukture pražnjenja u zavisnosti od provodnosti tečne mete i procenjeni su efektivna kontaktna površina i broj račvanja strimerskog pražnjenja. Ovaj deo istraživanja je urađenu saradnji sa kolegama iz *Istraživačke grupe za Hemiju plazme, Instituta za Fizičku i primenjenu hemiju, Hemijskog fakulteta Tehnološkog univerziteta u Brnu, Češka Republika*, pod rukovodstvom profesora dr Františka Krčme.

U okviru četvrtog poglavlja **Primena plazma mlaza sa šiljastom elektrodom u biotehnologiji i tehnologiji prečišćavanja vode** ispitani su uticaji plazme na tretirane tečne uzorke i mogućnost upotrebe tehnologije plazme u dve konkretne primene. U okviru dela **Tretmani biljnog materijala PAV-om**, urađeni su tretmani čiste vode sa ciljem dobijanja PAV-a i ispitivan je uticaja PAV-a na biološke sisteme – kaluse biljaka ili cele biljke. Druga primena je obuhvatala **Tretmane zagađene vode plazmom**. Predstavljeni su rezultati tretmana vode zagađene pesticidima i vode zagađene PFAS jedinjenjima, urađenih sa ciljem dobijanja prečišćene vode koja može ponovo da se upotrebi.

Na kraju disertacije, u petom poglavlju dat je opšti **Zaključak** o urađenom istraživanju, a poslednje poglavlje disertacije sadrži **Priloge**.

2. EKSPERIMENTALNA POSTAVKA I METODI

U ovom poglavlju predstavljen je detaljni opis elemenata eksperimentalnih postavki i aparature korišćene prilikom dijagnostike plazma mlaza sa šiljastom elektrodom u kontaktu sa tečnom metom. Izvor plazme koji radi na atmosferskom pritisku konstruisan je u Laboratoriji za neravnotežne procese i primenu plazme Instituta za fiziku u Beogradu. Opis plazma mlaza i osnovnih komponenti korišćene aparature (prikazanih na slici 2.1), kao i metode za dijagnostiku pražnjenja u gasnoj fazi nalaze se u potpoglavlju 2.1. Aparatura i fizičko-hemijske metode korišćene za karakterizaciju tečnih uzoraka tretiranih plazmom opisani su u delu 2.2.



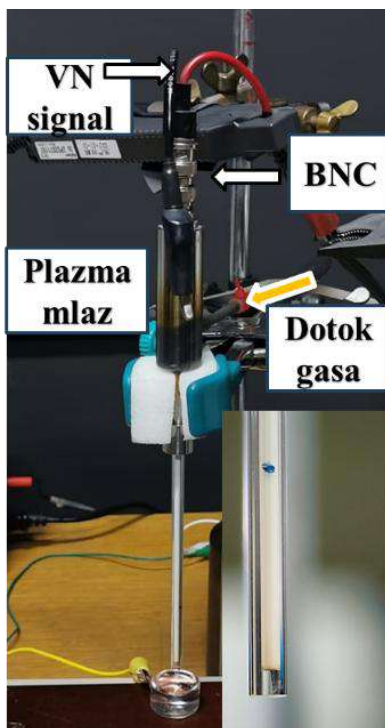
Slika 2.1: Fotografija eksperimentalne postavke. (1) Plazma mlaz, (2) uzemljeni tretirani uzorak u mikrotiter posudi (3) visokonaponsko napajanje – izvor snage, (4) visokonaponska sonda, (5) strujna sonda, (6) naponska sonda i uzemljeni merni element, (7) osciloskop, (8) protokomer, (9) računar.

2.1 PLAZMA MLAZ I DIJAGNOSTIČKI METODI

PLAZMA MLAZ SA ŠILJASTOM ELEKTRODOM

Izvor plazme koji je korišćen u svim eksperimentima konstruisan je u Laboratoriji za neravnotežne procese i primenu plazme u okviru Centra za neravnotežne procese Instituta za fiziku u Beogradu. Sastoji se od vertikalno pozicioniranog cilindričnog metalnog tela dijametra 22 mm unutar koga je duž aksijalne ose postavljena staklena cev unutrašnjeg i spoljašnjeg prečnika 4 mm i 6 mm, respektivno. Duž ose cevi prolazi žica od nerđajućeg čelika, prečnika 1 mm, zašiljena na vrhu i izolovana keramičkom cevčicom prečnika 1,2 mm. Donji zašiljeni kraj žice dužine 3 mm viri iz keramičke izolacije i sam vrh se nalazi unutar staklene cevi na rastojanju 1 mm od ivice cevi. Žica predstavlja napajanu elektrodu i povezana je BNC konektorom za gornji kraj metalnog tela. Na gornji kraj žice preko BNC konektora se dovodi visokonaponski (VN) naizmenični signal. Gas ili smeša gasova se uvode u staklenu cevčicu preko priključka sa bočne strane metalnog tela mlaza. Detalji konekcija su prikazani na slici 2.2.

Korišćeni su plemeniti gasovi He 5.0 i Ar 5.0, kao i smeše Ar i sintetičkog vazduha (Sintetički vazduh 5.0 bez CH, 20,5% O₂ u N₂) (procentualno u smeši 1 % ili 3 %). Protok radnog gasa je podešavan masenim protokomerima *Omega FMA5518*, opseg 0 - 5 l/min i *Bronkhorst MV 301*, opseg 10 – 200 ml/min.



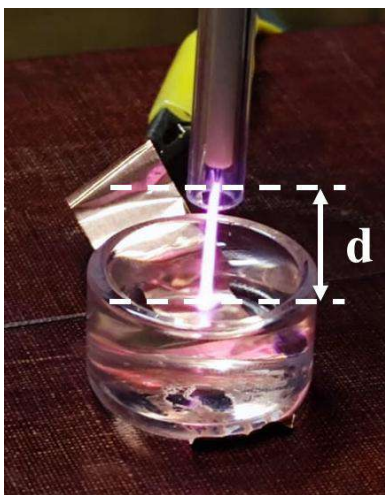
Slika 2.2: Plazma mlaz sa prikazanim priključcima za električni signal i gas. U donjem desnom uglu je prikazana napajana elektroda.

Visokonaponski izvor napajanja

Plazma je u svim eksperimentima inicirana i kontrolisana komercijalnim VN visokofrekventnim izvorom snage (*T&C Power Conversion AG 0201HV-OS 140 W – 500 kHz*) koji ima mogućnost samoregulacije impedanse u zavisnosti od impedanse ostatka sistema. Uređaj sadrži kontroler sa mikroprocesorskim upravljanjem koji prati faznu razliku između struje i napona, snagu na izlazu napajanja P_F (predata snaga, eng. forwarded power) i reflektovanu snagu P_R (eng. reflected power). Praćenjem snage P_R i pomoću sistema povratne sprege uređaj kontroliše radnu frekvenciju signala (u opsegu od 200 - 450 kHz) čime se reaktansa elemenata u sistemu plazma mlaza, a time i ukupna impedansa sistema, prilagođava izlaznoj impedansi uređaja. Približavanjem impedanse sistema optimalnoj impedansi za prenos snage - postiže se minimalna snaga P_R i maksimalna efikasnost prenosa energije (T&C Power Conversion, Inc., 2020). Obe snage (u jedinici W) su prikazane na prednjoj strani kućišta napajanja. Uređaj omogućava i ručno podešavanje rezonantne frekvencije signala koji se dovodi na plazma mlaz čime se dodatno smanjuje reflektovana snaga u sistemu. U svim eksperimentima, na napajanju elektrodi je dovođen VN sinusni signal frekvencije u opsegu od 330 kHz do 350 kHz.

Tretirani uzorak – uzemljena elektroda

Ispod plazma mlaza se nalazi posuda sa tečnim uzorkom koji je dodatno uzemljen preko dna posude tako što je na spoljnoj strani zalepljena bakarna traka (videti sliku 2.3). Strimer koji se formira u gasnoj fazi dolazi u kontakt sa površinom tečnosti, tako da tretirani uzorak u električnom kolu predstavlja drugu elektrodu. Parametar koji se fiksira tokom tretmana jeste rastojanje d [mm] između vrha napajane elektrode i površine tečnosti. Ispitivano je pražnjenje u kontaktu sa metama različitih fizičko-hemijskih osobina, zapremine i u različitim posudama. Uglavnom je kao meta korišćena voda (destilovana ili voda sa česme), ali su tretirane i otpadne vode različitog sadržaja.



Slika 2.3: Tretirani uzorak u kvarcnoj posudi. Rastojanje d – između napajane elektrode i površine tečnosti. Bakarna traka na dnu posude sa uzorkom je uzemljena elektroda.

DIJAGNOSTIČKI METODI

Dijagnostika gasne faze pražnjenja obuhvatala je električnu karakterizaciju sistema plazma mlaza i optičku dijagnostiku. U okviru električne karakterizacije su snimane strujno naponske karakteristike i primenjene su dve različite metode za merenje snage. Optička dijagnostika plazma mlaza, obuhvatala je:

- OES metodu za snimanje emisionih spektara u širokom opsegu talasnih dužina i određivanje rotacione temperature molekula OH,
- tehniku brzog slikanja plazme ICCD kamerom pomoću koje je dobijena prostorna raspodela zračenja u gasnoj fazi,
- tehniku snimanja vremenske evolucije strimerskog pražnjenja u kontaktu sa tečnošću brzom kamerom (fast-frame camera),
- metodu određivanja temperature gasa zasnovanu na Rejljevom rasejanju,
- metodu merenja koncentracije OH molekula zasnovanu na laserski indukovanoj fluorescenciji.

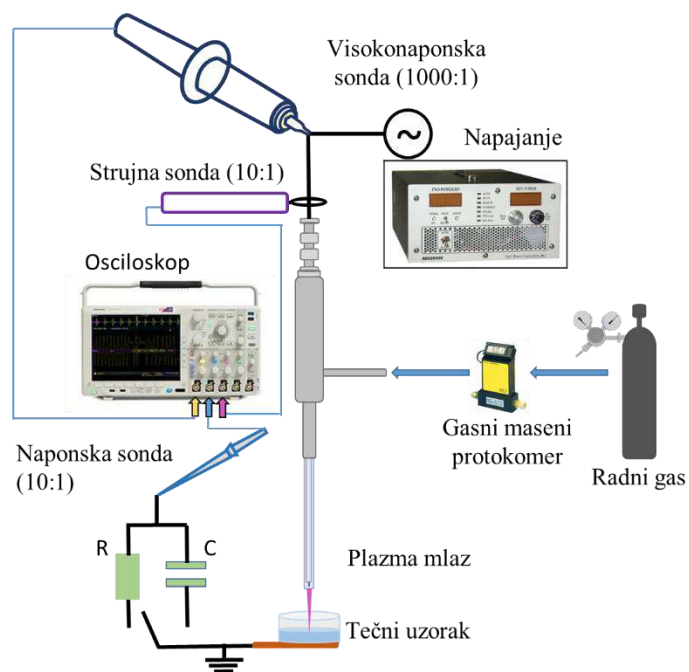
2.1.1 ELEKTRIČNA KARAKTERIZACIJA

Električna karakterizacija izvora plazme obuhvatala je snimanje i analizu merenih električnih signala, određivanje strujno-naponskih karakteristika uređaja i merenja snage pri različitim operativnim parametrima plazma mlaza. Ispitano je kako promene različitih parametara u sistemu kao što su vrsta i protok radnog gasa, fizičko-hemijske osobine tečne mete i rastojanje d , utiču na strujno-naponske karakteristike, kao i na snagu oslobođenu u sistemu i snagu predatu tečnom uzorku.

ODREĐIVANJE STRUJNO-NAPONSKE KARAKTERISTIKE

Električno kolo sistema plazma mlaza (videti Sliku 2.4) je konstruisano tako da omogućuje snimanje dve strujno-naponske karakteristike, u glavnoj i u uzemljenoj grani kola. Mereni su sledeći vremenski promenljivi električni signali:

- $v(t)$ – trenutni VN signal kojim se napaja plazma mlaz, korišćenjem pasivne visokonaponske sonde (Tektronix P6015A),
- $i(t)$ – struja koja prolazi kroz plazma mlaz merena je strujnom sondom hibridnog tipa (Agilent N2783B ili Tektronix TCP0030A),
- $i_G(t)$ – struja koja prolazi kroz uzorak i uzemljenu granu kola merena je indirektno, snimanjem pada napona na mernom elementu ubačenom serijski nakon uzorka u uzemljenu granu kola. Merenje je izvršeno pasivnom naponskom sondom (Agilent N2863B).



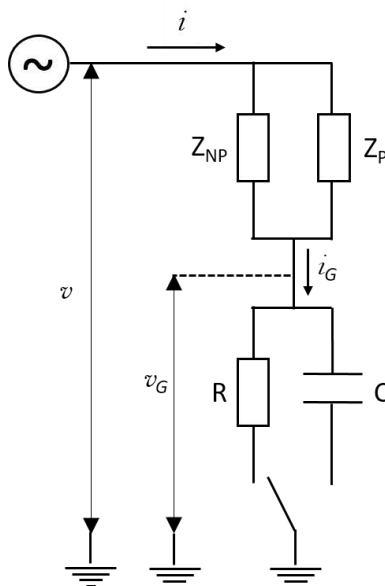
Slika 2.4: Shematski prikaz eksperimentalne postavke za električnu karakterizaciju sistema.

Merni elementi koji su korišćeni u uzemljenoj grani kola bili su: otpornik električne otpornosti $R = 1 \text{ k}\Omega$, kondenzatori kapacitivnosti $C = 0,8 \text{ nF}$ i $C = 10,3 \text{ nF}$. Svi vremenski promenljivi električni signali su snimljeni na osciloskopu (*Tektronix MDO3024*).

Snimanje je urađeno u dva režima:

- kada kroz plazma mlaz protiče radni gas i gasno pražnjenje je formirano (ON)
- kada nema protoka radnog gasa i pražnjeja (OFF).

Na slici 2.5 prikazano je električno kolo sistema sa plazma mlazom i označeni su strujni i naponski signali koji se mere u kolu. Impedansa Z_{NP} predstavlja ukupnu impedansu u kolu u kome se nalazi plazma mlaz, kada u kolu nema protoka gasa, a samim tim ni pražnjenja. Imedansa plazme je Z_P .



Slika 2.5: Električno kolo sistema plazma mlaza: v – vremenski promenljivi VN signal primenjen na napajanje elektrode, v_G – naizmenični naponski signal meren u uzemljenoj grani kola, i – struja koja prolazi kroz plazma mlaz u glavnoj grani kola, i_G – strujni signal kroz uzemljenu granu kola, R – otpornik i C – kondenzator - kao merni elementi u kolu, Z_{NP} – impedansa sistema plazma mlaza u konfiguraciji kada nema pražnjenja u kolu, Z_P – impedansa plazme.

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Prilikom određivanja strujno naponske-karakteristike postepeno je povećavana snaga P_F . Pošto u električnom kolu ima više različitih elemenata, snaga P_F nije jednaka snazi koja je predata plazma mlazu P . Tačno i precizno merenje snage oslobođene u pražnjenju - ključnog parametra za uspešnu primenu atmosferske plazme, bio je glavni cilj ovog dela istraživanja. Zbog toga je za svaku tačku merenja, za datu P_F , računata srednja snaga predata plazma mlazu u toku jednog perioda primenjenog visokog napona korišćenjem formule **1.8**.

Snimanjem električnih karakteristika u režimima sa i bez plazme bilo je moguće odrediti gubitke u električnom kolu. Konačna vrednost srednje snage $\langle P_{NET} \rangle$ dobijena je kao razlika snaga $\langle P_{sa\ protokom} \rangle$ i $\langle P_{bez\ protoka} \rangle$.

Isti postupak je ponovljen i u slučaju računanja snage oslobođene u uzemljenoj grani kola P_G . U ovom slučaju metod koji je korišćen za računanje zavisio je od mernog elementa u uzemljenoj grani kola – otpornika ili kondenzatora (videti poglavlje **1.2.1**). Izabrana je odgovarajuća otpornost i kapacitivnost naponske sonde tako da minimalno remeti električno kolo i utiče na merenja.

2.1.2 OPTIČKA EMISIONA SPEKTROSKOPIJA

SNIMANJE EMISIJE ZRAČENJA PLAZME

OES je jedna od najrasprostranjenijih metoda za dijagnostikovanje plazme i ovde je korišćena za identifikovanje pobuđenih vrsta (molekula, atoma, jona i radikala) koje su najznačajnije pri proučavanju mehanizama interakcije plazme i tečnosti jer omogućuju uvid u dominantne reakcije koje se odvijaju u plazmi. U ove svrhe korišćen je komercijalni optički emisioni spektrometar *Maya2000 Pro-UV-NIR, Ocean Insight-High Sensitive Spectrometer*.

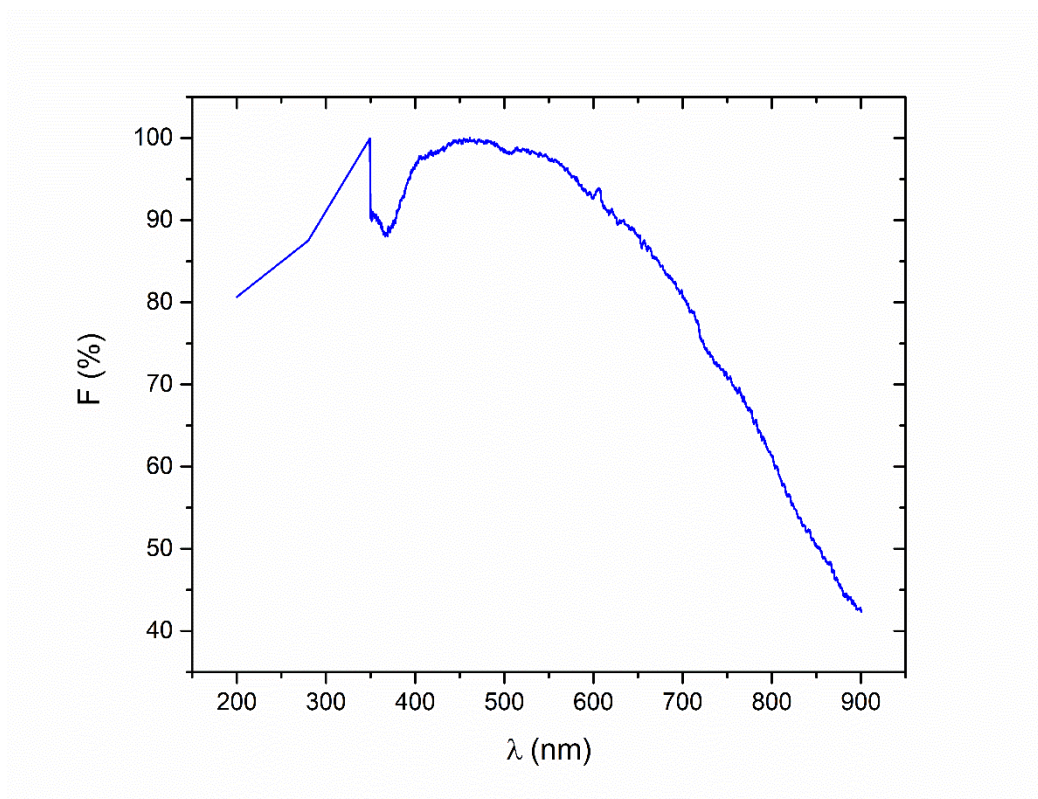
Osim za dijagnostikovanje pobuđenih vrsta u plazmi, ova metoda je korišćena i za određivanje rotacione temperature molekula OH, snimanjem intenziteta rotacionih spektralnih linija OH u uskom opsegu talasnih dužina. Za ova merenja korišćen je spektrometar *Omni-750i Monochromator and Spectrograph*.

Spektrometar Maya2000 Pro – kalibracija uređaja

Spektrometar Maya2000 Pro omogućuje merenje u opsegu talasnih dužina od 200 nm do 900 nm, sa rezolucijom od 0,5 nm (širina linije na polovini maksimalnog intenziteta, eng. full width at half maximum, FWHM). Uređaj sadrži 2 m dugačko optičko vlakno (Thorlabs *M114L02*), čiji je prečnik jezgra (eng. core diameter) 600 μm i koje ima konstantno visoku spektralnu efikasnost u širokom opsegu talasnih dužina. Na kraju optičkog vlakna pričvršćeno je sočivo *Ocean Optics UV74*.

Spektralna efikasnost optičkog sistema je dobijena merenjem intenziteta zračenja lampe *Osram WI 17/G*, snimljenog na poznatoj temperaturi $T = 2856\text{ K}$. Pošto je lampa od stakla, snimljeno je zračenje u spektru od 350 nm do 900 nm. Snimljen je signal kada je lampa uključena i signal kada je lampa isključena (pozadinsko zračenje, eng. background) i merenja su ponovljena tri puta. Od svakog snimljenog intenziteta oduzeto je pozadinsko zračenje i dobijen je normiran srednji intenzitet zračenja lampe I_{N_MEAN} . Izračunata je teorijska kriva zračenja lampe $B(T)$, pod pretpostavkom da je lampa crno telo i iz odnosa ove dve vrednosti pronađena je **funkcija odziva spektrometra F** , kao odnos snimljenog intenziteta i teorijske vrednosti intenziteta zračenja crnog tela (lampe): $F = I_{N\_MEAN}/B(T)$. Urađeno je normiranje vrednosti funkcije odziva kako bi mogla da se predstavi u procentima (%), tako što se za dati opseg talasnih dužina od 350 nm do 900 nm nađe maksimalna vrednost F_{max} i uzme se da je to 100%, a ostale vrednosti se skaliraju: $F(\%) = 100 \cdot F / F_{max}$.

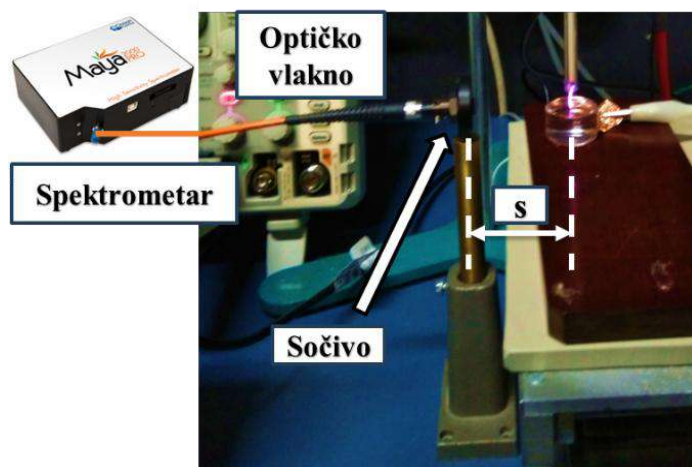
Funkcija odziva u opsegu talasnih dužina od 200 nm do 350 nm dobijena je uzimajući u obzir spektralne karakteristike svih optičkih elemenata u eksperimentalnoj postavci. Funkcija odziva spektrometra sa optičkim vlaknom u celom opsegu talasnih dužina (od 200 nm-900 nm) prikazana je na Slici 2.6.



Slika 2.6: Funkcija odziva optičkog sistema sa spektrometrom Maya2000 Pro.

Eksperimentalna postavka

Optičko vlakno sa sočivom na kraju je postavljeno duž ose normalne na osu plazma mlaza, tako da je rastojanje između sočiva i uspostavljenog strimerskog pražnjenja bilo $s=5,5$ cm (slika 2.7). Snimano je zračenje iz čitave oblasti između elektrode plazma uređaja i površine vode. Rastojanje između vrha elektrode i površine uzorka bilo je $d=10$ mm. Tokom snimanja spektara uzorak je bila destilovana voda zapremine 4 ml koja se nalazila u kvarcnoj posudi cilindričnog oblika, zapremine 4 ml. Merenja emisijonog spektra su izvršena različite radne gasove i protoke.



Slika 2.7: Eksperimentalna postavka za snimanje emisije zračenja plazme, s - rastojanje između sočiva i pražnjenja.

Tokom snimanja spektara korišćen je programski paket *OceanView 2.0*, koji omogućava podešavanje vremena uzorkovanja (eng. integration time) i usrednjavanje nekoliko signala emisije, što doprinosi minimizovanju fluktuacija dobijenih signala. Svako merenje se sastojalo od snimanja spektra zračenja plazme i snimanja pozadinskog zračenja, koje je oduzeto od prvobitnih signala. Snimljeni intenziteti su zatim skalirani pomoću funkcije odziva sistema. Sva merenja su urađena tri puta i prikazane su srednje vrednosti.

MERENJE ROTACIONE TEMPERATURE OH MOLEKULA

Tehnika snimanja emisionog spektra plazme iskorišćena je i za merenje rotacione temperature elektronski pobuđenog molekula $\text{OH}(A^2\Sigma^+ - X^2\Pi)$, koja može da aproksimira temperaturu gasne faze. Merenja su urađena u saradnji sa kolegama dr Antonom Nikiforovim i dr Mihailom Gromovim u Istraživačkoj grupi za tehnologiju plazme pri Univerzitetu u Gentu, u Belgiji.

Eksperimentalna postavka je prikazana na slici 2.8. Merenje je izvršeno pomoću spektrografa *Zolix Omni- λ 750i* opremljenog ICCD kamerom *Andor Solis iStar DH334*, čime je obezbeđen spektar sa visokom rezolucijom, u optičkom opsegu od 304 do 312 nm. Optička emisija je prikupljena optičkim vlaknom i analizirana pomoću monohromatora (rešetka sa 2400 zarezova mm^{-1} , sa maksimalnom efikasnošću na 250 nm), uz upotrebu ulaznog proreza širine 15 μm .

Na osnovu dobijenih spektara određivana je rotaciona temperatura (T_{rot}) prelaza $\text{OH}(A^2\Sigma^+ - X^2\Pi)$, uz pretpostavku Bolcmanove raspodele rotacionih nivoa. Dalje je pretpostavljeno da je T_{rot} jednaka temperaturi gasa (T_{gas}), usled veoma brzog prenosa rotaciono-translacione energije ($< 1 \text{ ns}$), čime je ispunjen uslov translaciono-rotacione ravnoteže: $T_{\text{rot}} \approx T_{\text{trans}} (= T_{\text{gas}})$ (Bruggeman et al., 2014; Van Rompaey et al., 2025). Rotaciona struktura prelaza $\text{OH}(A^2\Sigma^+ - X^2\Pi)$ prilagođena je simuliranoj strukturi korišćenjem softvera MassiveOES (Miles, Lempert, & Forkey, 2001; Voráč et al., 2017). Svi emisijski spektri su korigovani na pozadinu, a intenzitetska kalibracija spektrometara izvršena je pomoću volfram-halogene lampe (250–2400 nm, NIST).



Slika 2.8: Eksperimentalna postavka za određivanje temperature gasa metodom OES sa brzim slikanjem ICCD kamerom: 1- ICCD kamera, 2 - spektrograf, 3 – optički kabl, 4 – blenda, 5 – plazma mlaz, 6 – tečna meta, $s_1=6,3 \text{ mm}$ – rastojanje između blende i slita na optičkom vlaknu, $s_2=16,5 \text{ mm}$ – rastojanje između blende i plazma mlaza, $d=10 \text{ mm}$ – rastojanje između vrha elektrode i površine tečnosti.

Tokom merenja plazma je bila u kontaktu sa tečnom metom – 4 ml destilovane vode koja se nalazila u kvarcnoj posudi iste zapremine. Rastojanje između vrha elektrode i površine plazma mlaza je bilo $d = 10 \text{ mm}$. Rastojanje između blende i razreza na optičkom vlaknu je bilo $s_1 = 6,3 \text{ mm}$, a između blende i plazma mlaza $s_2 = 16,5 \text{ mm}$. Za svaku tačku merenja snimljeno je 50 slika ICCD kamerom, koje su zatim usrednjene.

2.1.3 TEHNIKA SNIMANJA PROSTORNIH PROFILA ZRAČENJA IZ PLAZME ICCD KAMEROM

U okviru ovog dela optičke dijagnostike pražnjenja u gasnoj fazi, izučavana je prostorna raspodela strimerskog pražnjenja u kontaktu sa tečnom metom. Snimljeno je ukupno zračenje plazme u opsegu talasnih dužina od 180 nm do 850 nm pri čemu su profili bili vremenski usrednjeni (vremenski integrisani profili). Korišćenjem uskopropusnih interferencionih filtera snimljeni su i profili razloženi po talasnim dužinama (profili pojedinačnih pobuđenih vrsta). Druga tehnika merenja je obuhvatala korišćenje brze kamere (eng. fast-frame imaging), za snimanje brzih promena pražnjenja – kretanja osnovnog strimerskog kanala po površini mete i njegovo grananje na filamente u blizini površine tečnosti.

Slikanje prostorno razložene strukture strimerskog pražnjenja urađeno je pomoću ICCD kamere (*Andor iStar DH734I*), u okviru precizno definisanog vremenskog perioda (eng. gating) kako bi se proučila kinetika čestica u plazmi. Slika 2.9 prikazuje eksperimentalnu postavku sa (1) ICCD kameru, opremljenu (2) podesivim prstenom PN11 koji omogućava uveličanje slike, smanjujući upola žižnu daljinu detekcionog sistema. Opciono je korišćen i objektiv (3) MicroNIKKOR 105 mm f/2.8 koji omogućuje snimanje u UV oblasti.



Slika 2.9: Snimanje prostornih profila pražnjenja ICCD kamerom.

Pored toga urađeno je i snimanje emisije razložene po talasnim dužinama, gde su korišćeni optički filteri sa uskim propusnim opsegom. Na taj način je praćena kinetika pojedinačnih ekscitovanih vrsta. Korišćeni su filteri:

- 310 nm – za snimanje OH(A-X) trake u opsegu od 306-309 nm,
- 660 nm – za slikanje linije H_{α} talasnoj dužini na 656,5 nm
- 780 nm – za snimanje linije atomskog kiseonika O na 778 nm
- 800 nm – zračenje koje potiče od Ar

UV-VIS spektrofotometrom *Beckman Coulter DU720* snimljeni su stransmisioni spektri filtera u odgovarajućim oblastima talasnih dužina. Prilikom obrade slika u softverskom paketu *Matlab* uzeti su obzir:

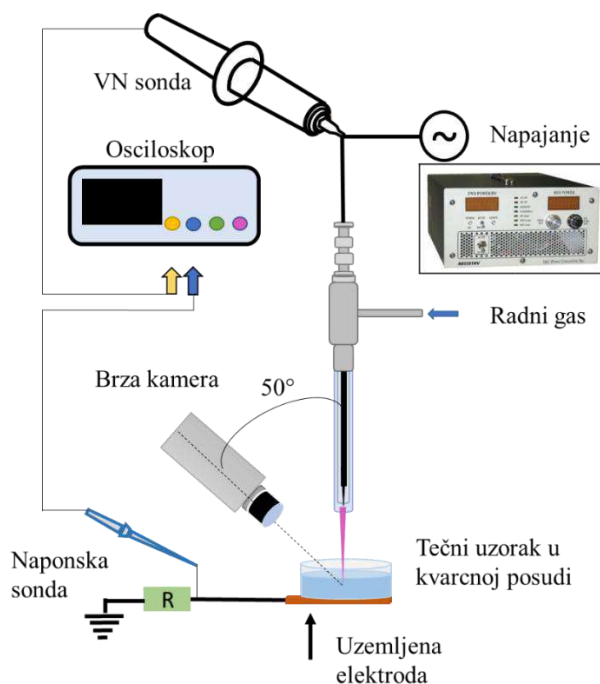
- parametri snimanja: vreme ekspozicije, širina definisanog vremenskog perioda snimanja (eng. gate pulse width) i pojačanje (eng. gain)
- spektralna funkcija kamere
- transmitivnost filtera

2.1.4 SNIMANJE KRETANJA STRIMERSKOG PRAŽNjenja U KONTAKTU SA TEČNOŠĆU BRZOM KAMEROM

U okviru ovog dela istraživanja korišćena je tehnika snimanja plazme brzom kamerom kako bi se proučavala evolucija emisije strimerskog pražnjenja u kontaktu sa vodom. Ove kamere omogućuju posmatranje prelaznog/promenljivog (eng. transient) ponašanja strimerskog pražnjenja snimanjem emisionih profila u dovoljno kratkim intervalima da bi se promene zapazile. Prostorna raspodela emisije zračenja između vrha elektrode i površine tečnosti snimljena je za različite parametre pražnjeja i različite vrednosti električne provodljivosti tečne mete. Posmatrani su razvoj, putevi prostiranja i grananje strimerskog pražnjenja ka površini vode.

Eksperimentalna postavka sa brzom kamerom prikazana je na Slici 2.10. Za iniciranje električnog pražnjenja korišćen je naizmenični sinusni signal frekvencije 350 kHz. Uzorak je postavljen u kvarcnu posudu ispod plazma mlaza, a zapremina uzorka bila je 4 ml. Rastojanje d od vrha igle do površine mete bilo je 10 mm. Korišćen je Ar 4.6 kao radni gas, a protok gasa od 1 slm je kontrolisan masenim protokomerom (*Bronkhorst EL-FLOW*). Kao meta korišćene su DV, VČ i fiziološki rastvor napravljen dodavanjem NaCl u DV. Električna provodljivost tečnih uzoraka je merena komercijalnim meračem sa sondom (*GMH3431-GE, Greisinger*).

Brza kamera (*FASTCAM SA-X2, Photron*) je postavljena iznad uzorka, pod uglom od 50° u odnosu na osu plazma mlaza (vertikalna osa) sa ciljem posmatranja razvoja i širenja strimera između napajane elektrode i površine tečnosti, kao i formiranja i prostiranja manjih filamenata po površini tečne mete. Pokušano je i snimanje kroz dno kvarce posude sa uzorkom, kada je kamera postavljena ispod uzorka, ali su snimci bili prilično mutni i nejasni, zbog čega se odustalo od ove postavke.



Slika 2.10: Snimanje vremenske evolucije strimerskog pražnjenja u kontaktu sa tečnom metom sa brzom kamerom – šematski prikaz.

2.2 ODREĐIVANJE OSOBINA TEČNIH UZORAKA

Istraživanja u okviru ove celine bila su fokusirana na analizu tečnih uzoraka tretiranih plazom. Upotrebene su različite dijagnostičke metode sa ciljem da se dobiju osnovne informacije o interakciji plazme i tečnosti. Prikupljeni rezultati su bili značajni za prilagođavanje parametara plazme za dobijanje PAV-a sa osobinama pogodnim za specifične primene. U primenama za prečišćavanje zagađene vode, cilj je bio da se ispita efikasnost primene plazma mlaza za razgradnju i mineralizaciju štetnih materija i da se optimizuju osobine plazma mlaza kako bi se povećala efikasnost sistema.

2.2.1 MERENJE FIZIČKO-HEMIJSKIH PARAMETARA

Fizičko hemijska karakterizacija tečne faze obuhvatala je merenja: temperature, električne provodljivosti, pH vrednosti, količine rastvorenog kiseonika i merenje dugoživećih kiseoničnih i azotnih vrsta korišćenjem semi-kvantitativnih i kvantitativnih kolorimetrijskih spektrofotometrijskih metoda. U ovom poglavlju prikazane su karakteristike korišćenih mernih instrumenta i opisane su procedure za njihovu kalibraciju i način merenja. Na kraju je data metoda koja je razvijena za mernje tri dugoživeće reaktivne vrste u tretiranim uzorcima: vodonik-peroksida H_2O_2 , nitrita NO_2^- i nitrata NO_3^- .

ANALIZA FIZIČKO-HEMIJSKIH OSOBINA TEČNOSTI MERNIM SONDAMA

Merenje **pH vrednosti** uzoraka vršeno je u opsegu od pH=2 do pH=12 komercijalnim mernim sondama (*Hanna Instruments, HI1131 i HI7071 pH sonde, HI5521 kontrolna jedinica*) i mernim trakama (*pH indikatorske trake, pH 0 – 14, Univerzalni indikator, Merck, Darmštat, Nemačka*). Sonde se razlikuju po veličini i samim tim, po minimalnoj potrebnoj količini uzorka za merenje. Kalibracija merača pH vrednosti urađena je pomoću puferskih rastvora:

- *Puferski rastvor, pH $4,00 \pm 0,01$ na 20°C , 10405, Reagecon, Šenon, Irska,*
- *Puferski rastvor, pH $7,00 \pm 0,01$ na 20°C , 10705, Reagecon, Šenon, Irska,*
- *Puferski rastvor, pH $10,00 \pm 0,01$ na 20°C , 101005, Reagecon, Šenon, Irska.*

Kontrolna jedinica omogućuje istovremeno korišćenje još dve sonde, za merenje temperature i za merenje **električne provodljivosti** (*Hanna Instruments, HI76312 sonda*). Kalibracija merača električne provodljivosti vršena je pomoću standardnih rastvora poznate provodljivosti.

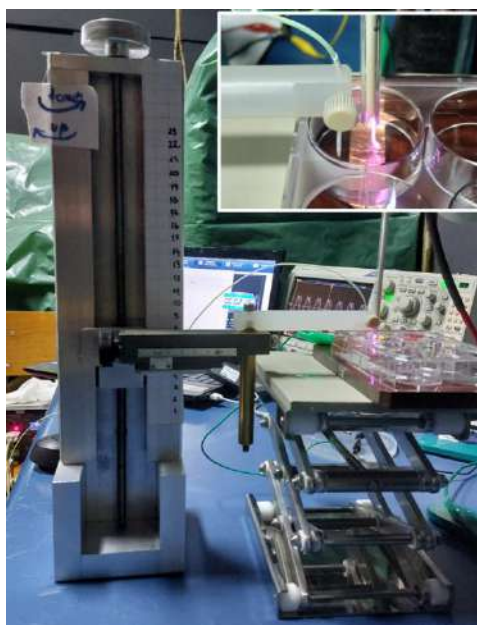
Određivanje **količine rastvorenog kiseonika** u tretiranim uzorcima urađeno je pomoću komercijalne sonde (*Hanna Instruments DO sonda HI764080, kontrolna jedinica HI2004*), koja sadrži kiseoničnu membransku elektrodu Klarkovog tipa (Popović, 2003). Merena vrednost prikazana je u ppm (eng parts per million, jedan deo na milion, milioniti deo), tako da je $1 \text{ ppm} = 1 \text{ mg/l}$, i u procentima (%). Kalibracija uređaja je urađena metodom koju je propisao proizvođač sa jednom referentnom tačkom za maksimalnu vrednost rastvorenog kiseonika – u vazduhu. U tom slučaju sonda je merila količinu u vazduhu, a ne u tečnom uzorku. Maksimalna vrednost je uzeta za 100% rastvorenog kiseonika.

U slučaju tretmana zagađene vode, da bi se utvrdio stepen mineralizacije u zagađenim uzorcima nakon tretmana plazmom meren je **ukupni organski ugljenik** (eng. Total Organic Carbon - TOC), pomoću komercijalnog analizatora (*Sievers 5310 C TOC Analyzer*). Merenja su urađena odmah nakon tretmana a zbog ograničenja opsega analizatora uzorci su pre merenja razblaženi dvadeset puta.

MERENJE TEMPERATURE TERMO-OPTIČKOM SONDOM

Merenje temperature tečnosti urađeno je upotrebom termooptičke sonde – optičkog vlakna *Opsens Solutions IMP00114 OTG-Q* (dužine 1,5 m, sa PFA omotačem, spoljašnjeg prečnika 1,4 mm) na čijem kraju se nalazi senzor u vidu kristala galijum arsenida (GaAs). Metod je zasnovan na promeni zavisnosti širine energijskog procepa (band gap) kristala GaAs od temperature. Na sobnoj temperaturi kristal je optički prozračan za svetlost talasne dužine 850 nm i više. Sa povećanjem temperature u kristalu, spektar transmitovane svetlosti se pomera ka većim talasnim dužinama, a promena se detektuje u kontrolnom uređaju (*Opsens Solutions IMP0073*) koji je kalibrisan i odmah prikazuje vrednosti izmerene temperature. Metod je primenljiv u opsegu temperatura od -40 °C do +250 °C, sa tačnošću od $\pm 0,8$ °C.

Temperatura gasa i tečnosti tokom tretiranja je merena na različitim rastojanjima od igle plazma mlaza. Za precizno merenje rastojanja sonde od plazma mlaza, korišćen je specijalno dizajniran nosač sonde koji ima dva milimetarska zavrtnja i omogućava pomeranje po 2 ose (videti sliku 2.14).



Slika. 2.11: Merenje temperature termooptičkom sondom

KOLORIMETRIJSKE METODE ZA ODREĐIVANJE KONCENTRACIJE REAKTIVNIH VRSTA KISEONIKA I AZOTA

Za detektovanje dugoživećih vrsta, vodonik peroksida, nitrita i nitrata u tečnom uzorku tretiranom plazmom, prilagođena je i usvojena kvantitativna merna metoda. Koncentracije ovih vrsta su su merene upotrebom komercijalnih kompleta za ispitivanje koji su zasnovani na kolorimetrijskoj analizi uz pomoć spektrofotometra. Prema Lamber-Berovom zakonu:

$$A_{\lambda} = \varepsilon \cdot l \cdot c \quad (2.1)$$

gde je:

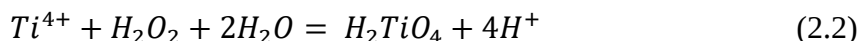
- A_{λ} - apsorbanca na određenoj talasnoj dužini λ ,
- ε - molarni apsorpcioni koeficijent ispitivane supstance,
- l - optički put,
- c – koncentracija ispitivane supstance.

U opseg linearne zavisnosti $A_{\lambda} = f(c)$ moguće je merenjem apsorbanse odrediti nepoznatu koncentraciju c .

Merenja su rađena na UV-VIS spektrofotometrima, jednozračnom (*Beckman Coulter DU720*) i dvozračnom sa mogućnošću merenja do osam uzoraka istovremeno (*Perkin Elmer Lambda 365*). Merenja su rađena sa kvarcnim kivetama, optički put 10 mm, (*Kvarcna kiveta, Q – 104, Amtast, SAD*) a kao blank uzorak je korišćena destilovana voda. Za svaku vrstu ponaosob je napravljena kalibraciona kriva uz pomoć standardnih rastvora, u opsegu u kojima je zavisnost koncentracija reaktivne vrste u rastvoru od apsorbanse rastvora (na karakterističnoj talasnoj dužini) linearna. Ispitivani uzorci su po potrebi razblaživani sa vodom, ukoliko je koncentracija reaktivne vrste izvan ove oblasti kako bi merenje uvek bilo vršeno unutar opsega kalibracije.

Vodonik-peroksid, H_2O_2

Metoda merenja vodonik peroksida je urađena pomoću rastvora titanijum oksisulfata ($TiOSO_4$) (*Titanijum (IV) oksisulfat – rastvor u H_2SO_4 , 27-31% baza sumporne kiseline, 89532-11, Sigma Aldrich, Francuska*). Metod je zasnovan na merenju apsorbanse žuto obojene pertitanske kiseline (H_2TiO_4), koja nastaje u reakciji H_2O_2 i jona titanijuma u kiselom rastvoru sumporne kiseline:



Pertitanska kiselina ima maksimum apsorpcije na 407 nm, stoga se merenja vrše na ovoj talasnoj dužini. Rastvor za merenje se pravi tako što se doda jedna zapremina reagenta ($TiOSO_4$) na deset zapremina vodenog rastvora koji sadrži H_2O_2 (Eisenberg, 1943). Apсорpcija se meri na 407 nm.

Kalibraciona kriva je prema uputstvu proizvođača napravljena u opsegu od 0 do 50 mg/l vodonik peroksida. U ove svrhe, napravljeno je sedam rastvora poznate koncentracije (1, 2, 5, 10, 12,5, 25, 50 mg/l), rastvaranjem 30% standardnog rastvora H_2O_2 (1.07210.1000, *Merck, Darmštat, Nemačka*) u destilovanoj vodi. U literaturi je takođe pokazana linearna zavisnost u ovom opsegu (Bruggeman et al. 2016).

Opisan metod je standardan, reproducibilan i precizan. Korišćen je za istraživanje PAV-a (Lu et al., 2017), vode u koju je uronjen biofilm (Zhou et al., 2019), fizioloških rastvora (rastvori NaCl u vodi) i fosfatnih puferskih rastvora (Machala et al. 2013) ali nije primenljiv kod uzoraka koji su već obojeni (nisu bezbojni). Nedostatak ovog metoda je slaba osetljivost, zbog čega su u opsegu od 0 do 5 mg/l kao dodatna potvrda rezultata korišćene merene trake koje mogu da mere u opsegu od 0 do 25 mg/l (videti niže pasus *Kolorimetrijske trake*).

Nitriti, NO_2^-

Za određivanje koncentracije nitrita, korišćena je kolorimetrijska metoda (*Spectroquant Nitrite test 1.14776.0001, Merck, Darmštat, Nemačka*) zasnovana na reakciji nitrita sa Grisovim (eng. Griess) reagentom u kiselj sredini. Nitritni joni reaguju sa sulfanilinskom kiselinom ($\text{C}_6\text{H}_7\text{NO}_3\text{S}$) praveći diazonijevu so, koja reaguje sa N-(1-Naftil)etilenediamin dihidrohlorid-om pri čemu se stvara crveno-ljubičasta azotna boja koja ima maksimum apsorpcije na 525 nm.

Grisov reagent: sulfanilamide + N-(1-Naftil)etilenediamin dihidrohlorid + fosforna kiselina (Tornin et al. 2021, Bruggeman et al. 2016).

Kalibraciona kriva je napravljena sa sedam rastvora poznate koncentracije $\text{NO}_2\text{-N}$ (0,05, 0,1, 0,2, 0,4, 0,6, 0,8, 1 mg/l) dobijenih rastvaranjem standardnog rastvora (*Standardni rastvor nitrita, 40 mg/L $\text{NO}_2\text{-N}$ u H_2O , 125042, Merck, Darmštat, Nemačka*) u destilovanoj vodi.

Koeficijenti koji služe za preračunavanje koncentracije $\text{NO}_2\text{-N}$ u NO_2^- i obrnuto omogućavaju konvertovanje spektrofotometrijski izmerene koncentracije ($\text{NO}_2\text{-N}$) u traženu koncentraciju (NO_2^-) (Sigma Aldrich, 2017):

$$\text{NO}_2^- = \text{NO}_2\text{-N} * 3,285, \quad (2.3)$$

i obrnuto:

$$\text{NO}_2\text{-N} = \text{NO}_2^- * 0,304 \quad (2.4)$$

Napravljene kalibracione krive služe za merenje koncentracija NO_2^- u opsegu od 0,07 do 3,28 mg/L. Merenje je moguće samo u kiselim rastvorima (pH u opsegu 2 – 2,5). Ukoliko je pH vrednost tretiranog uzorka bila veća od toga, u uzorak je pre merenja dodavano 10 μl sumporne kiseline (Sumporna kiselina, $c = 0,5 \text{ mol/l}$, Sigma Aldrich, Nemačka).

Metod je vrlo precizan, reproducibilan i veoma je osetljiv što omogućuje merenja vrlo malih koncentracija. Korišćen je za analizu različitih tipova uzoraka tretiranih plazmom, kao što su destilovana voda i voda sa česme (Kučerová et al., 2021), puferski rastvori (Machala et al., 2019b), rastvori želatina (Labay et al., 2020), medijumi za ćelijske kulture (Tornin et al., 2021). Ograničenja ovog metoda su što nije primenljiv u (hemijski) kompleksnim matricama, reaktivan je sa mnogim biomolekulima, toksičan je i veoma zavisi od pH vrednosti pri čemu u kiselj sredini dolazi do reakcije nitrita sa vodonik peroksidom.

Nitrati, NO_3^-

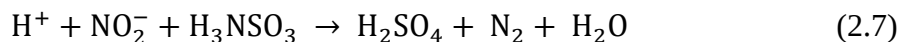
Za detektovanje NO_3^- korišćen je kolorimetrijski detekcioni metod (*Spectroquant Nitrate Test 1.09713.0002, Merck, Darmštat, Nemačka*) koji je zasnovan na nitriranju 2,6 dimetilfenola (poznatog i kao 2,6 ksileon) do stvaranja 4-nitro-2,6 dimetilfenola koji ima apsorpcioni maksimum na 357 nm, na kom su vršena merenja. Razblaživanjem standardnog rastvora $\text{NO}_3\text{-N}$ (*Standardni rastvor nitrata 200 mg/L $\text{NO}_3\text{-N}$ u H_2O , 125040, Merck, Darmštat, Nemačka*) u destilovanoj vodi napravljeno je deset rastvora poznate koncentracije $\text{NO}_3\text{-N}$ (0,1, 0,2, 0,5, 1, 2, 5, 10, 12,5, 20, 25) čime je dobijena kalibraciona kriva za merenje NO_3^- u opsegu od 0,4 – 110,7 mg/l. Kao i u slučaju merenja nitrita i ovde je bilo potrebno koristiti koeficijente za konverziju (Sigma Aldrich, 2017), kao u sledećim izrazima:

$$\text{NO}_3^- = \text{NO}_3\text{-N} * 4,43 \quad (2.5)$$

$$\text{NO}_3\text{-N} = \text{NO}_3^- * 0,226 \quad (2.6)$$

Metod zahteva da pH vrednost ispitivanog uzorka bude manja od 3, tako da je u uzorke sa manjom pH vrednošću dodavano 10 μl sumorne kiseline.

Glavni nedostatak ovog metoda je da nije selektivan kada su u pitanju joni azotnih oksida (NO_2^- i NO_3^-). Ukoliko u tretiranom uzorku ima NO_2^- bilo je potrebno dodavanje 10 μl 10% sulfaminske kiseline (ref) pre samog merenja, koja snižava pH vrednost uzorka i dovodi do pretvaranja NO_2^- u nereaktivan oblik:



Metod je do sada korišćen za detekciju nitrata u različitim tretiranim tečnostima: destilovanoj vodi (Lu et al. 2017), rečnoj vodi i otpadnim vodama (Montgomery et al. 1963), medijumima za ćelijske kulture (Tornin et al. 2021). Nedostatak metoda je što ne može da se koristi u slučajevima obojenih i kompleksnijih rastvora.

Kolorimetrijske trake

Semi-kvantitativna metoda za određivanje koncentracije ispitivane vrste u tečnom uzorku je korišćena za brzo određivanje opsega koncentracija H_2O_2 , NO_2^- , i NO_3^- pomoću kolorimetrijskih traka:

- *Peroksid test, 0,5 – 2 – 5 – 10 – 25 mg/l H_2O_2 , 1.1011.0001, Millipore, Kanada,*
- *Peroksid test, 0 – 100 – 200 – 400 – 600 – 800 – 1000 mg/l H_2O_2 , 1.10337, Millipore, Kanada,*
- *Nitrit test, 0 – 5 – 10 – 20 – 40 – 80 mg/l NO_2^- , 1.10007.0001, Millipore, Kanada,*
- *Nitrit test, 0,5 – 1 – 2 – 5 – 10 mg/l NO_2^- , 1.10057.0001, Millipore, Kanada,*
- *Nitrat test, 10 – 25 – 50 – 100 – 250 – 500 mg/l NO_3^- , 1.10020.0001, Millipore, Kanada,*

Merenja trakama su vršena pre kolorimetrijskih merenja spektrofotometrom, kako bi se uzorak po potrebi razblažio. Prednost ove metode je brzina, jednostavnost i širok opseg pH vrednosti u kojima je moguće merenje. Nedostaci su nepreciznost metode jer se koncentracija određuje upoređivanjem sa skalom nijansi boja pri čemu je moguće samo određivanje iz datih opsega koncentracija i netransparentost hemijskih procesa koji se dešavaju a koji izazivaju obojenje emulzije.

2.2.2 TEČNA HROMATOGRAFIJA VISOKIH PERFORMANSI (HPLC)

Hromatografija je analitička hemijska metoda koja služi za kvalitativno i kvantitativno proučavanje različitih uzoraka – smeša koje se sastoje iz većeg broja komponenata. Zasnovana je na razdvajanju komponenata smeše (molekula ili jona) uzorka između pokretne (mobilne) i nepokretne (stacionarne) faze. Postoje različiti tipovi hromatografije prema vrsti i polarnosti mobilne i stacionarne faze i mehanizmu razdvajanja. Jedna od najznačajnijih hromatografskih metoda koja se široko koristi zbog svoje osetljivosti, tačnosti i preciznosti jeste tečna hromatografija visokih performansi (eng. high performance liquid chromatography – HPLC). Cilj metode je da se u kratkom vremenskom periodu postigne visoka efikasnost razdvajanja, primenom pumpi koje održavaju visok pritisak i kontrolišu protok mobilne faze i interakcionim kolonama malog prečnika napunjenih stacionarnom fazom –česticama malog prečnika koji selektivno interaguju sa komponentama u tečnoj fazi. Selektivnost se pre svega ogleda u trajanju interakcije što uzrokuje Osnovni delovi tečnog hromatografa su: rezeorvari za mobilnu fazu, pumpa za visok pritisak, injektor (sistem za unošenje uzorka), interakcione kolone, termostatski blok, detektor i uređaj za sakupljanje, snimanje i obradu podataka (Odović et al., 2008).

Analiza produkata razgradnje pesticida malationa

Instrumentalnu metodu za određivanje proizvoda razgradnje malationa razvile su kolegice dr Svetlana Grujić i dr Ivana Matić Bujagić sa *Tehnološko-metalurškog fakulteta Univerziteta u Beogradu*. Malation je organosfosfatni pesticid koji je korišćen kao zagađivač pri ispitivanju uspešnosti prečišćavanja vode plazmom na atmosferskom pritisku. Polazni uzorak, rastvor malatona u destilovanoj vodi koncentracije 10 ppm, i uzorci tretirani plazmom su analizirani tečnim hromatografom uz korišćenje linearnog jonskog trapa kao masenog spektrometra (detektora u sistemu).

Proučavanje puteva razgradnje PFAS jedinjenja

Analitički metod za analizu ciljanih PFAS jedinjenja razvile su kolegice dr Mira Petrović i dr Barbara Topolovec sa *Katalonskog instituta za istraživanje voda (ICRA)*. Korišćeni metod je zasnovan na metodu opisanom u radu (Llorca et al., 2012), sa nekoiko izmena. Upotrebljen je 5500 QTRAP hibridni maseni spektrometar sa trostrukim kvadrupolom – linearni jonski trap (QqLIT-MS) sa jonskim raspršivačem kao izvorom (Applied Biosystems, Foster City, CA, USA) uparen sa tečnom hromatografijom (Waters Acquity Ultra-Performance™ liquid chromatograph (UPLC), Milford, MA, USA). Metoda tečne hromatografije visokih performansi uparena sa masenim spektrometrom Orbitrap Exploris 120 (OE120) primenjena je za identifikaciju mogućih produkata raspada svih ispitivanih jedinjenja. Detalji eksperimenta prikazani su u radu Topolovec et al., 2024.

3. DIJAGNOSTIKA GASNOG PRAŽNJENJA U OBLASTI kHz FREKVENCIJA NA ATMOSFERSKOM PRITISKU U KONTAKTU SA TEČNOM METOM

Proučavanje strimerskog pražnjenja u gasu na atmosferskom pritisku koje je u kontaktu sa tečnom metom obuhvatalo je:

- Električnu karakterizaciju pražnjenja (potpoglavlje 3.1) – gde su snimane strujno-naponske (I-V) karakteristike pražnjenja i korišćene su dve metode za računanje snage predate u električnom pražnjenju;
- Detekcija zračenja iz plazme (potpoglavlje 3.2) – koja je obuhvatila optičke dijagnostičke metode:
 - Optičku emisionu spektroskopiju za detekciju pobuđenih vrsta u plazmi i određivanje temperature gasa;
 - Snimanje kretanja strimerskog pražnjenja i snimanje grananja filamenata pri kontaktu sa tečnostima brzom kamerom.

3.1 ELEKTRIČNA KARAKTERIZACIJA STRIMERSKOG PRAŽNJENJA

Merenja u okviru električne karakterizacije izvora plazme obuhvatala su snimanje vremenski zavisnih strujnih i naponskih signala, određivanje I - V karakteristika pražnjenja i snage predate u pražnjenju pri različitim uslovima u sistemu. Ispitivani su različiti režimi pražnjenja kako bi se našli najoptimalniji uslovi za različite primene izvora plazme.

Eksperimentalna postavka i korišćene metode opisane su u potpoglavlju 2.1.1. Sva merenja urađena su na frekvenciji od 340 ± 10 kHz, dok su kao radni gasovi korišćeni He ili Ar, a protoci radnih gasova su bili 1, 1,5 ili 2 slm. Tokom svih merenja plazma mlaz je bio u kontaktu sa tečnom metom zapremine 4 ml. Merenja su izvršena kada je kao meta korišćen je komercijlno dostupan rastvor pesticida malationa (Galenika-Fitofarmacija a.d., 2011) u destilovanoj vodi, koncentracije 500 ppm (dalje u tekstu korišćen je izraz - **malation**) ili **destilovana voda**.

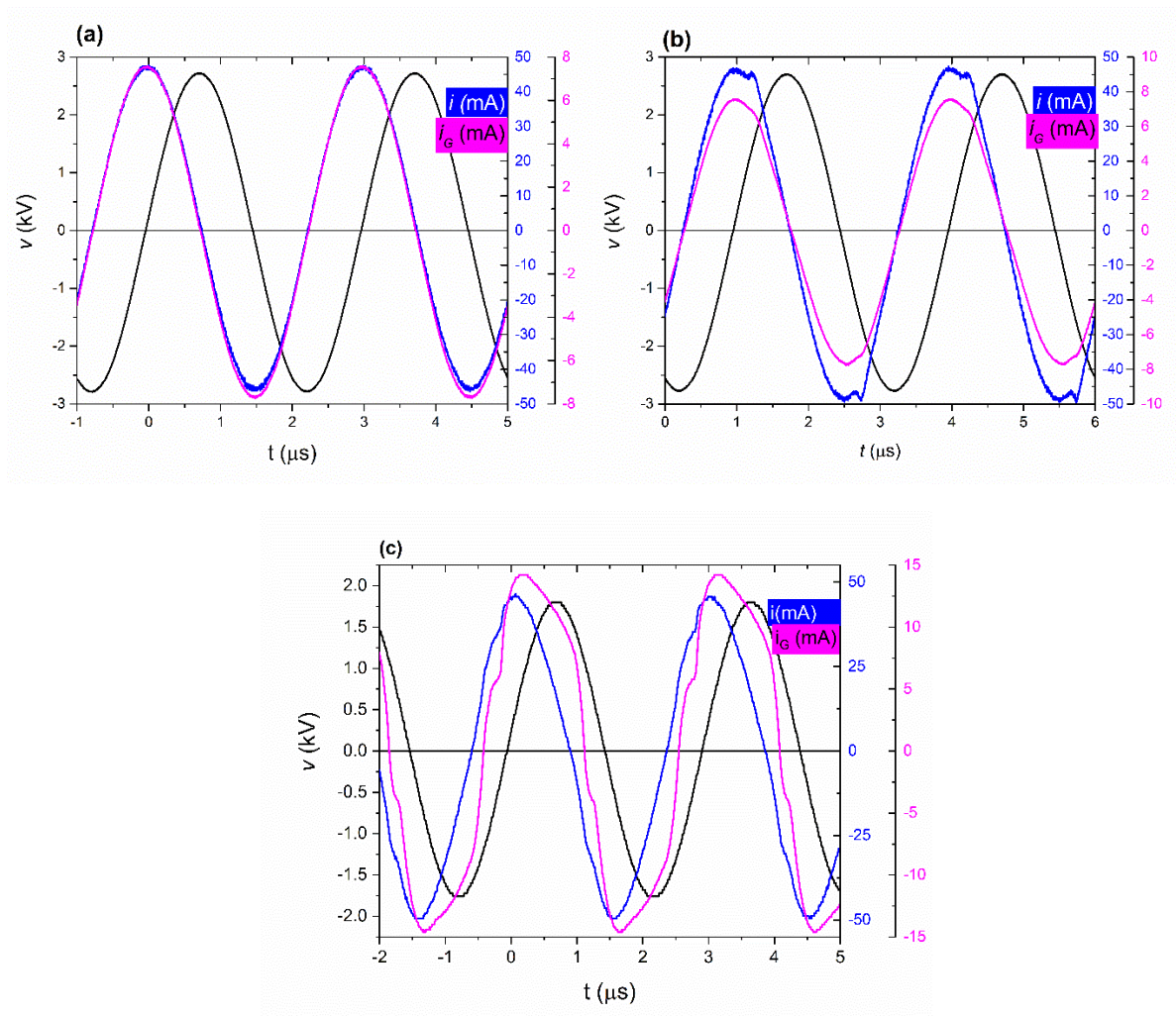
U potpoglavlju 3.1.1 prikazane su **I - V karakteristike** za dve struje merene u kolu – I_{rms} i I_{G_rms} . Prikazani su rezultati dobijeni variranjem sledećih parametara u sistemu:

- **rastojanja d** između napajane elektrode i površine tretirane tečnosti (radni gas He; $q = 2$ slm i bez protoka gasa; tečna meta - malation; $d = 5$ mm, 10 mm i 15 mm);
- **protoka radnog gasa q** (radni gas Ar; $q = 1$ slm, 1,5 slm i bez protoka gasa; tečna meta - malation; $d = 10$ mm);
- **vrste tečne mete** (radni gasovi He, $q = 2$ slm i Ar, $q = 1$ slm i bez protoka gasa; tečne mete - malation $\sigma = 12,5$ μ S/cm i destilovana voda $\sigma = 2$ μ S/cm, $d = 10$ mm).

Određivanje **srednjih snaga $\langle P \rangle$** (snaga predata plazma mlazu) i $\langle P_G \rangle$ (snaga predata u pražnjenju i u uzemljenoj grani kola) predstavljeno je u potpoglavlju 3.1.2. Određivanje snaga urađeno je za iste parametre u sistemu korišćene u potpoglavlju 3.1.1. Za određivanje snage $\langle P_G \rangle$ u uzemljenoj grani kola, primenjene su dve standardne metode u zavisnosti od merenog elementa povezanog u ovom delu kola. **V - I metoda** je primenjena kada je u uzemljenoj grani serijski vezan otpornik otpornosti $R = 1$ k Ω , a **Q - V metoda** kada je vezan kondenzator, za dve kapacitivnosti $Q = 0,8$ nF i $Q = 10,3$ nF.

VREMENSKI PROMENLJIVI STRUJNI I NAPONSKI SIGNALI

Prikaz vremenske zavisnosti snimljenog naponskog signala $v(t)$ i strujnih signala $i(t)$ i $i_G(t)$ dat je na slici 3.1 u slučajevima: **(a)** kada u sistemu nema protoka gasa, **(b)** sa protokom He od 2 slm i **(c)** sa protokom Ar od 1 slm. Prikazani signali su dobijeni usrednjavanjem 16 signala na osciloskopu. Odabrani su protoci gasova tako da strimersko pražnjenje u oba slučaja dodiruje površinu tečnosti. Zbog toga su prikazani rezultati za istu vrednost snage P_F od 17 ± 1 W sa izvora napajanja. Ovakvi uslovi bili su najčešći prilikom tretmana tečnosti u delu istraživanja sa primenama plazma mlaza (poglavlje 4). Prikazani su rezultati dobijeni korišćenjem malationa kao tečne mete. Korišćenjem destilovane vode kao mete, nisu primećene značajne promene u obliku signala, usled male razlike u električnoj provodljivosti ovih uzoraka.



Slika 3.1: Primeri vremenski promenljivih signala primenjenog visokog napona $v(t)$, struje kroz plazma mlaz $i(t)$ i struje koja protiče kroz uzorak i uzemljenu granu kola $i_G(t)$, u slučaju: a) kada u sistemu nema radnog gasa tj. bez plazme, b) pražnjenja u He (protok 2 slm) i c) pražnjenja u Ar (protok 1 slm). Signali su snimljeni kada je plazma mlaz u kontaktu sa vodenim rastvorom malationa zapremine 4 ml, a rastojanje između elektrode i tečnosti $d=10$ mm.

U slučaju kada u sistemu nema protoka gasa (slika 3.1a), svi snimljeni signali imaju sinusoidni oblik, a fazna razlika između struja i napona iznosila je $90^\circ \pm 5^\circ$ što ukazuje da je sistem dominantno kapacitivan. Mala odstupanja od potpuno kapacitivne impedanse sistema (kada je fazna razlika tačno 90°) postoji usled prisustva parazitne otpornosti elemenata električnog kola. Odstupanja od 90° ukazuju na gubitke u kolu, koji postoje čak i kada u sistemu nema električnog pražnjenja u gasu.

U prisustvu pražnjenja (slika 3.1b i 3.1c) dolazi do deformacije strujnih signala u oblasti maksimalnih i minimalnih vrednosti i pojave pikova, što ukazuje na nelinearnost sistema usled uključivanja pražnjenja. Prisustvo pikova kod strujnih signala ukazuje na strimerski tip pražnjenja. Dobijene promene u signalu su primećene u ranijim istraživanjima kod drugih atmosferskih plazma mlazeva (Chang et al., 2018; Darny et al., 2017; Li et al., 2021; Maletić et al., 2015; Puać et al., 2012).

Uočena deformacije u talasnom obliku strujnih signala bila je rezultat diskretne propagacije pražnjenja izvan gasne cevi u obliku brzih jonizacionih paketa (eng. Pulsed Atmospheric pressure Plasma Streamer - PAPS). Ove promene nastaju zato što se struja napajanja plazma mlaza preklapa (superponira) sa strujom pomeranja (eng. displacement current). Struja pomeranja (kapacitivna) se direktno meri kada cev nije ispunjena radnim gasom i kada nema plazme.

Nakon što dođe do proboja, u slučaju pražnjenja u helijumu (**3.1b**) fazna razlika ostaje gotovo ista, ukazujući da je veoma malo snage predato u samom pražnjenju. Sa druge strane, kod pražnjenja u argonu (**3.1c**) došlo je do promene faznih razlika struja $i(t)$ i $i_G(t)$ u odnosu na primenjeni napon $v(t)$, koje su nakon proboja u gasu iznosile 66° i 41° , respektivno. Procenjena greška merenja fazne razlike iznosi 2° na osnovu tri nezavisna merenja za istu radnu tačku. Pojavom pražnjenja u gasu došlo je do značajne promene impedanse sistema koja je pored kapacitivne komponente dobila i rezistivnu komponentu. Značajna promena fazne razlike pokazuje da je veći deo snage predate izvoru plazme predat kako u pražnjenju tako i u samom uzorku koji je deo uzemljene grane kola.

3.1.1 STRUJNO – NAPONSKE KARAKTERISTIKE

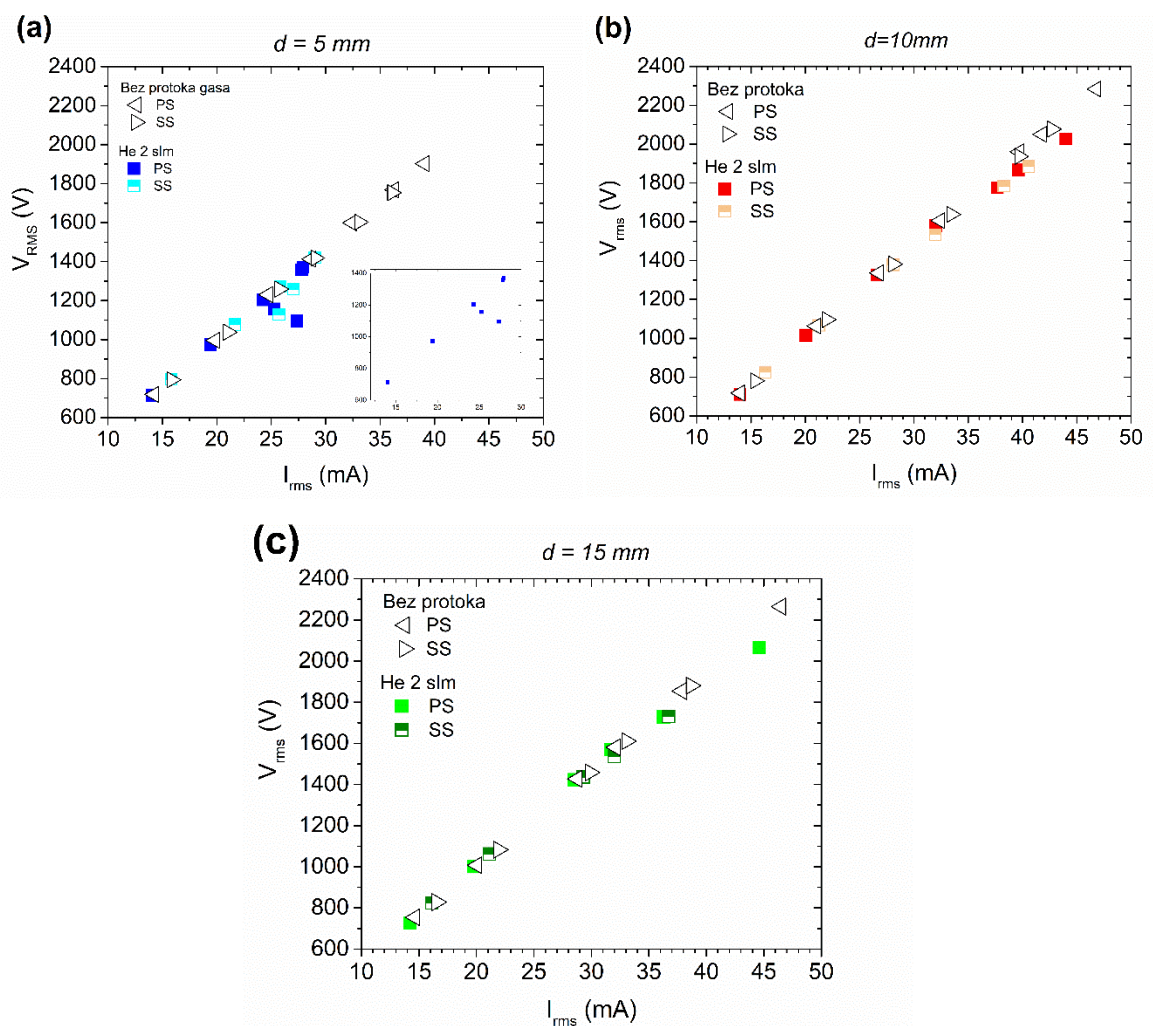
I - V karakteristike su predstavljene za dve merene struje u električnom kolu plazma mlaza. Iz dobijenih vremenski zavisnih signala sa osciloskopa preračunate su odgovarajuće RMS vrednosti, prema izrazima **1.6** i **1.7** i prikazane su zavisnosti $V_{rms} = f(I_{rms})$ i $V_{rms} = f(I_{G\_rms})$. Snimanje I - V karakteristika urađeno je u režimima sa i bez protoka radnog gasa u sistemu. Urađena su merenja kada se snaga koja se dovodi plazma mlazu (P_F) povećava i kada se smanjuje, kako bi se videla pojava histerezisa u sistemu. Električne karakteristike pražnjenja su proučavane pri različitim radnim uslovima u sistemu – korišćenjem različitih radnih gasova i protoka gasova kao i menjanjem rastojanja između napajane elektrode i površine tečnosti. Sva merenja su izvršena u tri ponavljanja, pri čemu uočene razlike u dobijenim vrednostima nisu prelazile 7 % u odnosu na srednju vrednost.

STRUJA NAPAJANJA PLAZMA MLAZA I_{rms}

Variranje rastojanja d između napajane elektrode i površine tretirane tečnosti

Na slici **3.2** prikazane su I - V karakteristike za tri rastojanja d (mm) od 5 mm, 10 mm i 15 mm, u slučajevima kada u sistemu nema protoka gasa (simbol trougla) i kada je kao radni gas korišćen He sa protokom od 2 slm (simbol kvadrata). Snimanjem istih radnih tačaka u slučajevima kada se snaga na napajanju povećava i smanjuje, nije primećena značajna promena u radnim tačkama u sva tri slučaja.

Merenje I - V karakteristike kada nema protoka radnog gasa omogućuje određivanje impedanse kola koje uključuje izvor plazme, kapacitivnosti mete i međuelektrodnog rastojanja i gubitke u sistemu (parazitske kapacitivnosti kablova i priključaka). U sva tri slučaja kada u sistemu nema pražnjenja i protoka gasa dobijena je ista linearna zavisnost $V_{rms} = f(I_{rms})$, a iz nagiba linearnog fita izmerenih tačaka dobijena je vrednost impedanse kola od $|\hat{Z}_{NP}| = 48,0 \pm 0,3 \text{ k}\Omega$. Pod pretpostavkom da se u slučaju bez protoka gasa u sistemu ponaša uglavnom kapacitivno (dobijena fazna razlika između struje i napona je 90°), dobijenoj vrednosti impedanse odgovara kapacitivnost od 9,8 pF. U literaturi koja se bavi modelovanjem ekvivalentnih električnih kola sa izvorom plazme, pokazano je da se ona može predstaviti serijskom vezom kapacitivnosti elemenata kola (Lin et al., 2021; Bastin et al., 2023). Kapacitivnost međuelektrodnog rastojanja (vazdušnog sloja) predstavlja jednu od komponenti, koja se menja promenom rastojanja d , ali se u ovom slučaju pokazalo da uticaj na promenu ukupne impedanse nije značajan u korišćenom opsegu rastojanja d .



Slika 3.2: I - V karakteristika pražnjenja za struju I_{rms} , u slučajevima bez protoka gasa i pražnjenja u He pri protoku od 2 slm, za tri različita rastojanja između napajane elektrode i tečnosti ($d = 5, 10, 15$ mm). Snimljene su I - V karakteristike kada se snaga na napajanje povećava (PS) i kada se smanjuje (SS).

Za najkraće rastojanje od $d = 5$ mm, kada je u sistemu bilo protoka radnog gasa, u opsegu V_{rms} napona od 700 V do 1200 V nije bilo vidljivog pražnjenja. I - V karakteristika u ovom delu imala je linearni oblik i dobijene su radne tačke koje leže na pravoj koja ima isti nagib kao u slučaju kada nema protoka gasa. U tački kada je napon dostigao vrednost $V_{rms}=1200$ V došlo je do električnog proboja u gasu, strimerski kanal se odmah pri uspostavljanju pražnjenja spojio sa površinom mete, ali je pražnjenje bilo slabog intenziteta. Do vrednosti napona od 1370 V i struje od 28 mA, I - V karakteristika je zadržala linearni oblik. Fazna razlika između struje I_{rms} i napona u ovoj radnoj tački iznosila je 85° . Sa daljim povećanjem snage na napajanje plazma mlaza, došlo je do pada napona i struje (1160 V, 25 mA) i smanjenja fazne razlike na vrednost 70° . Osim promene u amplitudi strujnog signala uočeno je i veće izobličenje strujnog kanala. U poslednjoj snimljenoj radnoj tački, napon je pao na vrednost od 1100 V, ali je ponovo zapažen porast struje I_{rms} na vrednost od 27 mA. Fazna razlika između struje i napona iznosila je 65° .

Pri promeni oblika I - V karakteristike iz linearnog u nelinearni, vizuelno je uočena promena oblika pražnjenja što je opaženo kroz proširivanje prečnika strimerskog kanala pražnjenja i intenzivniju emisiju zračenja. Pojavili su se dodatni strimerski kanali koji su se račvali od centralnog kanala, formirajući konusnu površinu u blizini dodira pražnjenja sa površinom tečnosti. Uočene promene u snimljenim naponskim i strujnim signalima, kao i vizuelno primećene promene u strukturi pražnjenja ukazuju na to da je došlo do promene režima pražnjenja i promene impedanse celog kola.

Yoon i saradnici (2017) su ispitivali promenu režima pražnjenja u He, koristeći sličnu geometriju izvora plazme koje je napajan signalom frekvencije u oblasti kHz. Korišćena je metoda snimanja emisije zračenja plazme i emisije H_{β} iCCD kamerom i merenjem brzine strimera. Pokazali su da se na rastojanju 1 mm iznad tečne mete (elektrolit čija je površina na rastojanju 10 mm od napajane elektrode) dolazi do prelaska oblika plazme iz plazmenog paketa prstenastog oblika u pražnjenje nalik strimerskom (eng. pin-jet streamer). Ovakvo ponašanje su objasnili time da u oblasti 1 mm iznad mete dolazi do formiranja sloja vlažnog vazduha usled isparavanja tečne mete. Time dolazi do promene mehanizma jonizacije u pražnjenju iz dominantno Peningove jonizacije molekula N_2 metastabilima helijuma, u direktnu jonizaciju molekula vode elektronskim udarom. Dodatno su pokazali da akumulacija slobodnog naelektrisanja na površini mete i promena temperature tečnosti ubrzavaju prelazak u strimerski režim pražnjenja i formiranje novih filamenata u ovoj oblasti.

Kersten i saradnici (2023) su u istraživanju plazma mlaza u DBD konfiguraciji pratili formiranje filamentarnog pražnjenja brzim kamerom i predložili model koji objašnjava uočenu promenu režima iz stacionarnog u „locked“ režim. U „locked“ režimu dolazi do rotacije filamenata oko vertikalne ose plazma mlaza i uočeno je da povećanje snage pražnjenja utiče na brzinu rotiranja filamenata.

Na većim rastojanjima plazma mlaza od mete, $d = 10$ mm i $d = 15$ mm, I - V karakteristika zadržava linearnu zavisnost napona od struje i oni istovremeno rastu sa postepenim povećanjem snage na napajanju. Na ovim rastojanjima I - V karakteristika je linearnog oblika bez obzira da li u sistemu ima protoka radnog gasa ili ne.

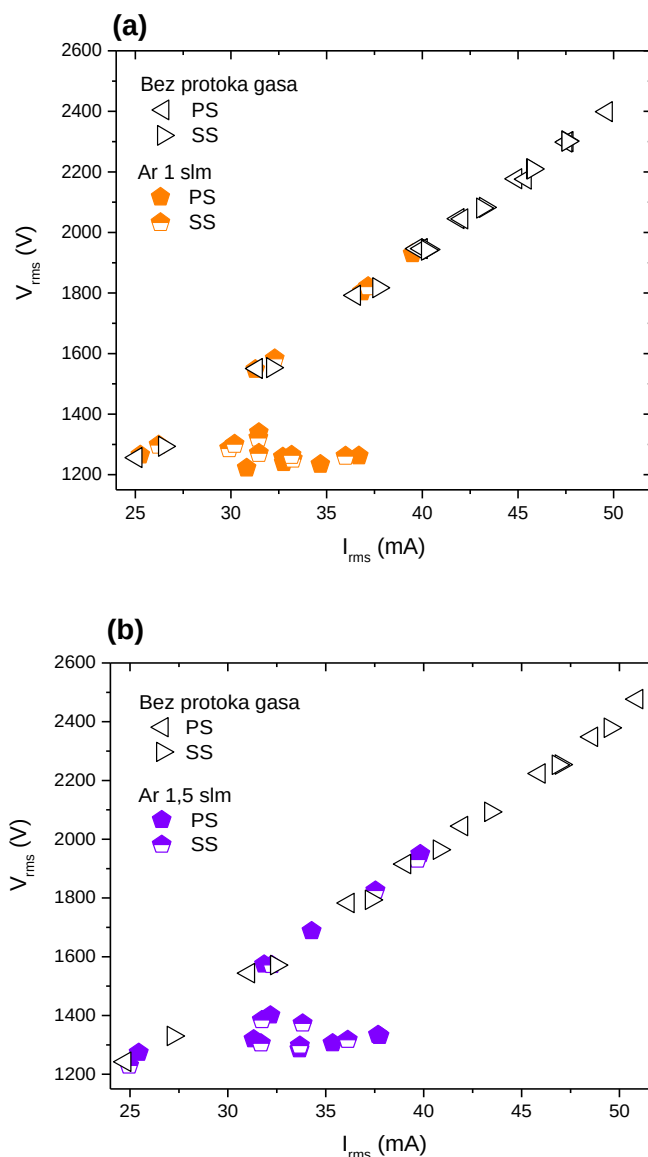
Za ova dva rastojanja d , linearne zavisnosti $V_{rms} = f(I_{rms})$ sa i bez protoka gasa su gotovo identične u radnim tačkama pre spajanja strimerskog kanala sa površinom mete, odnosno do vrednosti struje od 33 mA i napona od 1600 V. Nakon toga se primećuje da I - V karakteristika kada ima pražnjenja leži ispod karakteristike kada nema protoka gasa. Merenjem impedanse kola u sistemu kada ima protoka gasa i pražnjenja dobijene su vrednosti od $44,0 \pm 1,5$ k Ω za oba rastojanja d , što znači da se sa pojavom plazme ukupna impedansa sistema nije značajno promenila, odnosno da je uticaj impedanse plazme na ukupnu impedansu mali.

Kada je u sistemu postojao protok gasa, pri naponima manjim od 1300 V u sistemu nije bilo pražnjenja. Nakon uspostavljanja pražnjenja, ono je prvo uočeno samo pri vrhu elektrode, izlazilo je iz staklene cevčice, ali se nije spojilo sa metom. Daljim povećavanjem snage koja se predaje plazma mlazu, pražnjenje se produžavalo i pri naponu od oko 1750 V i struji od 37 mA (peta tačka) dodirivalo površinu tečnosti. Strimerski kanal bio je uzak i stabilan sa malim konusnim proširenjem u oblasti neposredno pre površine tečnosti, a intenzitet emisije je rastao sa povećanjem snage na napajanju.

Urađeno je i snimanje I - V karakteristike u slučaju kada se snaga koja se predaje izvoru plazme sa napajача (P_F) smanjuje, što je na graficima na slici 3.2 označeno sa SS (smanjenje snage). Rezultati su pokazali da za sva tri rastojanja d , nije primećena pojava histerezisa u sistemu i da se odgovarajuće snimljene tačke u slučajevima PS i SS poklapaju, u okviru greške merenja.

Variranje protoka radnog gasa

Ispitivanje električnih karakteristika kada se u sistemu koriste različiti protoci argona, izvršeno je kako bi se našli najoptimalniji uslovi za primenu plazma mlaza. Merenja su urađena za dva protoka gasa od 1 slm i 1,5 slm i na rastojanju $d = 10$ mm. U slučaju pražnjenja u argonu, veće promene u protoku radnog gasa i međuelektrodnog rasojanja nisu bile moguće jer se nije moglo uspostaviti stabilno pražnjenje. Na slici 3.3 prikazane su I - V karakteristike za struju I_{rms} za protoke argona od: **(a)** 1 slm **(b)** 1,5 slm, kada je kao tečna meta korišćen malation. Prikazana su i merenja bez protoka gasa (simbol trougla) merenja kada se u sistemu povećava snaga na napajanju (PS) i kada se smanjuje (SS).



Slika 3.3: I - V karakteristika pražnjenja za struju I_{rms} , u slučajevima bez protoka gasa i pražnjenja u Ar pri protocima od 1 slm i 1,5 slm, za rastojanja između napajane elektrode i tečnosti $d=10$ mm. Snimljene su I - V karakteristike kada se snaga na napajanju povećava i kada se smanjuje.

I-V karakteristika kada nema protoka gasa ranije je prikazana na slici **3.2b**. Pri protoku gasa od 1 slm (**3.3a**), povećavanjem snage koja se predaje sa napajача P_F rastu i struja i napon, ali se pražnjenje ne uspostavlja do vrednosti napona od 1950 V i struje od 40 mA. *I-V* karakteristika se preklapa sa onom u slučaju kada nema protoka gasa. Sa daljim povećanjem snage P_F i pojavom pražnjenja, dolazi do pada vrednosti i napona i struje na vrednosti od 1350 V i 32mA, respektivno. Pražnjenje se odmah pri uspostavljanju prostire duž celog rastojanja d i dodiruje površinu tečnosti. Dalje povećanje snage na napajачu dovodi do povećanja struje I_{rms} (od 31 mA do 37 mA), a napon je prvo opao na vrednost oko 1250 V ostao gotovo konstantan.

Daljim povećanjem snage P_F (rezultati nisu prikazani), vizuelno je primećena promena strukture pražnjenja. Za sve izmerene radne tačke sa pražnjenjem na *I-V* karakteristici, dobijen je strimerski kanal roze-ljubičaste boje koji se širi pri kontaktu sa tečnošću i sa povećanjem snage na napajanju postaje sve intenzivniji i širi. Pri predatim snagama sa napajanja P_F , većim od 21 W, oblik pražnjenja prelazi u uzak beli strimerski kanal, koji se odlikuje strujom veće amplitude. Međutim, rad u ovom režimu dovodi do značajnog isparavanja tečnosti u kratkom vremenskom intervalu čime se menja rastojanje d , a samim tim i radni uslovi pražnjenja. Do isparavanja tečnosti dolazi usled prenosa energije iz pražnjenja u tečnost, koji se ostvaruje preko prenosa mase (udarom čestica iz pražnjenja o površinu vode) i toplote (Bruggeman et al., 2016). Povećavanje struje pražnjenja znači da se povećava količina čestica iz pražnjenja koje dolaze u kontakt sa površinom vode, usled čega više vode isparava. Ovaj proces je detaljnije opisan u potpoglavlju **4.2.1**, gde je proučavana promena temperature i zapremine uzorka tokom tretmana plazmom. Zbog toga je *I-V* karakteristika snimana isključivo u prvom režimu rada pražnjenja, do maksimalne snage P_F od 21 W i nije snimana za veće snage predate plazma mlazu, iako je moguće uspostaviti pražnjenje i u tim radnim tačkama.

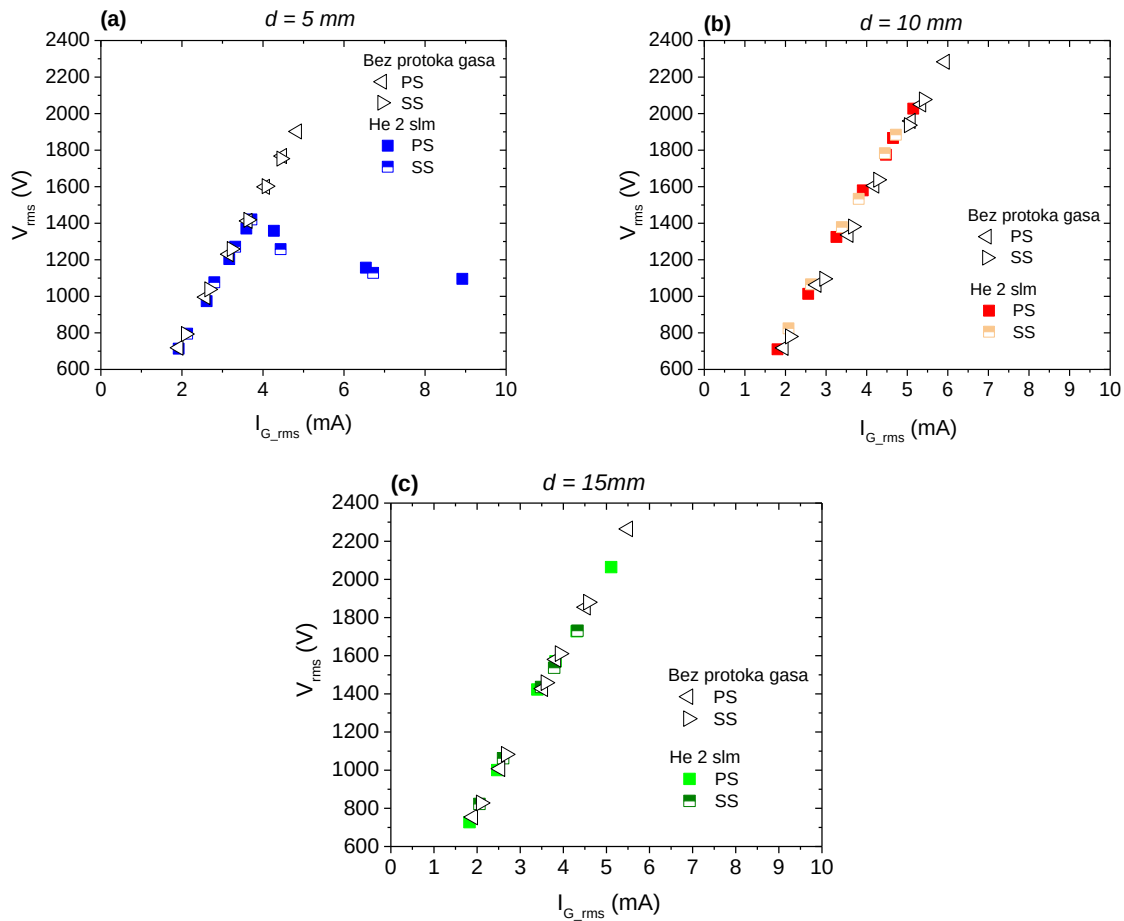
Primećene promene u obliku strimerskog pražnjenja u argonu kao što je sužavanje strimerskog kanala, promena boje i povećana emisija zračenja sa porastom struje pražnjenja, ranije je opisana u literaturi (Bruggeman et al., 2009; Lim et al., 2020). U radu Lim i saradici (2020) su proučavali argonsko pražnjenje u kontaktu sa destilovnom vodom, gde su koristili sličnu geometriju izvora plazme, sinusnu pobudu frekvencije 50 kHz i primenjene rms napone u opsegu od 1,8 kV – 2,2 kV. Pokazali su da sa porastom snage koja se predaje plazma mlazu raste struja dok napon ostaje konstantan u okviru primenjenih napona, što je posledica smanjene impedanse kola usled formiranja provodnog kanala. Za napone iznad ovog opsega (rms napon veći od 2,3 kV), primećeno je da dolazi do povećanja koncentracije elektrona i OH molekula i emisije zračenja O i Ar linija.

Za veći protok gasa od 1,5 slm dobijena je vrlo slična *I-V* karakteristika kao u slučaju protoka od 1 slm. Razlika je u tome što je pražnjenje teže uspostaviti, jer pri istim vrednostima primenjenog napona ne dolazi uvek do proboja, a kada se uspostavi pražnjenje ono osciluje po površini tečnosti i česti su prelazi između dva gore opisana oblika strimerskog pražnjenja.

STRUJA PRAŽNJENJA MERENA U UZEMLJENOJ GRANI KOLA I_{G_rms}

Variranje rastojanja d između napajane elektrode i tretirane tečnosti

Grafik zavisnosti $V_{rms}=f(I_{G\_rms})$, za tri različita rastojanja d (mm), za pražnjenje u He pri protoku od 2 slm prikazano je na slici 3.4. Kada u sistemu nema protoka gasa, snimljene su I - V karakteristike u opsegu primenjenog napona od 700 V do 2200 V (1900 V u slučaju rastojaje $d = 5$ mm). Dobijena je linearna zavisnost napona od struje, bez obzira na rastojanje d . Primećene su male razlike u nagibu I - V karakteristika za različita rastojanja d , ali su one u okviru greške merenja. Snimanjem karakteristike kada se u kolu pojačava i smanjuje snaga na napajaju (simbol trougla, PS i SS), dobijeni su isti rezultati u okviru greške merenja, za sva tri rastojanja d .



Slika 3.4: I - V karakteristika pražnjenja za struju I_{G_rms} , u slučajevima bez protoka gasa i pražnjenja u He pri protoku od 2 slm, za tri različita rastojanja između napajane elektrode i tečnosti ($d=5,10,15$ mm). Kao meta korišćen je malation. Snimljene su I - V karakteristike kada se snaga na napajanju povećava (PS) i kada se smanjuje (SS).

Pri protoku He od 2 slm (simbol kvadrata na slici 3.4), kao i u slučaju struje I_{rms} , primećena je razlika u obliku I - V karakteristika za rastojenje $d = 5$ mm i druga dva rastojanja. Kod rastojanja $d = 5$ mm, do vrednosti napona od 1370 V, I - V karakteristika je imala linearan oblik i u potpunosti se preklapala sa krivom kada nema protoka gasa. Povećanjem snage na napajaču, došlo je do vizuelne promene pražnjenja i promene oblika I - V karakteristike iz linearnog u nelinearni. Pri merenju struje I_{G_rms} , sa povećanjem snage predate u sistem, primećeno je da odmah dolazi do rasta struje (sa 3,6 mA na 4,3 mA) dok vrednost napona ostaje gotovo konstantna. U slučaju struje I_{rms} (slika 3.2a), na ogovarajućem mestu na I - V karakteristici došlo je do preklapanja dve radne tačke i nije primećen rast struje. Daljim povećanjem snage na napajanje, struja I_{G_rms} je nastavila da raste do vrednosti od 9 mA, dok je napon opadao do vrednosti od 1100 V (slika 3.4a). Merenjem I - V karakteristike kada se snaga na napajanje smanjuje (označeno sa SS), primećeno je postojanje histerezisa u kolu. Za istu primenjenu snagu na napajanje, snimljene su radne tačke na nižim primenjenim naponima od onih u slučaju PS, u nelinearnim delu I - V karakteristike. U linearnom delu I - V karakteristika one leže na istoj pravoj liniji kao u slučaju PS.

Na većim međuelektrodnim rastojanjima od 10 mm i 15 mm, dobijena je linearna I - V karakteristika i u slučaju sa protokom radnog gasa i pražnjenjem i u slučaju bez protoka gasa. Iz linearnog fita je izračunata impedansa u slučaju kada nema protoka gasa od 397 ± 8 k Ω i 423 ± 4 k Ω , za rastojanja 10 mm i 15 mm, respektivno. Krive sa i bez protoka radnog gasa, za isto rastojanje d , imaju gotovo isti nagib i preklapaju se u okviru greške merenja. Slične I - V karakteristike zabeležene su kod drugih plazma mlazeva koji rade na različitim frekvencijama, geometrijama elektroda i sa različitim metama (Gerling et al., 2017; Maletić et al., 2017).

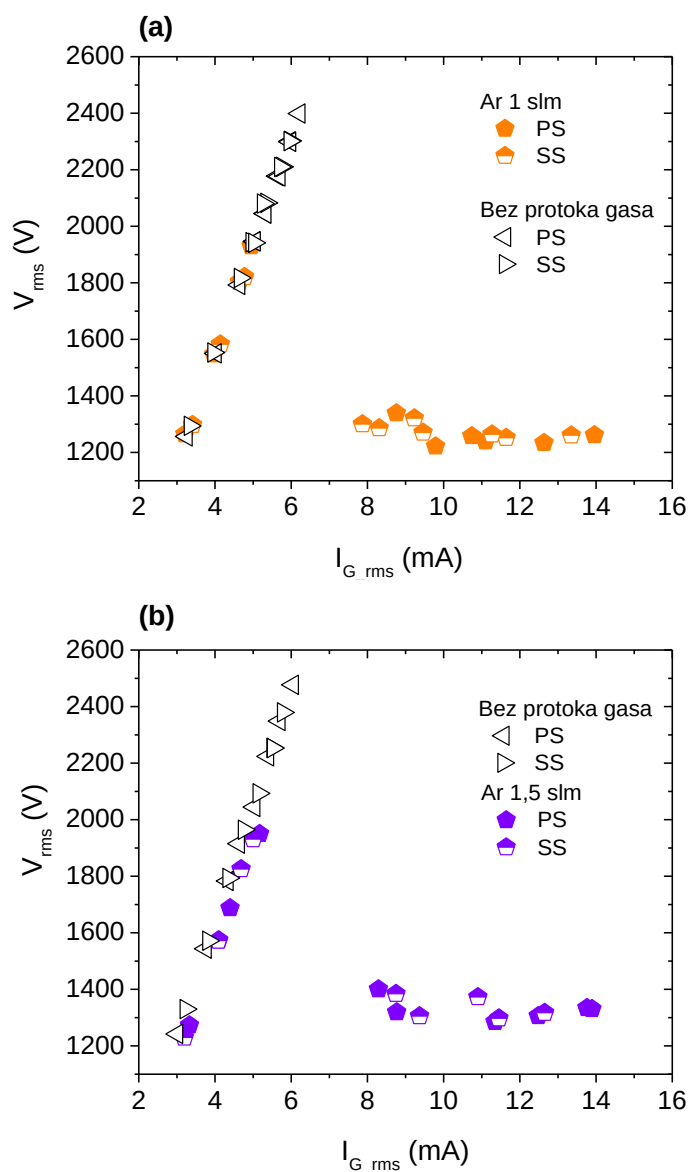
U slučaju većih rastojanja d , naksimalne dostignute struje pražnjenja su bile oko 5 mA pri naponu od 2000 V. Kod rastojanja od $d = 5$ mm, nakon pojave pražnjenja dolazi do smanjenja radnog napona i povećanja struje pražnjenja koja je dostigla vrednost od 9 mA, što je skoro duplo veća vrednost nego maksimalna izmerena struja kod većih rastojanja. Ovakva razlika nastala je zbog uspostavljanja jačeg strimerskog pražnjenja sa većom strujom pražnjenja, jer su na malom međuelektrodnom rastojanju radialni gubici nosioca naelektrisanja difuzijom iz provodnog kanala manji (Papadakis, 2013). Na većim rastojanjima (10 mm i 15 mm), ni u ovom delu kola pojava plazme nije uticala na nagib krive i promenu impedanse sistema. Dobijene struje su za red veličine manje od odgovarajućih struja I_{rms} , što znači da je samo deo struje struje iz plazma mlaza prosleđen u metu i registrovan kao struja pražnjenja.

Variranje protoka radnog gasa

Kod pražnjenja u argonu (slika 3.5), nije primećena velika razlika u izmerenim strujama I_{G_rms} za različite protoke gasa (1 slm i 1,5 slm). Podsetimo, u oba slučaja pražnjenje je dodirivalo površinu tečnosti. Deo I - V karakteristike kada je protok gasa bio 1 slm ali nema pražnjenja (za napone manje od 1930 V), poklapa se sa krivom kada nema pražnjenja (slika 3.5a). Izmerena impedansa bila je 391 ± 1 k Ω . U slučaju većeg protoka od 1,5 slm dobijena je impedansa od 382 ± 2 k Ω .

Nakon pojave pražnjenja, došlo je do pada napona na vrednost od 1300 V i 1400 V, za protoke od 1 slm i 1,5 slm, respektivno. Dalje povećanje snage na napajanje dovodilo je samo do povećanja struje pražnjenja I_{G_rms} , dok je vrednost napona ostala gotovo konstantna, što je ranije primećeno kod struje I_{rms} . Najveća izmerena struja u oba slučaja bila je oko 14 mA.

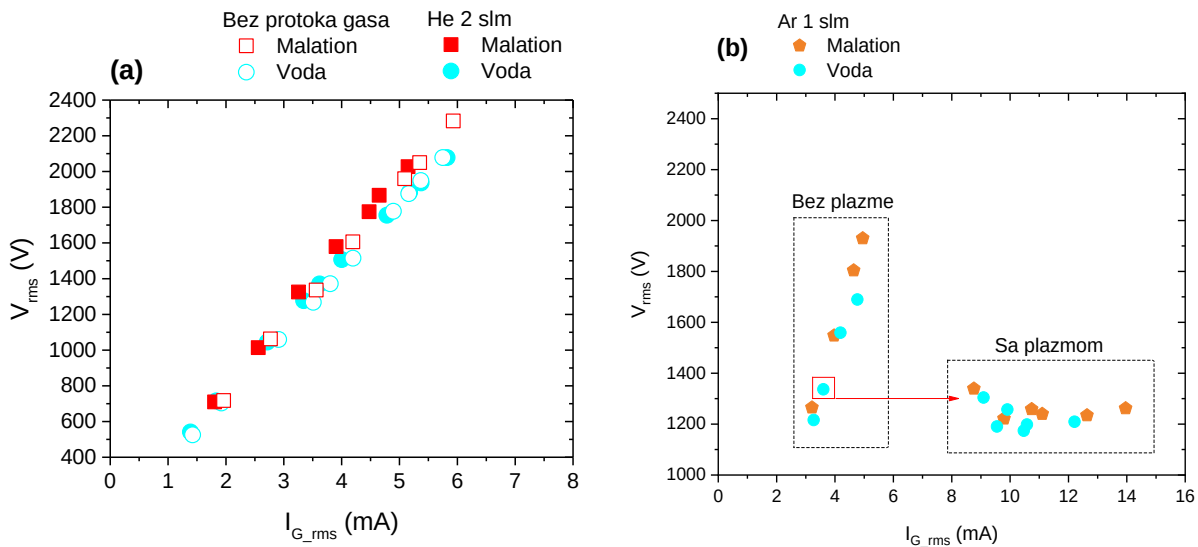
Merenja u oba smera – sa povećanjem i smanjenjem snage (označeno sa PS i SS) nisu pokazala razliku u slučaju bez protoka gasa (simbol trougla) i u slučaju kada ima protoka gasa, ali nema pražnjenja. Kod dela I - V karakteristika kada je prisutno pražnjenje, primećeno je blago odstupanje snimljenih radnih tačaka, što je posledica manjih fluktuacija vrednosti napona i struje sa promenom snage na napajanje P_F .



Slika 3.5: I - V karakteristika pražnjenja za struju I_{G_rms} , u slučajevima bez protoka gasa i pražnjenja u Ar pri protocima od 1 slm i 1,5 slm, za za rastojanja između napajane elektrode i tečnosti $d = 10$ mm. Snimljene su I - V karakteristike kada se snaga na napajanju povećava i kada se smanjuje.

Uticaj tečne mete

Kako bi se ispitaio uticaj osobina tečne mete na pražnjenje, urađena je i I - V karakterizacija sa metama koje su bile od značaja u okviru dela istraživanja primena plazma mlaza. Poređenje rezultata dobijenih sa rastvorom pesticida **malationa** ($\sigma = 12,5 \mu\text{S/cm}$) i **destilovanom vodom** ($\sigma = 2 \mu\text{S/cm}$) prikazano je na slici 3.6 za rastojanje $d = 10 \text{ mm}$ u slučajevima kada nema protoka gasa, kod pražnjenja u **helijumu** ($q=2 \text{ slm}$) (3.6a) i u **argonu** ($q=1 \text{ slm}$) (3.6b). Rezultati za pražnjenje kada je meta bila rastvor malationa, prikazani su i na slikama 3.4b i 3.5a, gde su detaljnije diskutovani. Ovde su opisani u svrhi poređenja sa slučajem kada su svi parametri pražnjenja isti osim tipa tečne mete.



Slika 3.6: I - V karakteristika za struju $I_{G,\text{rms}}$ za različite tipove tečne mete – vodeni rastvor pesticida malationa koncentracije 500 ppm i destilovane vode. (a) Bez protoka gasa i u slučaju pražnjenja u He ($q = 2 \text{ slm}$) i (b) za pražnjenje u Ar ($q = 1 \text{ slm}$). Uokvirena tačka (crveni kvadrat) na grafiku (b) predstavlja tačku sa plazmom.

I - V karakteristike bez protoka gasa i sa protokom **helijuma** u sistemu su linearnog oblika (videti sliku 3.6a) i gotovo se preklapaju, za istu tečnu metu. Kao što je ranije uočeno, pojava pražnjenja nema značajan uticaj na promenu impedanse sistema, bez obzira na korišćenu metu. Dobijene impedanse su bile $364 \pm 1 \text{ k}\Omega$ za destilovanu vodu i $397 \pm 8 \text{ k}\Omega$ za malation.

Uočena je blaga razlika nagiba I - V karakteristika za pražnjenje u He za različite mete. Karakteristika dobijena sa destilovano vodom se nalazi ispod krive za malation, odnosno za istu vrednost struje $I_{G,\text{rms}}$ pražnjenje je postignuto za niži primenjeni napon. Druga razlika je primećena u tome što kada je meta destilovana voda, pražnjenje postoji u celom opsegu primenjenih napona od 500 V do 2200 V. U slučaju malationa, do pojave pražnjenja je došlo pri vrednosti napona od 1300 V, kada je vrednost struje bila 3,4 mA. Počevši od ove radne tačke, dolazi do blagog razdvajanja I - V karakteristika za različite mete, ali u okviru maksimalne procenjenije greške merenja koja za mereni napon iznosi 90 V, može se reći da nema razlike za ove dve tečne mete.

Kod pražnjenja u **argonu** (slika **3.6b**), povećanjem snage na napajanju prvo rastu i struja i napon, ali za vrednosti struja I_{G_rms} između 3 mA i 5 mA nije bilo pražnjenja kod obe mete. I - V karakteristike za vodu i malation se u ovom delu poklapaju. Izmerene impedanse kada nema pražnjenja su bile $365 \pm 5 \text{ k}\Omega$ i $391 \pm 1 \text{ k}\Omega$, za vodu i malation respektivno. Međutim, kao i u slučaju kod helijuma, bili su potrebni niži naponi za uspostavljanje pražnjenja u slučaju vode (1700 V) nego u slučaju malationa (1950 V). Nakon uspostavljanja pražnjenja, kod obe mete dolazi do brzog pada napona na istu vrednost od oko 1350 V.

U slučaju vode, pražnjenje se javlja samo na vrhu elektrode i radna tačka se nalazi u delu karakteristike označene sa „Bez plazme“. U ovoj tački došlo je do pada i napona i struje. Vrednost struje bila je 3,6 mA. Ove karakteristike odgovaraju korona režimu pražnjenja, koje ne dovodi do promene impedanse sistema. Dalje povećanje snage dovodi do prelaska u strimerski režim pražnjenja. Sa druge strane, u slučaju kada je meta malation, do formiranja strimerskog kanala dolazi odmah pri pojavi pražnjenja. Kod oba slučaja, usled formiranja provodnog strimerskog kanala struja naglo poraste na vrednost oko 9 mA. Stalan porast struje sa povećanjem snage na napajaču, ispoljavao se kroz širenje strimerskog kanala. Istovremeno, napon se ustalio na vrednosti od oko 1200 V. U ovom delu I - V karakteristike označene izrazom „Sa plazmom“ na grafiku **3.6b**, u okviru greške merenja nema velike razlike u vrednosti struja I_{G_rms} za različite tečne mete.

3.1.2 SREDNJE SNAGE PREDATE U KOLO SA PLAZMA MLAZOM

U okviru ovog dela prikazani su rezultati merenja dve snage: srednje snage predate plazma mlazu $\langle P \rangle$ i srednje snage oslobođene u pražnjenju $\langle P_G \rangle$ - snage koja je merena u uzemljenoj grani kola i oslobođena je u pražnjenju i u uzorku. Snaga $\langle P \rangle$ računata je korišćenjem V - I metoda opisanog u poglavlju **1.2.1**, iz direktno izmerenih vrednosti visokog napona $v(t)$ i struje $i(t)$. Za merenje snage $\langle P_G \rangle$ korišćena su dva standardna metoda V - I i Q - V (poglavlje **1.2.1**), u zavisnosti od mernog elementa (otpornika ili kondenzatora) koji je bio u uzemljenoj grani kola.

Merenja snage urađena su u slučajevima kada u sistemu ima protoka gasa (sa protokom), kao i kada nema (bez protoka) i izračunata je njihova razlika (NET):

$$\langle P_{NET} \rangle = \langle P_{sa\ protokom} \rangle - \langle P_{bez\ protoka} \rangle \quad (3.1)$$

Merenje snage $\langle P_{bez\ protoka} \rangle$ je značajno jer ona predstavlja gubitke koji nastaju u kolu zbog postojanja parazitskih otpornosti različitih elemenata kola i ona je uvek uključena u izmerenu snagu $\langle P_{sa\ protokom} \rangle$. Snaga $\langle P_{NET} \rangle$ predstavlja srednju snagu koja je predata u odgovarajućem delu kola: ili na plazma mlazu ili u samom pražnjenju, tj. u uzemljenom delu kola. Pronađeno je da je standardna greška merenja za snagu $\langle P_{NET} \rangle$ bila 0,2 W za RMS vrednosti primenjenog napona ispod 1,5 kV.

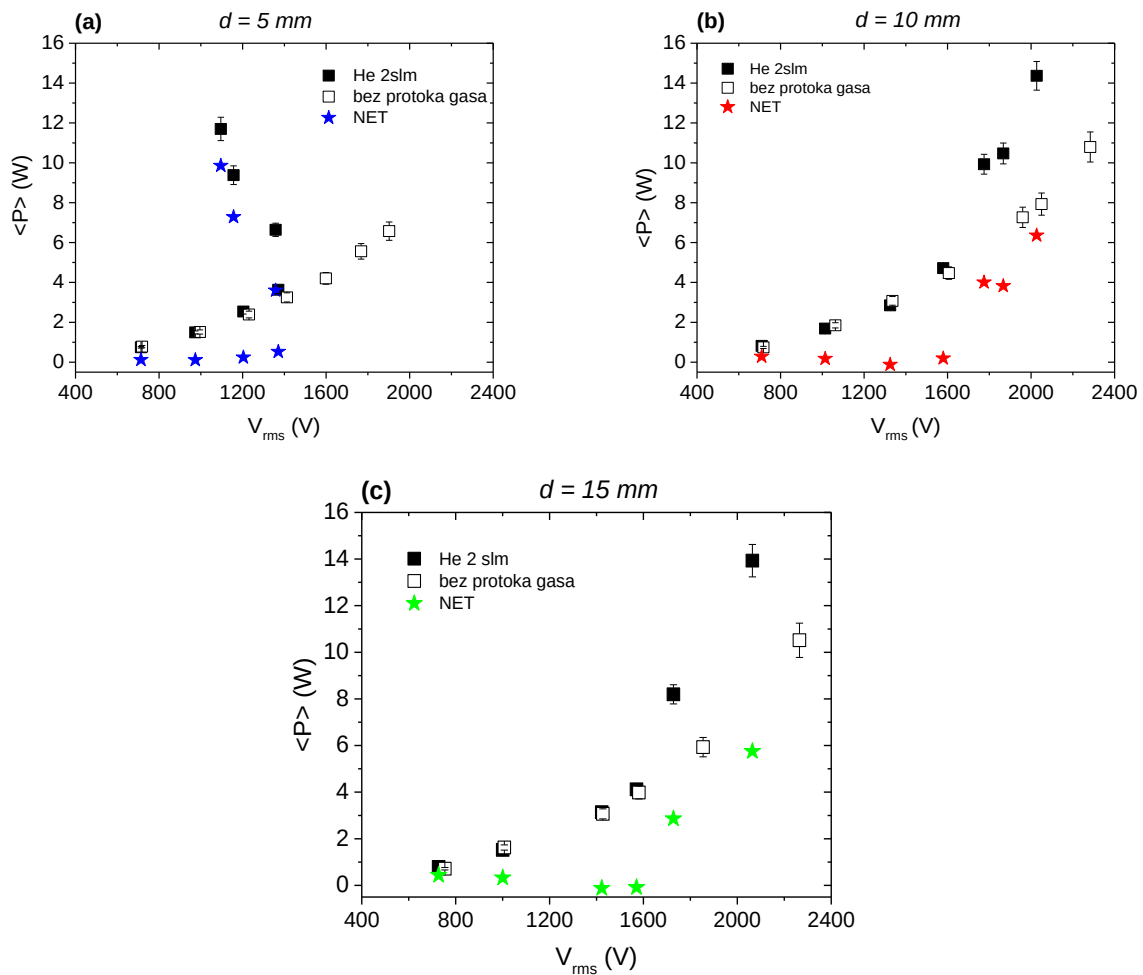
Rezultati merenja su pokazali da snage $\langle P_{bez\ protoka} \rangle$ rastu sa primenjenim naponom V_{rms} i mogu se fitovati polinomskom funkcijom $y = Ax + Bx^2$. Fitovanjem dobijenih rezultata na ovu funkciju zavisnosti mogu se odrediti gubici snage $\langle P_{bez\ protoka} \rangle$ za bilo koji primenjeni napon V_{rms} .

SREDNJA SNAGA PREDATA PLAZMA MLAZU $\langle P \rangle$

Variranje rastojanja d između napajane elektrode i tretirane tečnosti

Na slici 3.7 prikazana je zavisnost srednje snage predate plazma mlazu $\langle P \rangle$ u zavisnosti od primenjenog napona V_{rms} , u slučaju kada je kao radni gas korišćen **He 2 slm** i u slučaju bez protoka gasa, za malation i tri različita rastojanja d .

Merenje snage $\langle P_{bez\ protoka} \rangle$ pokazalo je da se sa povećanjem napona značajan deo snage gubi na samom izvoru plazme i za primenjene napone V_{rms} do 2,2 kV dostiže vrednost od 7,5W. Primećena razlika između izmerenih snaga verovatno je posledica povećanih gubitaka u prenosu signala od izvora ka plazmi koji se javljaju pri većim vrednostima primenjenih napona. Krive zavisnosti snage $\langle P_{bez\ protoka} \rangle$ od napona V_{rms} za tri različita rastojanja d su se poklapale u okviru greške merenja.



Slika 3.7: Srednja snaga predata plazma mlazu $\langle P \rangle$ u zavisnosti od RMS vrednosti primenjenog napona, za tri različita rastojanja između napajane elektrode i tečnosti ($d = 5, 10, 15\text{ mm}$). Srednje snage su snimljene u režimu sa protokom He od 2 slm i bez protoka radnog gasa i izračunata je razlika te dve vrednosti $\langle P_{NET} \rangle$.

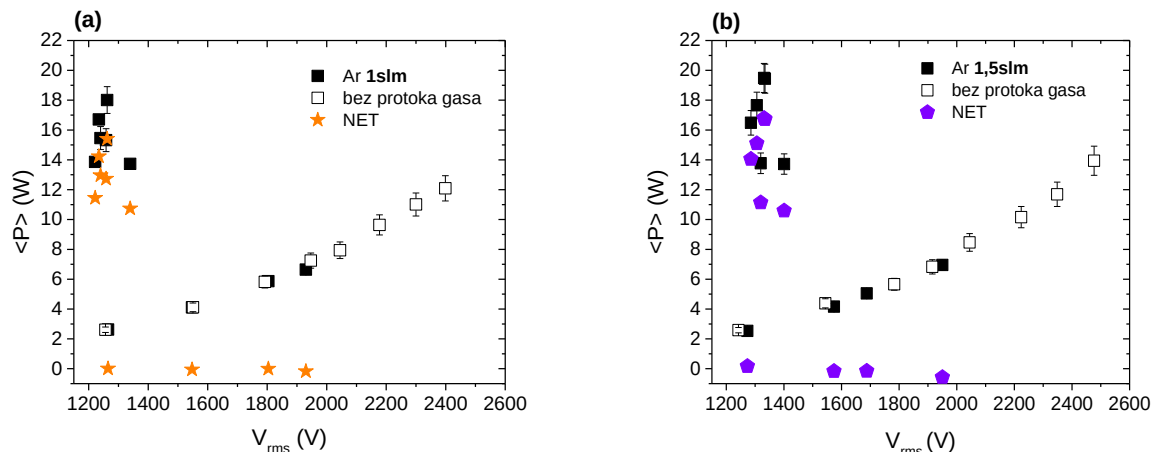
Rezultati dobijeni za snagu $\langle P_{sa\ protokom} \rangle$ za rastojanje $d=5$ mm (**3.7a**), razlikovali su se od vrednosti koje su dobijene na druga dva rastojanja, kao i kod I - V karakteristika. Vrednost snage $\langle P_{sa\ protokom} \rangle$ rasla je sa povećanjem napona do vrednosti od $V_{rms}=1370$ V, do trenutka kada I - V karakteristika pražnjenja prelazi iz linearnog u nelinearni oblik. Usled promene režima pražnjenja koji se dešava u ovoj radnoj tački, došlo je do pada vrednosti napona, ali je snaga $\langle P_{sa\ protokom} \rangle$ rasla sa snagom predatom sa napajanja. Kako je impedansa kola u promenjenom režimu rada pražnjenja sasvim drugačija od slučaja za $d=10$ mm i $d=15$ mm, snaga ima drugačiju zavisnost od napona. Ovo je posledica toga što su signali struje i napona drugačijeg oblika i radne tačke kola nalaze se u sasvim drugim oblastima I - V karakteristike.

U opsegu napona u kojima su urađena merenja, najveća izmerena vrednost snage $\langle P_{sa\ protokom} \rangle$ bila je oko 12 W za $d=5$ mm (**3.7a**), odnosno oko 14 W za rastojanja 10 mm i 15 mm (slike **3.7b** i **3.7c**). Preračunavanjem snage $\langle P_{NET} \rangle$, pokazano je da je njena vrednost 0 W do vrednosti napona od $V_{rms} = 1370$ V, odnosno do promene režima pražnjenja na rastojanju $d = 5$ mm. Na većim rastojanjima, njena vrednost je bila bliska nuli do vrednosti napona od 1600 V, odnosno do momenta spajanja pražnjenja sa površinom tečne mete. Nakon uočenih promena u sistemu, dolazi do rasta vrednosti snage $\langle P_{NET} \rangle$ sa porastom snage koja se sa napajanja predaje izvoru plazme. Za sva tri rastojanja d , razlika između snage $\langle P_{sa\ protokom} \rangle$ i $\langle P_{NET} \rangle$ je rasla sa porastom vrednosti napona V_{rms} . U opsegu primenjenih napona V_{rms} , najviša izmerena vrednost snage $\langle P_{NET} \rangle$ bila je 10 W, 6,5 W i 6 W za rastojanja d od 5 mm, 10 mm i 15 mm, respektivno.

Variranje protoka radnog gasa

Na slici **3.8** prikazani su rezultati dobijeni za pražnjenje u **argonu**, pri različitim protocima gasa (**1 slm i 1,5 slm**), za malation i rastojanje $d=10$ mm. Podaci dobijeni za snagu $\langle P_{bez\ protoka} \rangle$, bili su isti kao i oni prikazani na slici **3.7**. Merenja su pokazala da promena protok gasa ne utiče značajno na vrednosti snage $\langle P \rangle$. Za oba korišćena protoka, nakon što dođe do pojave plazme i pada napona (ustali se na vrednosti oko 1200 V), snaga $\langle P_{sa\ protokom} \rangle$ sporo raste sa postepenim povećavanjem snage na napajanju. Ove snage su nakon pojave pražnjeja bile su u opsegu od 14 W, do 18 W za protok 1 slm, odnosno do 19,5 W za protok 1,5 slm. U oba slučaja za maksimalnu izmerenu snagu $\langle P_{sa\ protokom} \rangle$ izračunato je da $\langle P_{NET} \rangle$ iznosi 85 % te vrednosti. Promena protoka radnog gasa nije dovela do promena u snazi predatoj plazma mlazu.

Poređenjem dobijenih grafika zavisnosti $\langle P \rangle$ od V_{rms} i vrednosti snaga $\langle P_{NET} \rangle$ dobijenih za pražnjenje u argonu (slika **3.8**) i helijumu (**3.7b**) za rastojanje $d = 10$ mm, primećuje se da je efikasnost prenosa snage sa napajanja u sistem plazma mlaza i samo pražnjenje efikasnije u slučaju argona. Rezultati dobijeni za argon su sličniji onima dobijenim za dvostruko manje rastojanje $d = 5$ mm kod pražnjenja u helijumu (slika **3.7a**).



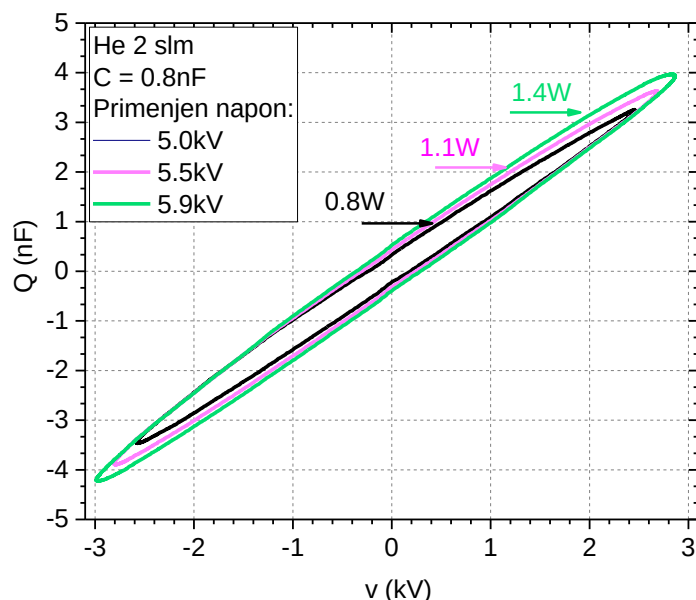
Slika 3.8: Srednja snaga predata plazma mlazu $\langle P \rangle$ u zavisnosti od RMS vrednosti primenjenog napona, za različite protoke Ar: 1 slm i 1,5 slm. Srednje snage su snimljene u režimu sa i bez protoka radnog gasa i izračunata je razlika te dve vrednosti $\langle P_{NET} \rangle$.

SREDNJA SNAGA PREDATA U PRAŽNJENJU $\langle P_G \rangle$

Poređenje rezultata dve primenjene metode za računanje snage (V-I i Q-V)

Računanje snage predate u pražnjenju urađeno je pomoću dve standardne metode (V-I i Q-V). U prvoj metodi korišćen je otpornik otpornosti 1 k Ω , koji je ubačen serijski sa tečnim uzorkom u uzemljenu granu kola. Tada je za računanje snage $\langle P_G \rangle$ korišćen izraz **1.8** (poglavlje **1.2.1**). Kada je u uzemljenoj grani kola bio kondenzator, za računanje snage je korišćen izraz **1.9**. Urađena su nezavisna merenja sa dva kondenzatora kapacitivnosti 0,8 nF i 10,3 nF.

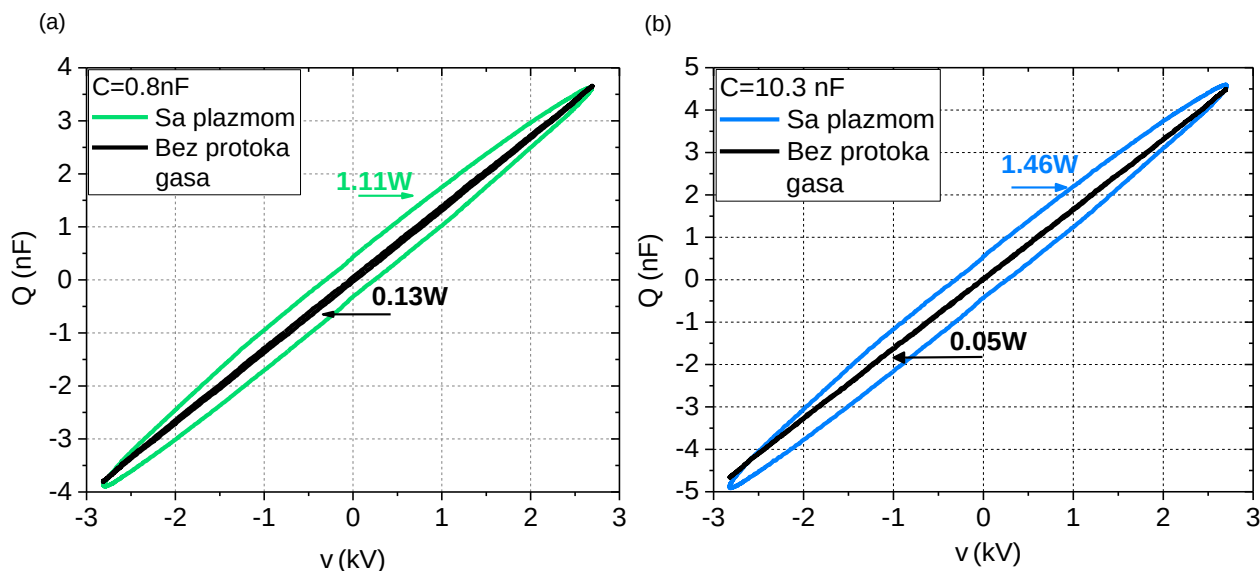
Grafik zavisnosti naelektrisanja koje protiče kroz merni kondenzator kapacitivnosti **0,8 nF** u toku jednog perioda naponskog signala, od primenjenog napona v (Lissajous-ova figura), za tri različite vrednosti primenjenog napona (peak-to-peak) od 5 kV, 5,5 kV i 5,9 kV. prikazan je na slici **3.9**. Dati rezultati su dobijeni za pražnjenje u **He**, kada je protok gasa bio **2 slm**, meta je bila destilovana voda i rastojanje **d=10 mm**.



Slika 3.9: Lissajous-ova figura i izmerene snage $\langle P_{G\_sa\ protokom} \rangle$ za pražnjenje u He (protok 2 slm, $d = 10$ mm), za tri primenjena (peak-to-peak) napona (5 kV, 5,5 kV, 5,9 kV) i frekvenciju naponskog signala od 330 kHz. Merni element u uzemljenoj grani je bio kondenzator kapacitivnosti 0,8 nF.

Pošto obe veličine – naelektrisanje Q i radni napon v osciluju istom frekvencijom, dobijena Lissajous-ova figura ima eliptični oblik. Ovakav oblik je u prethodnim istraživanjima dobijen kada struja protiče duž celog perioda primenjenog visokonaponskog signala (Peeters & Butterworth, 2019; Xiong et al., 2010). Sa slike 3.9 se vidi da Lissajous-ova figura zadržava isti oblik, ali menja nagib (nagib prave linije koja povezuje tačke minimalne i maksimalne vrednosti naponskog signala) sa promenom napona. Centar elipse se poklopio sa tačkom 0,0 koordinatnog sistema.

Kao i u slučaju računanja snage $\langle P \rangle$ i ovde su računate snage kada u sistemu ima protoka radnog gasa i kada nema, kako bi se procenili gubici koji potiču od elemenata u kolu. Slika 3.10 prikazuje Lissajous-ove figure i odgovarajuće snage $\langle P_{G\_sa\ protokom} \rangle$ (He 2 slm) i $\langle P_{G\_bez\ protoka} \rangle$ izmerene sa dva različita kondenzatora kapacitivnosti 0,8 nF i 10,3 nF, za primenjeni (peak-to-peak) napon od 5,5 kV i frekvenciju signala od 330 kHz.



Slika 3.10: Lissajous-ova figura i izmerene snage kada u sistemu protiče 2 slm He (zelena i plava figura) i bez protoka radnog gasa (crne figure), za primenjeni peak-to-peak napon od 5,5 kV frekvencije 330 kHz i rastojanje $d=10$ mm. Korišćeni su kondenzatori od a) 0,8 nF i b) 10,3 nF.

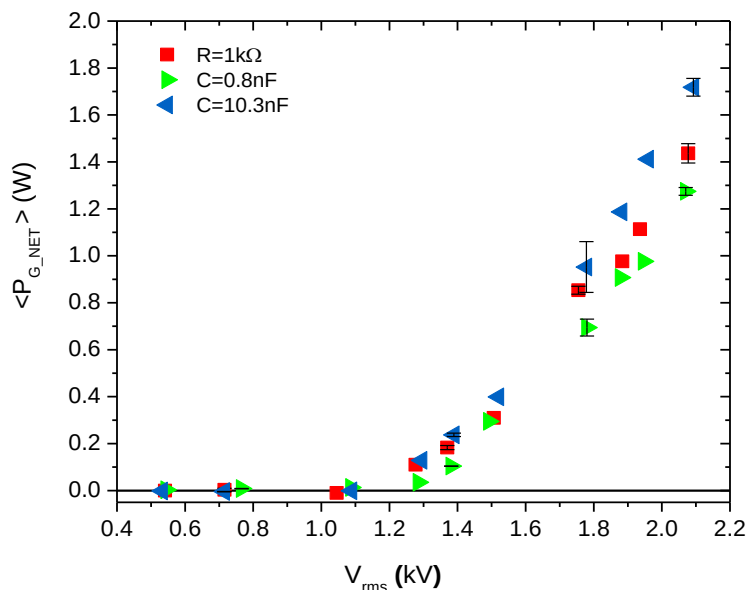
Poređenjem rezultata dobijenih iz dva nezavisna merenja, primećeno je da je sa kondenzatorom od 10,3 nF dobijena za oko 23% veća vrednost snage $\langle P_{G,sa\ protokom} \rangle$, nego u slučaju kada je mereno sa kondenzatorom od 0,8 nF. Kondenzator u električnom kolu utiče na vrednost impedanse u uzemljenj grani kola. Ukoliko se impedansa kondenzatora aproksimira vrednošću njegove reaktanse ($X_C = \frac{1}{2\pi fC}$) pri radnoj frekvenciji od $f=330$ kHz, ona za kondenzatore kapacitivnosti $C = 0,8$ nF i $C = 10,3$ nF iznosi 597Ω i 46Ω , respektivno. Razlika u izmerenim snagama $\langle P_{G,sa\ protokom} \rangle$ sa dva različita kondenzatora, može biti uzrokovana njihovim različitim karakteristikama ili prilagođavanju plazme različitim elementima kola (Ashpis et al., 2017; Gerling et al., 2017; Wang et al., 2021).

Merenja snage $\langle P_{G,bez\ protoka} \rangle$ sa oba kondenzatora su pokazala da se u odsustvu plazme (i protoka gasa), dobija nenulta površina figure – koja ima oblik izdužene elipse. Q-V dijagram bez protoka gasa bi teorijski trebao da bude prava linija, kao što je pretpostavljeno u drugim istraživanjima sa različitim izvorima plazme (Biganzoli et al., 2014; Pipa et al., 2012). Postojanje izdužene elipse kada nema protoka gasa (i nema plazme) može biti rezultat tehničkih problema kao što su neispravne žičane veze ili nekompenzovane sonde (Peeters i Butterworth, 2019). U našem slučaju, ove nepredviđene situacije su proverene i isključene. Dakle, disipacija snage u režimu bez protoka gasa predstavlja parazitski otpor koji je prisutan u sistemu i određena snaga se troši u ovom delu kola i bez postojanja plazme. Ovakvi gubici su primećeni i kod merenja snage $\langle P \rangle$ (Slika 3.7b).

Poređenjem grafika na slici 3.10, može se primetiti da su gubici snage kada je merenje urađeno sa kondenzatorom od 10,3 nF za red veličine manji od gubitaka dobijenih sa kondenzatorom od 0,8 nF. Za oba kondenzatora je u odsustvu plazme dobijeno da Lissajous-ova figura ima izdužen oblik sa oštrim vrhovima u tačkama maksimalnog napona, što ukazuje na to da su količina naelektrisanja koja protiče kroz kondenzator i primenjeni napon u fazi i da dostižu ekstremnu vrednost u isto vreme. Kada dođe do pojave plazme, Lissajous-ove figure postaju zaobljene u krajnjim tačkama ukazujući na dodatne omske gubitke u kolu iako napon na krajevima kondenzatora $v_G(t)$ i visoki napon $v(t)$ ostaju u fazi.

Iz toga se može zaključiti da pojava plazme ne utiče na takvo povećanje impedanse ovog dela kola koje bi dovelo do promene faze između dva merena napona. Povećanje površine Lissajous-ove figure sa pojavom plazme ukazuje da se u sistemu pojavljuje dodatna disipacija energije, što sugerise da snaga $\langle P_{G\_sa\ protokom} \rangle$ dobijena kada u sistemu ima plazme, sadrži i snagu predatu u pražnjenju i snagu disipiranu u sistemu. Zbog toga je i u ovom slučaju računata snaga $\langle P_{G\_NET} \rangle$, kao razlika snaga dobijenih pomoću Lissajous-ovih figura dobijenih sa i bez protoka radnog gasa, za isti primenjeni napon $v(t)$.

Da bi se uporedili rezultati dobijeni sa dve metode (V-I i Q-V), na slici 3.11 prikazana je zavisnost snage $\langle P_{G\_NET} \rangle$ od primenjenog napona V_{rms} , za tri merna elementa u uzemljenoj grani kola: **$R = 1\text{ k}\Omega$, $C = 0,8\text{ nF}$ i $C = 10,3\text{ nF}$** . Dobijene snage $\langle P_{G\_NET} \rangle$ leže u opsegu od 0 W do 1,7 W, za V_{rms} napone u opsegu između 0,5 kV i 2 kV. U poređenju sa rezultatima dobijenim za snagu $\langle P_{NET} \rangle$, primećeno je da je manje od 30% snage $\langle P_{NET} \rangle$ oslobođene u pražnjenju prosleđeno u uzemljenu granu kola. Sa slike 3.11 se može videti dobro slaganje rezultata dobijenih sa različitim mernim elementima.



Slika 3.11: Srednja snaga oslobođena u pražnjenju $\langle P_{G\_NET} \rangle$ u zavisnosti od RMS vrednosti primenjenog napona, pri protoku He od 2 slm i rastojanje $d = 10\text{ mm}$, za tri različita merna elementa postavljena u uzemljenu granu kola.

Prikazane greške merenja predstavljaju standardne greške izračunate za tri nezavisna merenja. U opsegu V_{rms} napona od 0,5 kV do 1,1 kV, razlike snaga u prisustvu i odsustvu pražnjenja su bliske nultoj vrednosti. Međutim, u slučajevima kada ima protoga gasa, plazma je mogla da se opazi kao mala svetla oblast na vrhu napajane elektrode čak i u ovim radnim tačkama (korona pražnjenje). Stoga se može zaključiti da su stvarne vrednosti oslobođenih snaga u ovim slučajevima ispod granice osetljivosti korišćenih metoda.

Prednosti i mane primenjenih metoda za merenje snage

Jedan od izazova pri merenju snage predstavljaju neželjeni gubici u električnom kolu, koji mogu uticati na preciznost merenja snage, zbog čega je njihov uticaj pažljivo analiziran i minimizovan kako bi se obezbedila ponovljivost merenja. Gubici u sistemu plazma mlaza nastaju u spojevima elemenata kola, kablovima i mernoj opremi (posebno kada se približava opsegu visokih frekvencija). U uzemljenoj grani kola, primećeno je da svaka promena u spojevima, kao što su kablovi, konektori ili izabran način povezivanja sa uzemljenjem utiču na izmerene vrednosti. Da bi se umanjili gubici energije i osigurali reproducibilni rezultati, potrebno je pre merenja odabrati kablove i konktore odgovarajuće dužine i kvaliteta.

Kod primene Q-V metoda, najveće poteškoće pri merenju na koje treba obratiti pažnju su: odabir odgovarajućih kondenzatora, korišćenje odgovarajućih kablova za uzemljenje kondenzatora i pojava čvorova ili diskontinuiteta kod Lisažuovih figura. Kada je u pitanju odabir odgovarajućeg kondenzatora primećeno je da kondenzatori iste kapacitivnosti ali različite vrste, dovode do različitih rezultata merenja. Primećeno je da korišćenje film kondenzatora dovodi do nefizičkih rezultata. Na primer, upotrebom metalizovanog poliesterskog (PET) kondenzatora kapacitivnosti 10 nF, izmerene su veće oslobođene snage kada nema električnog gasnog pražnjenja u kolu nego kada ga ima. Pored toga je dolazilo do samopresecanja Q-V krive u oblasti gde napon i količina naelektrisanja menjaju znak. Ovi rezultati sugerisu da PET kondenzatori ne bi trebalo da se koriste u Q-V tehnici merenja snage, barem ne pri visokim frekvencijama signala.

Kapacitivnost kondenzatora treba da bude odabrana u skladu sa kapacitivnošću sistema plazma mlaza, odnosno treba odabrati kondenzator koji može da skladišti ukupno naelektrisanje koje prođe kroz pražnjenje tokom jednog poluperioda primenjenog napona. Ovo se može ispuniti ako je kapacitivnost kondenzatora najmanje jedan red veličine veća od kapacitivnosti izvora plazme. Zbog toga su u ovom radu izabrani kondenzatori čija je kapacitivnost za tri reda veličine veća od kapacitivnosti plazma mlaza.

Kako veze u uzemljenoj grani kola u velikoj meri utiču na ponovljivost merenja, male promene u položaju kabla između uzemljene elektrode i mernog kondenzatora mogu izazvati promene u nagibu i širini Q-V krive eliptičnog oblika. Za merenje treba izabrati odgovarajući kabl koji ne utiče na karakteristike Lisažuove figure.

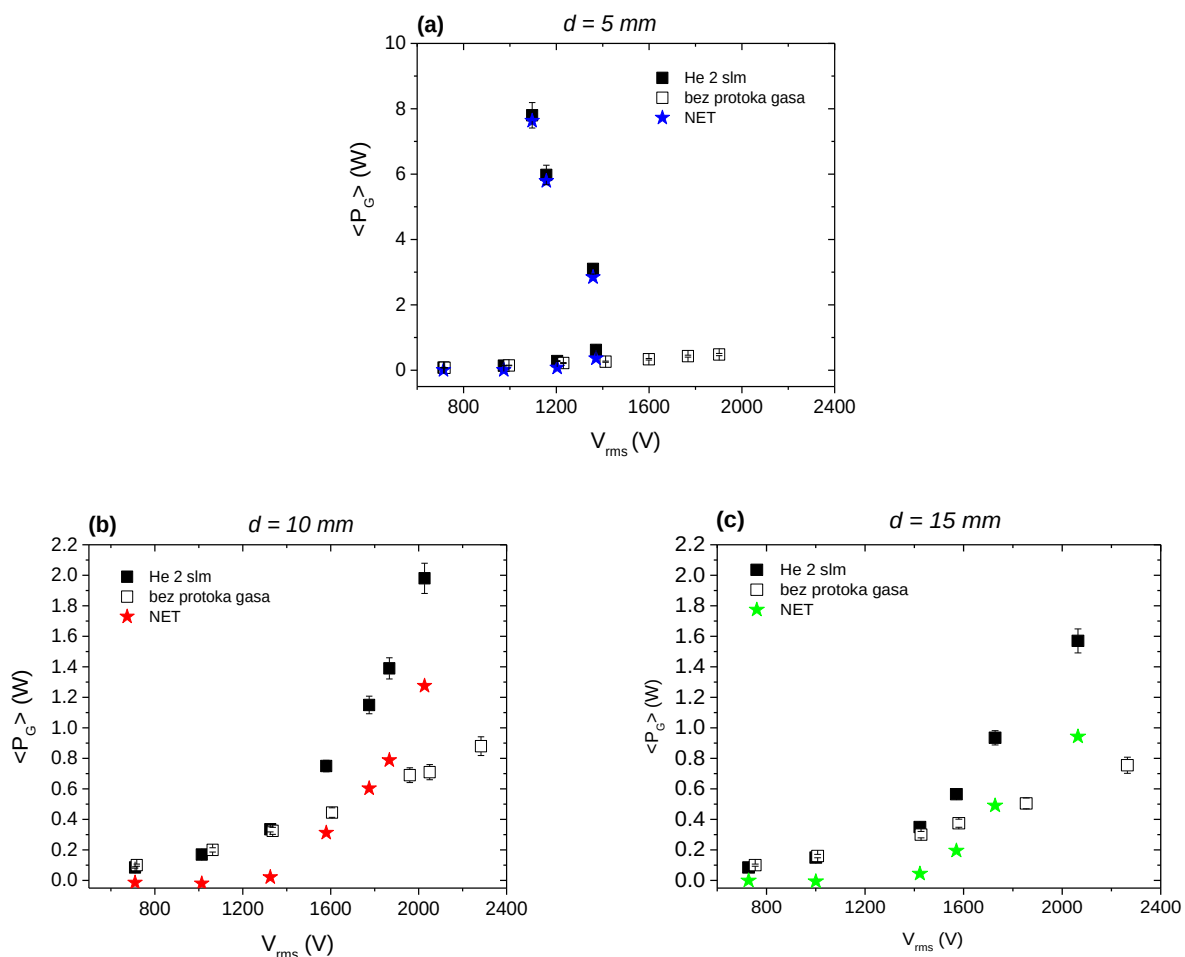
Uprkos svom tipu i kapacitetu, svaki kondenzator koji je korišćen u ovom radu davao je otvorene figure (nekontinualne). Q-V dijagram nije zatvoren jer se kriva posle jednog perioda primenjenog napona ne vraća na početnu tačku, odnosno vrednosti naelektrisanja i napona su različite u tačkama $t = 0$ i $t = T$. Ova karakteristika Lisažuove figure je već primećena za DBD plazma reaktore (Ashpis et al., 2017; Jiang et al., 2013). Uprkos tome, integracija oblasti figure je i dalje moguća, iako postoji praznina u krivulji. Ovaj problem se može umanjiti korišćenjem osciloskopa visoke rezolucije koji obezbeđuje snimanje talasnih oblika signala sa velikim brojem tačaka.

Glavni izazov sa upotrebom I-V metode je izbor otpornika. Otpornost treba da bude dovoljno velika da ograniči struju pražnjenja, što je posebno važno u slučaju kada je meta, koja se tretira plazmom, niske otpornosti (provodne mete). Nekontrolisano povećanje struje pražnjenja može dovesti do promene vrste pražnjenja, a zatim do zagrevanja i isparavanja tečne mete. Međutim, merni otpornik mora biti dovoljno male otpornosti, tako da se napon na krajevima otpornika može izmeriti pomoću naponske sonde.

Takođe, postoje određeni problemi sa upotrebom I - V metode kada se u eksperimentu koristi impulsni izvor napajanja. U tom slučaju, prilikom izračunavanja snage, potrebno je izvršiti integraciju tokom celog perioda trajanja impulsa. Integracija jednog dela perioda ulaznog signala koji je kraći od trajanja impulsa, daje netačan rezultat za deponovane snage jer može uključiti samo pozitivan ili samo negativan deo oscilacije talasnog oblika. To bi dovelo do potcenjivanja ili precenjivanja izmerene snage.

Variranje rastojanja d između napajane elektrode i tretirane tečnosti

Na slici 3.12 je prikazana zavisnost snage $\langle P_G \rangle$ od napona V_{rms} , za tri različita rastojanja d kada jemalation korišćen kao meta. Prikazani su rezultati u slučajevima sa i bez protoka radnog gasa u sistemu i njihova razlika $\langle P_{G\_NET} \rangle$.



Slika 3.12: Srednja snaga oslobođena u pražnjenju $\langle P_G \rangle$ u zavisnosti od RMS vrednosti primenjenog napona, za tri različita rastojanja između napajane elektrode i tečnosti ($d = 5, 10, 15$ mm). Srednje snage su snimljene u režimu sa protokom He od 2 slm i bez protoka radnog gasa i izračunata je razlika te dve vrednosti $\langle P_{G\_NET} \rangle$.

Za rastojanje $d = 5$ mm, do vrednosti napona od 1370 V (pre nego što je došlo spajanja provodnog kanala i površine tečnosti) vrednosti snage $\langle P_{G\_NET} \rangle$ su bile sbliske nuli. Nakon toga, povećanjem snage koja se predaje plazma mlazu dovodi do porasta snage $\langle P_{G\_NET} \rangle$ sa padom napona V_{rms} .

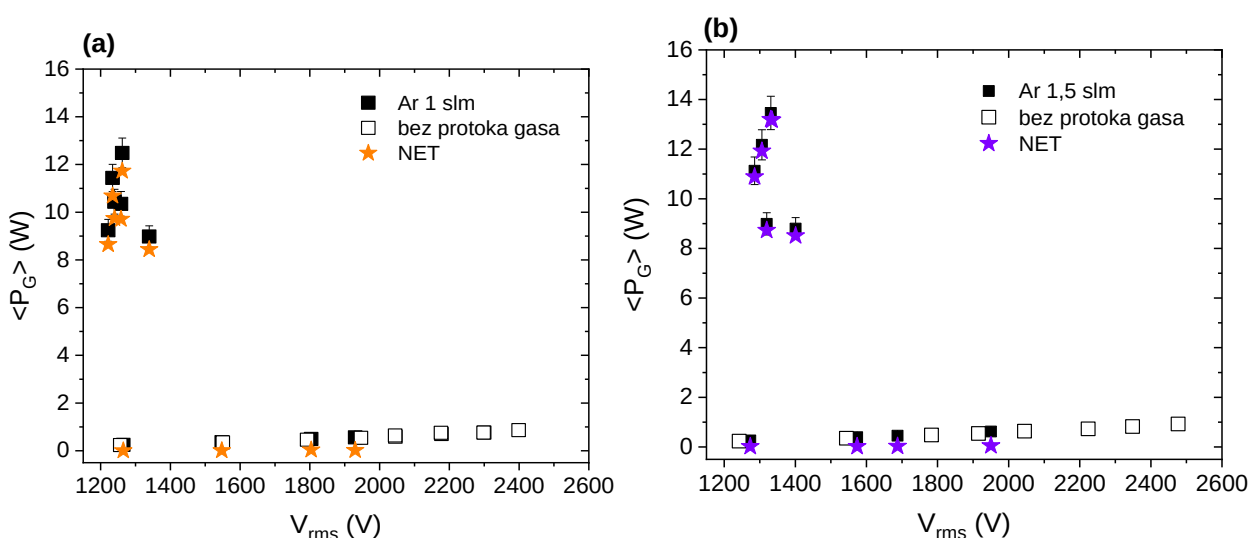
Srednje vrednosti snage oslobođene u pražnjenju $\langle P_{G\_sa\ protokom} \rangle$, za rastojanja od 10 mm i 15 mm, konstantno su rasle sa povećanjem napona V_{rms} i bile su približno jednake. Maksimalne vrednosti izmerenih snaga $\langle P_{G\_sa\ protokom} \rangle$ u su bile oko 2 W i 1,6 W, za rastojanja od 10 mm i 15 mm, respektivno

Srednje vrednosti snage predate izvoru plazme $\langle P_{sa\ protokom} \rangle$ takođe su rasle sa porastom napona (videti sliku 3.7). Kod rastojanja od 10 mm, pri manjim snagama predatim sa napajanja izvoru plazme P_F , vrednosti snaga $\langle P_{sa\ protokom} \rangle$ i $\langle P_{G\_sa\ protokom} \rangle$ su slične. Daljim povećanjem primenjenog visokonaponskog signala iz izvora, snaga predata izvoru plazme $\langle P_{sa\ protokom} \rangle$ bila je veća od snage predate u uzemljenoj grani kola $\langle P_{G\_sa\ protokom} \rangle$ i ta razlika za maksimalnu snagu iznosi 5 W. Ova razlika je bila mnogo uočljivija kod rastojanja od $d = 15$ mm (videti slike 3.7c i 3.12c), gde je iznosila 8 W za maksimalnim vrednostima primenjene snage sa napajanja P_F . Primećena razlika između izmerenih snaga verovatno je posledica povećanih gubitaka u prenosu signala od izvora ka plazmi koji se javljaju pri većim vrednostima primenjenih snaga izvora.

Pri većim rastojanjima, vrednosti snaga $\langle P_{G\_NET} \rangle$ bile su bliske nuli do momenta spajanja strimerskog kanala sa površinom mete pri vrednostima napona od 1300 V i 1400 V, za 10 mm i 15 mm, respektivno. Nakon toga snaga $\langle P_{G\_NET} \rangle$ je rasla sa porastom napona V_{rms} i izmerene maksimalne vrednosti su bile 1,3 W i 1 W za 10 mm i 15 mm, respektivno.

Variranje protoka radnog gasa

Na slici 3.13 prikazane su srednje snage $\langle P_G \rangle$ oslobođene u argonskom pražnjenju za dva različita protoka radnog gasa, za rastojanje $d = 10$ mm i malation korišćen kao meta. Za oba korišćena protoka (1 slm i 1,5 slm) dobijene su slične vrednosti snaga $\langle P_G \rangle$ koje su, nakon proboja u gasu, bile u opsegu od 8 W do 13,5 W. U poređenju sa rezultatima dobijenim za pražnjenje u helijumu za isto rastojanje d (slika 3.12b), primećeno je da su za red veličine veće snage oslobođene u argonskom pražnjenju. Takođe, u ovom slučaju su dobijene i daleko manje razlike između snaga $\langle P \rangle$ i $\langle P_G \rangle$, što znači da kada se kao radni gas koristi Ar bude efikasniji prenos električne energije u pražnjenje. Ovi rezultati su u saglasnosti sa promenama koje su uočene kod strujnih signala i promenama fazne razlike između struje i napona nakon uspostavljanja pražnjenja u argonu, koje nisu primećene kod helijuma.



Slika 3.13: Srednja snaga oslobođena u pražnjenju $\langle P_G \rangle$ u zavisnosti od RMS vrednosti primenjenog napona, za različite protoke Ar: 1 slm i 1,5 slm. Srednje snage su snimljene u režimu sa i bez protoka radnog gasa i izračunata je razlika te dve vrednosti $\langle P_{G\_NET} \rangle$.

3.1.3 DISKUSIJA I ZAKLJUČAK

U ovom delu predstavljeni su rezultati električne dijagnostike plazma mlaza koji je napajan kontinualnim sinusnim visokonaponskim signalom i koji radi na atmosfersom pritisku u kontaktu sa tečnom metom. Opisane su električne karakteristike pražnjenja u helijumu i u argonu, pri različitim uslovima u sistemu kao što su protok radnog gasa i međuelektroдно rastojanje. Ovaj deo istraživanja je za glavni cilj imao sistematično merenje električnih parametara kako bi se odredila srednja snaga oslobođena u pražnjenju $\langle P_G \rangle$. Srednja snaga $\langle P_G \rangle$ određena je korišćenjem dve standardne metode I - V i Q - V i urađeno je poređenje tako dobijenih rezultata.

U tu svrhu, praćeni su talasni oblici napona i struje pomoću kojih su nacrtane I - V karakteristike plazma mlaza. Fazna razlika između strujnog i naponskog signala kada u sistemu nema protoka radnog gasa, pokazala je kapacitivnu prirodu sistema. Linearna I - V karakteristika je u ovom slučaju iskorišćena je za određivanje impedanse celog sistema iz linearnog fita zavisnosti napona od struje. Fazna razlika ostaje gotovo nepromenjena kada postoji protok helijuma, iako prisustvo plazme u sistemu unosi dodatnu rezistivnu komponentu. Sa druge strane, u slučaju argonskog pražnjenja pojava plazme u sistemu unosi rezistivnu komponentu koja značajno menja impedansu celog sistema, što utiče na promenu fazne razlike između struje i napona i nelinearnost I - V karakteristike.

Proučavanje uticaja parametara sistema na gasno pražnjenje urađeno je kako bi se pronašli najoptimalniji uslovi rada plazma mlaza koji su kasnije korišćeni u konkretnim primenama. Kod pražnjenja u helijumu ispitivana je promena međuelektrodnog rastojanja d .

Na najkraćem rastojanju $d = 5$ mm do proboja dolazi na nakon postizanja vrednosti napona od $V_{rms}=1200$ V, a za napone veće od 1370 V I - V karakteristika menja oblik iz linearne u nelinearnu što je praćeno naglim porastom intenziteta zračenje i prečnika strimera. Na većim rastojanjima, $d = 10$ mm i $d=15$ mm, do pada napona usled proboja dolazi već na primenjenom naponu od $V_{rms}=1300$ V. I - V karakteristike zadržavaju linearnu zavisnost i nakon proboja, a struja i napon rastu istovremeno sa postepenim povećanjem snage na napajanju.

Na ovim međuelektrodnim rastojanjima $d = 10$ mm i $d = 15$ mm, pražnjenje se prvo javlja na vrhu napajane elektrode, a zatim se produžava ka tečnoj meti sa porastom snage koja se sa napajача šalje u sistem, nakon čega dolazi do kontakta strimera sa metom.

U slučaju pražnjenja u argonu, stabilno pražnjenje je bilo moguće uspostaviti u vrlo uskom opsegu rastojanja d ($d=10\pm 2$ mm), zbog čega su sva merenja rađena na rastojanju $d=10$ mm. Takođe, pri protocima manjim od 1 slm i većim od 1,5 slm nije bilo moguće uspostaviti stabilan režim pražnjenja. Dobijene I - V karakteristike za dva primenjena protoka (1 slm i 1,5 slm) su gotovo identične, ali se značajno razlikuju od onih dobijenih sa helijumom.

Merenje srednjih snaga koje se dovode na plazma mlaz $\langle P \rangle$ i snaga oslobođenih u pražnjenju $\langle P_G \rangle$ (izmerenih u uzemljenoj grani kola) urađeno je pomoću dve metode. U slučaju pražnjenja u helijumu ($q=2$ slm, $d=10$ mm) upoređeni su rezultati dobijeni za snagu $\langle P_G \rangle$ pomoću I - V metode kada je u uzemljenoj grani kola bio otpornik $R=1$ k Ω i Q - V metode kada je otpornik zamenjen kondenzatorom, za dve vrednosti kapacitivnosti 0,8 nF i 10,3 nF. U oba slučaja kada je kondenzator ubačen u uzemljenu granu električnog kola, dobija se eliptični oblik Lisažuoove figure. Međutim, primećeno je da se izmerena snaga sa dva različita kondenzatora razlikuje se za 23%, što sugerise nešto drugačije podešavanje radne tačke plazme usled različitih uslova u sistemu. Pokazano je da svojstva kondenzatora ne treba zanemariti prilikom izračunavanja snage koja se isporučuje plazmi, jer kondenzator ne treba smatrati idealnim elementom kola. Ipak, korišćenje različitih mernih tehnika je pokazalo da je zavisnost snage $\langle P_G \rangle$ od V_{rms} uvek ista bez obzira na izabranu metodu.

Glavna razlika koja je primećena između pražnjenja u helijumu i u argonu jeste u energetskej efikasnosti sistema. Za pražnjenje u helijumu, kada je strimer u kontaktu sa tečnom metom ($q=2$ slm, $d=10$ mm) je dobijeno da se manje od 30% snage $\langle P \rangle$, koja se preda plazma mlazu, potroši u pražnjenju i detektuje u uzemljenoj grani ($\langle P_G \rangle$). Tada su izmerene snage $\langle P_G \rangle$ bile u intervalu od 0 do 1,7 W za V_{rms} opseg između 0,7 kV i 2 kV. Ove snage su bile veće samo u slučaju manjeg međuelektrodnog rastojanja $d=5$ mm, koji nije bio od interesa za dalje primene sistema pošto je usled intenzivnog pražnjenja dolazilo do naglog isparavanja uzorka.

Nasuprot tome, u slučaju pražnjenja u argonu ($q=2$ slm, $d=10$ mm) izmerene su snage $\langle P_G \rangle$ u opsegu od 0 do 12 W za V_{rms} opseg između 0,7 kV i 2 kV, a u pražnjenju je predata 76% ukupne snage predate plazma mlazu $\langle P \rangle$.

Što se tiče tipa tečne mete, ideja merenja sa dva različita tečna uzorka - destilovanom vodom i vodom kontaminiranom malationom, je bila da se ispita kako tip tečnog uzorka, tj. dodatak organiskih molekula utiče na električne osobine pražnjenja. Dodavanje organiskih molekula nije uticalo na provodljivost pa ova dva uzorka imaju sličnu električnu provodljivost u zapremini ($2 \mu\text{S/cm}$ destilovana voda i $12,5 \mu\text{S/cm}$ malation). Sa druge strane, pojedini organski molekuli imaju osobine površinski aktivne materije (surfaktanti) pa su merenja I - V karakteristika urađena kako bi se ispitala i mogućnost lokalne promene provodljivosti i uticaja na pražnjenje. U oba slučaja različitih uzoraka, dobijeni su rezultati električne karakterizacije koji se razlikuju u okviru eksperimentalne greške. Prema tome, može se zaključiti da data koncentracija organskih jedinjenja u destilovanoj vodi (od 500 ppm) nije dovoljna da bi došlo do promena u režimu pražnjenja, u odnosu na pražnjenje u kontaktu sa čistom destilovanom vodom.

3.2 DETEKCIJA EMISIJE ZRAČENJA IZ PLAZME

3.2.1 OPTIČKA EMISIONA SPEKTROSKOPIJA

Metoda optičke emisijske spektroskopije primenjena je kako bi se dobio uvid u pobuđene reaktivne vrste koje nastaju u gasnoj fazi pražnjenja. Snimljeni su vremenski i prostorno usrednjeni spektri emisijonog zračenja pražnjenja iz oblasti između napajane elektrode i površine tečne mete. U svim slučajevima je tečna meta bila destilovana voda, a međuelektrodno rastojanje je bilo $d=10$ mm. U drugom delu ovog poglavlja predstavljeni su rezultati određivanja temperatura gasa, pomoću snimljene emisije zračenja rotaciono pobuđenog $\text{OH}(A^2\Sigma^+ - X^2\Pi)$ molekula.

DETEKCIJA REAKTIVNIH VRSTA IZ GASNE FAZE PRAŽNJENJA

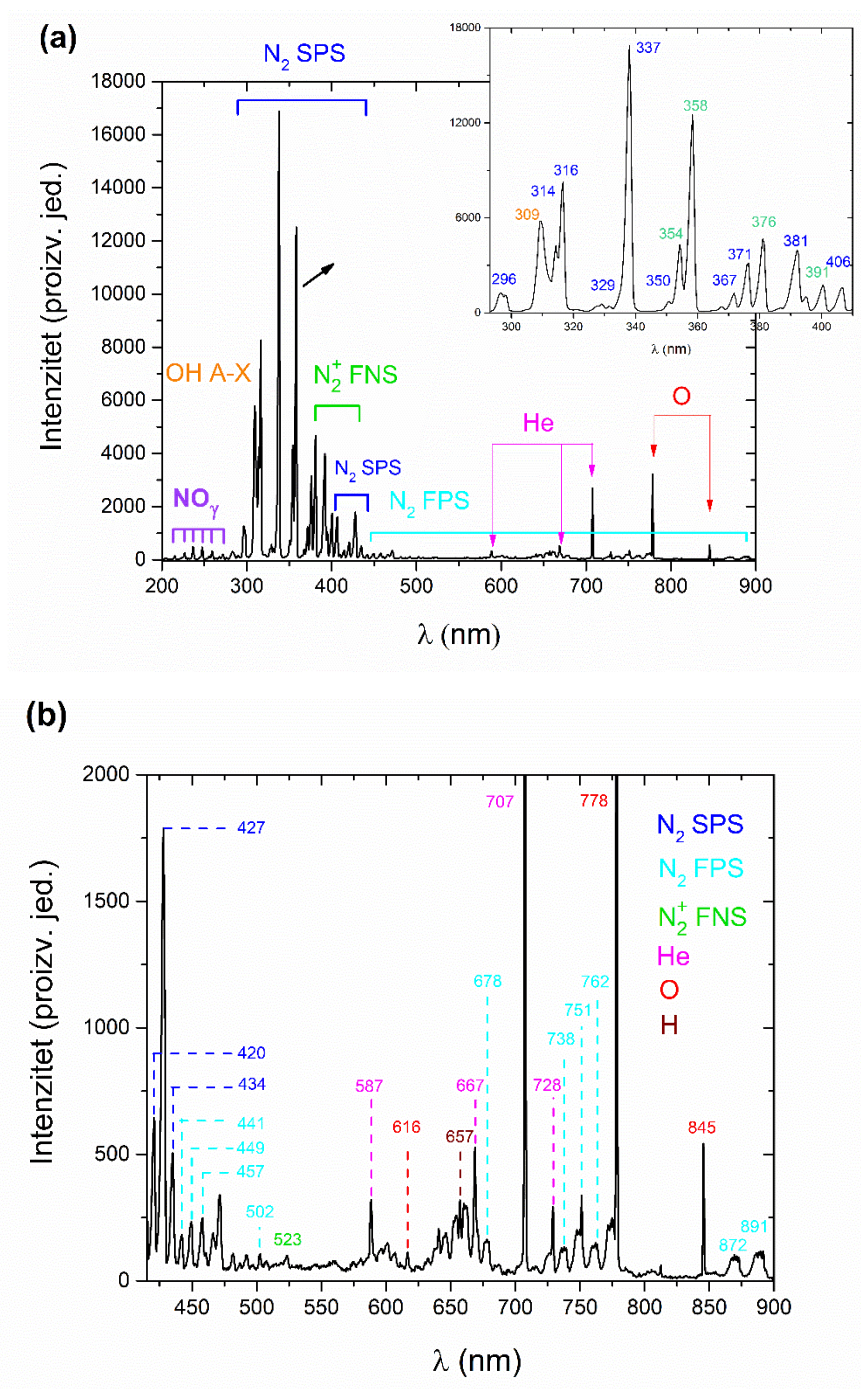
Tokom merenja pražnjenje je bilo u kontaktu sa destilovanom vodom (zapremina uzorka 4 ml) u kvarenoj posudi. Ispitani su uticaji sledećih parametara na stvaranje pobuđenih vrsta:

- snage koja sa predaje plazma mlazu $\langle P_{G\_NET} \rangle$
- vrste radnog gasa ($\text{He } q = 2$ slm i argon $q = 1$ slm)
- dodavanja sintetičkog vazduha radnom gasu (Ar, Ar +1% vazduha, Ar+3% vazduha)

Optički sistem je postavljen tako da je omogućeno da se kroz sočivo i detektor spektrometra sakuplja zračenje koje potiče iz cele oblasti od vrha napajanje elektrode do površine tečnosti ($d=10$ mm). Snimljeni spektri su korigovani u odnosu na funkciju efikasnosti detekcionog sistema i na parametre snimanja – usrednjavanje signala i trajanje ekspozicije. Prikazani rezultati predstavljaju srednju vrednost intenziteta zračenja za tri nezavisna merenja. Identifikacija linija snimljenog spektra urađena je poređenjem sa NIST-ovom bazom podataka za atomske vrste, dok je za molekulske vrste korišćena publikacija (Pearse & Gaydon, 1963).

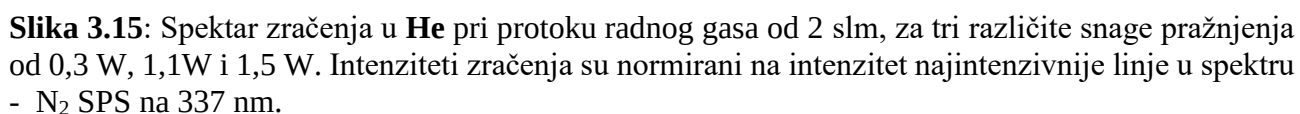
Pražnjenje u helijumu

Na slici **3.14** prikazan je spektar zračenja iz pražnjenja u **helijumu (protok gasa 2 slm)**, u opsegu talasnih dužina od 200 nm do 900 nm, dobijen usrednjavanjem deset signala detektovanih spektrometrom pri ekspoziciji od 100 ms. Izmerena snaga oslobođena u pražnjenju $\langle P_G \rangle$ bila je 1,1 W. Identifikacijom dobijenih spektralnih linija i traka utvrđeno je da su u plazmi dominantno pobuđeni molekuli i molekulski joni azota, ali i reaktivne vrste kiseonika kao što su OH molekul i atomski kiseonik.



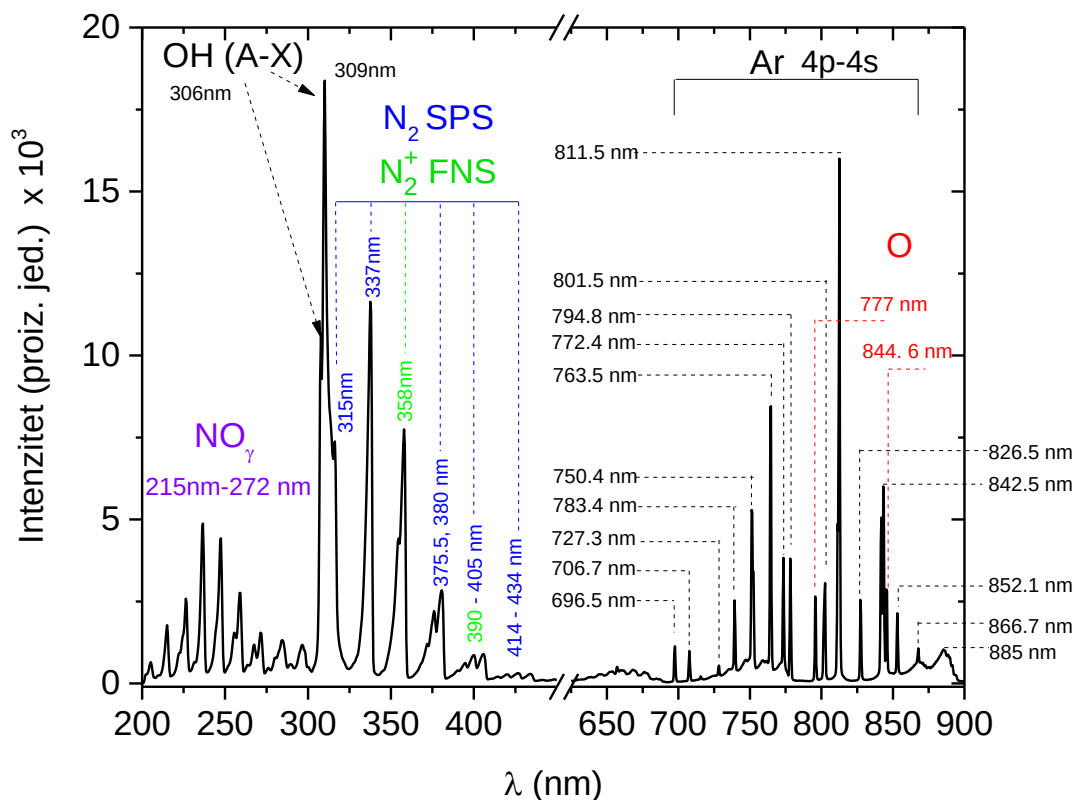
Slika 3.14:Spektar zračenja u He pri protoku radnog gasa od 2 slm: (a) u oblasti talasnih dužina od 200 nm do 900 nm, sa uveličanim prikazom spektra u oblasti od 250-450 nm i (b) u oblasti od 500 nm do 900 nm. Snaga predata u pražnjenju bila je $\langle P_{G\_NET} \rangle = 1,1\text{W}$.

Kako bi se ispitalo stvaranje ekscitovanih vrsta pri različitim uslovima u sistemu, spektar zračenja je sniman za **različite srednje snage pražnjenja** $\langle P_{G\_NET} \rangle$, a rezultati su prikazani na slici 3.15. Merenja su urađena za tri različite snage predate u pražnjenju $\langle P_{G\_NET} \rangle$, od 0,3 W, 1,1 W i 1,5 W. Sva merenja su urađena u slučajevima kada je pražnjenje u kontaktu sa vodom. Rezultati su pokazali da povećavanje snage utiče na stvaranje reaktivnih vrsta u UV oblasti spektra. Nakon što snaga pražnjenja dostigne vrednost od 1,1 W detektovano je zračenje koje potiče od molekula NO_γ i značajan porast intenziteta zračenja molekula OH i N_2 SPS. Takođe, pojavilo se i zračenje u oblasti talasnih dužina od 580 nm do 780 nm gde su detektovane linije helijuma na 587 nm i 667 nm i linija vodonika na 657 nm. U preostalom delu spektra, povećavanjem snage $\langle P_{G\_NET} \rangle$ nisu primećena stvaranja novih pobuđenih vrsta, već samo povećavanja intenziteta snimljenih linija/traka.



Pražnjenje u argonu

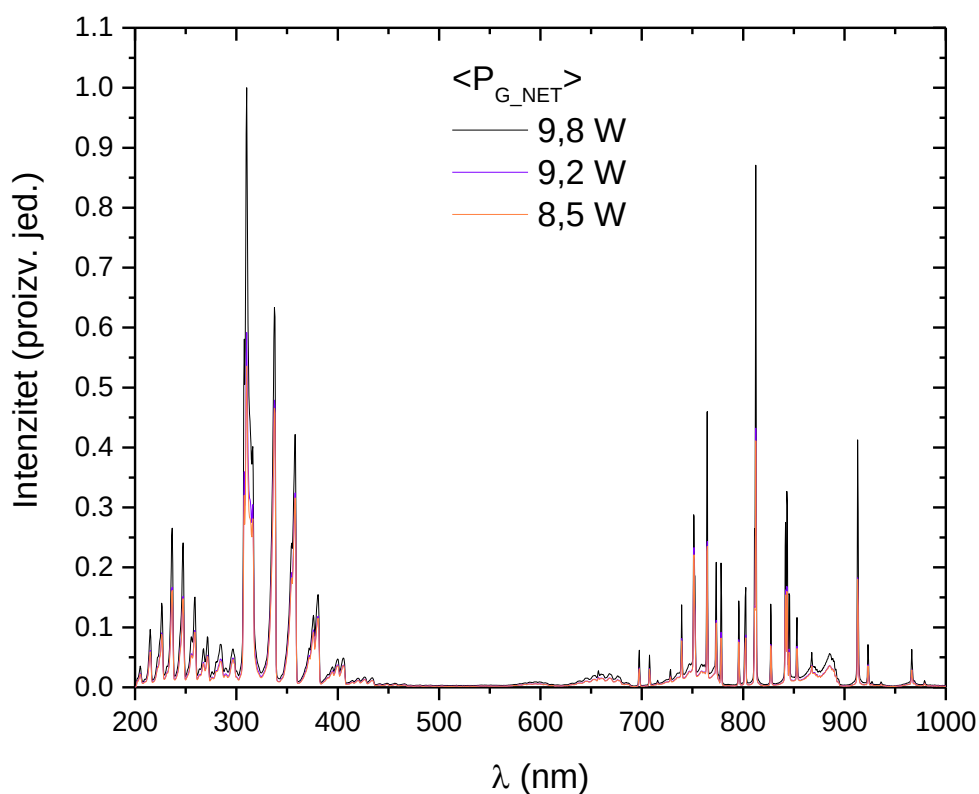
Na slici 3.16 prikazan je spektar zračenja u **argonskoj** plazmi pri protoku gasa od **1 slm**, kada je snaga pražnjenja bila 9,8 W. U ultraljubičastom delu spektra, detektovane su gotovo iste rekativne vrste kao i u slučaju pražnjenja u helijumu, ali su intenziteti emisijih traka NO_γ i OH daleko veći u slučaju argona. Maksimum emisije NO_γ trake primećena je na 236 nm, dok najintenzivnija emisija u spektru potiče od molekula OH (306 nm i 309 nm).



Slika 3.16: Spektar zračenja u **Ar** pri protoku gasa od 1 slm u opsegu talasnih dužina od 200 – 900 nm. Snaga predana u pražnjenju bila je $\langle P_{G\_NET} \rangle = 9,8 \text{ W}$

U oblasti talasnih dužina od 600 nm do 900 nm, primećene su manje intenzivne emisije **atomskog kiseonika** na 777 nm i 844 nm (prelaz 3s-3p) i vodonika H_α na 656 nm (Kramida et al., 2016). Preostale linije u spektru pripadaju prelazima **argona** sa nivoa 4p na nivo 4s (697 nm, 707 nm, 715 nm, 728 nm, 739 nm, 751 nm, 764 nm, 773 nm, 795 nm, 802 nm, 812 nm, 827 nm, 841 nm, 843 nm, 853 nm, 868 nm, 885 nm). Slični spektri su dobijeni i u drugim istraživanjima izvora neravnotežne plazme na atmosferskom pritisku (Bruggeman & Brandenburg, 2013; Lukes et al., 2012; Machala et al., 2019; Oh et al., 2016; Xiong et al., 2010).

Kako bi se ispitao efekat snage koja se predaje u sistem plazma mlaza na emisiju reaktivnih vrsta, urađeno je merenje spektara pri različitim snagama predatim u pražnjenju $\langle P_{G\_NET} \rangle$. Na slici 3.17 prikazani su spektri zračenja dobijeni za snage $\langle P_{G\_NET} \rangle$ od 8,5 W, 9,2 W i 9,8 W. Intenzitet zračenja normiran je na intenzitet najintenzivnije linije u spektru – linije molekula OH na 309 nm.

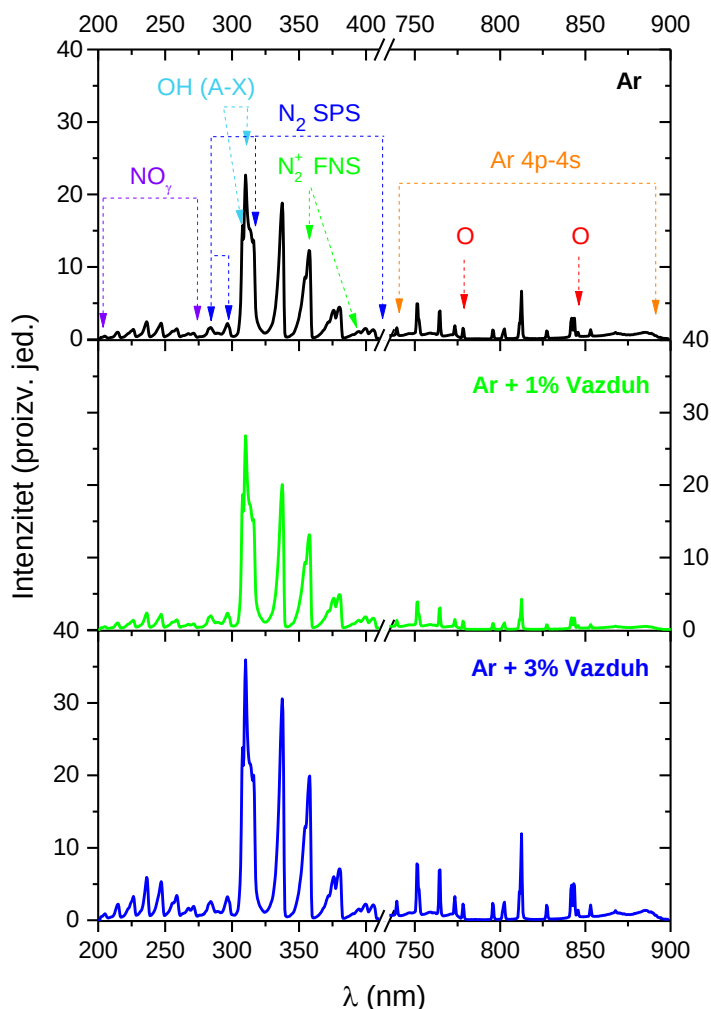


Slika 3.17: Spektar zračenja u **Ar** pri protoku gasa od 1 slm u opsegu talasnih dužina od 200 – 1000 nm, za tri **različite snage** pražnjenja: 8,5 W, 9,2 W i 9,8 W. Intenziteti zračenja su normirani na intenzitet najintenzivnije linije u spektru - OH na 309 nm.

Povećanjem predate snage došlo je do povećanja intenziteta zračenja svih linija u spektru, ali ne i do pojave novih linija. Povećanjem predate snage sa 8,5 W na 9,2 W, u UV delu spektra dovelo je do manjeg povećanja emisije OH trake (306 nm i 309 nm) i linije Ar na 812 nm. Intenziteti ostalih linija su bili nepromenjeni. Dalje povećanje snage za 0,6 W (crna linija na slici 3.17), dovelo je do porasta intenziteta svih linija u spektru. Najveća promena zapažena je kod emisije OH i kod linija argona. Stvaranje OH radikala u plazmi zavisi od prisustva molekula vode i eksitovanih atoma Ar, ali i koncentracije i temperature elektrona i temperature gasa. Sa povećanjem snage koja se oslobodi u pražnjenju, dolazi do porasta struje pražnjenja odnosno do povećanja koncentracije elektrona, kao i do više ekscitacija argona. Na ovaj način povećava se broj čestica koji može da učestvuje u disocijaciji molekula vode i stvaranju OH molekula. Povećanje koncentracije OH molekula sa porastom snage pražnjenja (Zaptołnik et al., 2021), ali i nelinearno povećanje koncentracija drugih vrsta kao što su N_2 i O koji dovode do stvaranja NO, primećeno je kod sličnih izvora plazme koji rade u prisustvu vodene pare (Wang et al., 2010) ili tečne mete (Srikanth et al., 2024).

Dodavanje sintetičkog vazduha u gasnu smešu sa Ar

Pored merenja intenziteta emitovanog zračenja u argonskom pražnjenju, urađena su i merenja kada je kao radni gas korišćena smeša argona i sintetičkog vazduha. Ukupan protok radnog gasa je ostao nepromenjen (1 slm), a korišćene su smeše sa 1% i 3% sintetičkog vazduha. Snimljeni spektri prikazani su na slici 3.18, pri čemu je snaga oslobođena upražnjenju bila 10,75 W. Za dobijanje smeša gasova su podešeni sledeći protoci: Ar + 1% vazduh – 0,99 slm Ar i 10 sccm vazduha; Ar + 3% vazduh – 0,97 slm i 30 sccm vazduh.



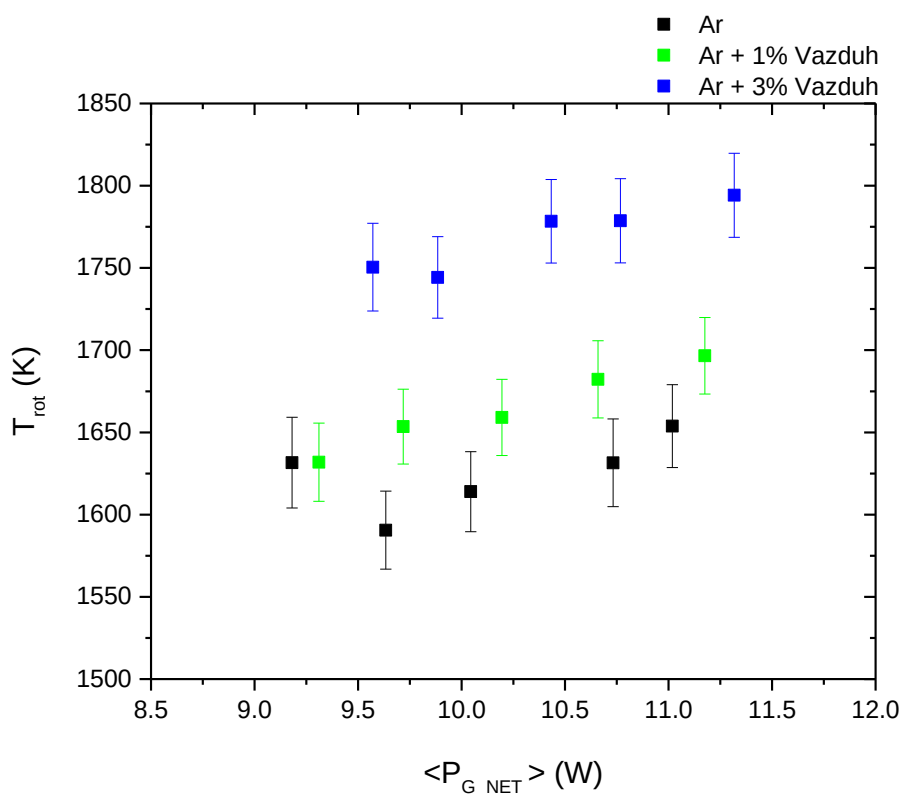
Slika 3.18: Spektar detektovanog zračenja u opsegu talasnih dužina od 200 – 900 nm u **argonu** i smešama **argona i vazduha**, pri čemu su udeli vazduha bili 1% (10 sccm) i 3%.(30 sccm). Ukupni protok radnog gasa bio je 1 slm.

Kada je korišćena smeša sa 1% sintetičkog vazduha, došlo je do povećanja intenziteta emisionih traka molekula OH, N₂ SPS, i N₂⁺ FNS i blagog smanjenja intenziteta linija atomskog kiseonika (777 nm i 845 nm) i pojedinih linija argona, dok je emisija NO ostala nepromenjena. U slučaju kada je procenat sintetičkog vazduha u gasnoj smeši bio 3%, došlo je do povećanja intenziteta većine emitovanih linija u spektru u odnosu na slučaj kada nije korišćen sintetički vazduh. Pored toga došlo je i do emitovanja linija argona na 913nm i 923 nm, koje nisu primećene u druga dva slučaja.

Analizom snimljenih emitovanih spektara utvrđeno je da dodavanje sintetičkog vazduha u gasnu smešu dovodi do linearnog porasta intenziteta emisije OH linija na 306 nm i 309 nm. Preciznije, za smeše Ar + 1% vazduh i Ar + 3% vazduh, dobijeni intenziteti su uvećani za aproksimativno 20% i 50-60%, respektivno.

MERENJE ROTACIONE TEMPERATURE OH MOLEKULA

Na slici 3.19 prikazana je zavisnost rotacione temperature OH molekula od snage $\langle P_G \rangle$ (NET), za tri različite smeše gasova: Ar, Ar + 1% vazduha i Ar+ 3% vazduha. U slučaju pražnjenja u čistom Ar, izmerenja je srednja temperatura od 1630 ± 60 K u opsegu snaga u okviru kojih su urađena merenja. Kako je opseg snaga bio prilično uzak, oko 2 W, ovakvo ponašanje sistema pokazuje da je pražnjnje uspostavljeno u stabilnom režimu gde raspodela energije i toplotni gubici ostaju gotovo nepromenjeni.



Slika 3.19: Vrednosti temperature gasa određene analizom rotacione strukture emisije $\text{OH}(A^2\Sigma^+ - X^2\Pi)$, usrednjene duž celog međuelektrodnog rastojana, u zavisnosti od snage predate u pražnjenju $\langle P_{G\_NET} \rangle$, za tri različite gasne smeše: Ar, Ar+1%Air, Ar+3%Air. Ukupan protok gasa bio je 1 slm. Predstavljene greške merenja potiču od odstupanja između eksperimentalno izmerenih i simuliranih spektara. Svako merenje je ponovljeno tri puta, a prikazane vrednosti predstavljaju srednju vrednost dobijenu iz tih ponavljanja.

Relativno visoka izmerena temperatura odražava intenzivno zagrevanje gasa, što sugerise da se značajan deo energije pretvara u translacionu energiju gasa, uporedo sa procesima pobuđivanja i hemijskim reakcijama. Ovakvo efikasno zagrevanje gasa, karakteristično je za atmosferske plazma malazove sa strimerskim tipom pražnjenja u argonu u sredini sa povišenim sadržajem vodene pare. U ovakvim uslovima sudarni procesi dovode do deekscitacije pobuđenih stanja argona i gašenja molekula $\text{OH}(\text{A}^2\Sigma^+)$ vodenom parom, što u velikoj meri doprinosi pretvaranju energije u toplotu (Iseni et al., 2014). Pošto prikupljena emisija zračenja predstavlja ukupan signal iz čitave oblasti između napajane elektrode i površine vode, dobijena temperatura uglavnom odgovara oblasti sa najvećom koncentracijom molekula $\text{OH}(\text{A}^2\Sigma^+)$, odnosno jezgu plazme (eng. plasma core).

Sa povećavanjem udela vazduha u gasnoj smeši, uočeno je postepeno povećanje temperature gasa (simboli kvadrata plave i zelene boje na slici **3.19**). Srednje temperature u datom opsegu snaga iznosile su 1670 ± 60 K i 1770 ± 60 K, za slučajeve sa 1% i 3 % vazduha u gasnoj smeši, respektivno. Opaženi trend je u saglasnosti sa ranijim istraživanjima pražnjenja u plemenitim gasovima sa primesama molekulskih gasova, gde dodatak N_2 i O_2 dovodi do pojačanog prenosa toplotne energije (Bruggeman et al., 2009; Wang & Srivastava, 2010). Prisustvo ovih molekulskih vrsta omogućava pobuđivanje vibracionih stanja, koja se brzo deekscituju putem vibraciono-translacionog (V–T) prenosa energije, čime dolazi do zagrevanja gasa (Ono, 2016). Kako se udeo vazduha povećava, sve veći deo energije predate sistemu se troši na zagrevanje gasa i radijacione procese, nego na hemijske reakcije, čime se smanjuje energetska efikasnost i udaljava od netermalnog režima pražnjenja. Budući da je cilj ovog istraživanja podsticanje stvaranja reaktivnih vrsta azota i kiseonika u netermalnim uslovima, prekomerno zagrevanje gasa je nepoželjno za praktične primene kao što je proizvodnja PAV-a.

DISKUSIJA I ZAKLJUČAK

U ovom potpogavlju opisani su rezultati OES metode koja je u ovom slučaju korišćena u svrhu detektovanih reaktivnih ekscitovanih vrsta u strimerskom pražnjenju u helijumu i argonu u kontaktu sa vodom. U slučaju oba tipa pražnjenja, pored ekscitovanih vrsta korišćenog radnog gasa (He ili Ar), u spektrima su identifikovane iste azotne i kiseonične vrste, ali su primećene razlike u dominantnim vrstama. Pored uticaja vrste radnog gasa, ispitani su uticaji promena drugih parametara pražnjeja - snage predate u pražnjenju i dodavanje sintetičkog vazduha u gasnu smešu, na zračenje ekscitovanih vrsta iz plazme. Na kraju, u slučaju pražnjenja u argonu (čist argon i gasne smeše sa 1% i 3% vazduha), određena je temperatura gasa pomoću rotacione strukture molekula $\text{OH}(\text{A}^2\Sigma^+ - \text{X}^2\Pi)$.

Nastanak ekscitovanih vrsta helijuma i stvaranje atomskog kiseonika u reakciji disocijacije molekula O_2 elektronima iz plazme, opisani su u poglavlju **1.3.2**. Stvaranje N_2^+ može se objasniti visokim jonizacionim potencijalom helijuma, koji je značajno viši od energije jonizacije molekula N_2 .

Pri sudaru He^+ i molekula N_2 dolazi do reakcije prenosa naelektrisanja (eng. charge transfer) i do stvaranja visoko ekscitovanih elektronskih stanja $(\text{N}_2^+)^*$ (Beseda et al., 2022):



Usled kontakta strimerskog kanala sa površinom vode, dolazi do isparavanja vode i povećane koncentracije vodene pare u pražnjenju u gasnoj fazi. Ovakvi uslovi u sistemu omogućavaju da dođe do disocijativne rekombinacije molekula vode elektronima iz plazme, metastabilima He ili atomskim kiseonikom (videti izraze **1.11**, **1.12** i **1.17** u potpogavlju **1.3.2**) pri čemu nastaje molekul OH.

Pored toga, jonizacijom vodene pare elektronima iz plazme (reakcija **1.13**) omogućava se proces multiplikacije elektrona i povećavanja struje pražnjenja (Lim et al., 2020). Veća koncentracija elektrona u plazmi može dovesti do većeg stvaranja atomskog kiseonika i azota kroz reakcije elektronskog udara (**1.16** i **1.18**), a dalje kroz interakcije ovih vrsta sa ekscitovanim molekulima O_2 i N_2 do stvaranja azot-monoksida NO (Wandell et al., 2019).

Kod pražnjeja u Ar (1 slm), struja pražnjenja, a time i koncentracija elektrona u plazmi, je veća nego kod helijuma (videti slike **3.6** i **3.7**), što povećava stvaranje N, N_2 , O i O_2 i dalje stvaranje NO. U slučaju argonskog pražnjenja kontaktna površina plazme i površine vode je veća usled veće širine strimera, što posledično dovodi do značajnijeg isparavanja vode i stvaranja OH radikala.

Dodavanje sintetičkog vazduha u smešu radnog gasa i time unosenje dodatne količine N_2 i O_2 uticalo je na povećanje intenziteta reaktivnih vrsta koje su već uočene kod pražnjenja u čistom Ar. Povećanje intenziteta je raslo sa udelom sintetičkog vazduha u gasnoj smeši. Dodatkom 1% vazduha u gasnu smešu, uticalo je na porast intenziteta emisionih traka OH, N_2 SPS i N_2^+ FNS, dok je emisija NO ostala nepomenjena. Ove promene praćene su blagim padom intenziteta linija atomskog kiseonika koje odgovaraju prelazima $3s - 3p$ na 777 nm i 844 nm, kao i argonskih linija koje odgovaraju prelazima $4s - 4p$ u opsegu talasnih dužina od 738-811 nm. Ovakve promene mogu nastati usled gašenja metastabilnih stanja Ar ($^3P_2$, $^3P_0$) kroz sudare Penning-ovog tipa sa molekulima $N_2(X^1\Sigma_g^+)$ i $O_2(X^3\Sigma_g^-)$ u osnovnom stanju. U ovim reakcijama nastaje pobuđeno ste pobuđeno stanje molekula azota $N_2(C^3\Pi_u)$.

Povećavanje udela vazduha u gasnoj smeši na 3%, dovelo je do porasta intenziteta većine emisionih linija u odnosu na pražnjenje u čistom argonu. Primećen je linearni porast emisije molekula OH sa udelom sintetičkog vazduha.

Na kraju, ispitan je uticaj promene snage predate u pražnjenju na emisione spektre u helijumu i argonu. Povećanje snage dovelo je do porasta emisije zračenja u UV oblasti i na formiranje NO molekula, u slučaju pražnjenja u helijumu. Emisija zračenja je rasla sa porastom predate snage. Kod pražnjenja u argonu je uočen nelinearni porast emisije zračenja sa porastom snage, u uskom opsegu primenjenih snaga od 1,3W.

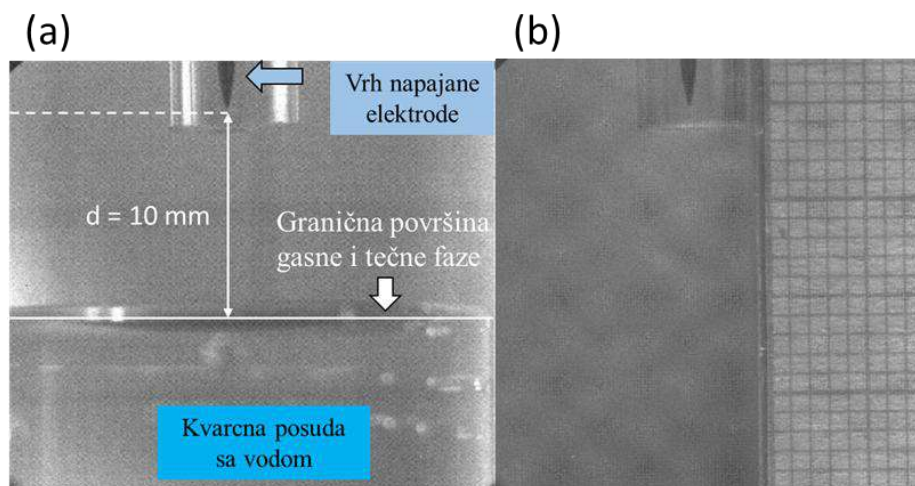
Sa stanovišta primene tehnologije plazme, pražnjenje u argonu se pokazalo efikasnijim u stvaranju reaktivnih vrsta u gasnoj fazi u odnosu na pražnjenje u helijumu. Prednost korišćenja argona, pogotovu uz dodavanje sintetičkog vazduha, jeste smanjenje troškova ove tehnologije. Iz tih razloga, dalje istraživanje i primene tehnologije plazme u okviru ove disertacije bili su usmereni na pražnjenje u argonu.

3.2.2 SNIMANJE PROSTORNE EVOLUCIJE STRIMERSKOG PRAŽNJENJA ICCD KAMEROM

Druga optička metoda koja je korišćena za dijagnostiku gasne faze strimerskog pražnjenja u kontaktu sa vodom, uključivala je vremenski integrisano slikanje prostorne raspodele emisije zračenja ICCD kamerom. Pored snimanja ukupne emisije zračenja iz plazme, urađeno je i merenje razloženo po talasnim dužinama kako bi se snimilo zračenje koje potiče od pojedinačnih reaktivnih vrsta prethodno detektovanih OES metodom (OH , N_2 , H_α , O , Ar). Cilj je bio da se odredi u kom delu pražnjenja nastaju određene reaktivne vrste.

Merenje prostorne strukture strimerskog pražnjenja je urađeno pri protoku argona od 1 slm, u kontaktu sa destilovanom vodom koja se nalazila u kvarcnoj posudi. Zapremina uzorka bila je 4 ml, a rastojanje između elektrode i vode je bilo $d = 10$ mm. Snimanje strukture pražnjenja urađeno je u potpunom mraku, pri temperaturi od $T = 24$ °C i vlažnosti vazduha od $H = 58,2$ %. Merenje je urađeno za različite snage $\langle P_G \rangle$, ali kako su u slučaju pražnjenja u argonu radne tačke veoma bliske (videti sliku 3.13a) i opseg električnih parametara u kojima je moguće merenje je veoma mali, u nastavku ovog poglavlja biće prikazani rezultati dobijeni za dve snage $\langle P_G \rangle$ kod kojih je primećena različita struktura pražnjenja.

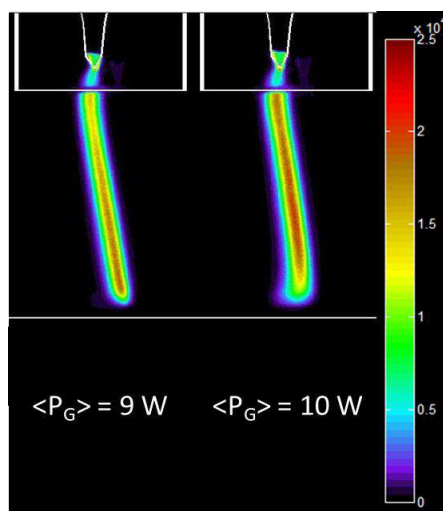
Pre snimanja pražnjenja, optički sistem je bio podešen tako da se kamera nalazila na 24 cm od ose plazma mlaza, a fokus kamere je bio na vrhu napajane elektrode. Sniman je prostor između elektrode i površine vode i deo zapremine vode koja se nalazi u kvarcnoj posudi (videti sliku 3.20). Kod merenja razloženog po talasnim dužinama, filteri su postavljeni na 2 cm od objektiva kamere. Kalibracija prostornih dimenzija u sistemu (1 mm : broj piksela) je izvršena slikanjem sistema sa milimetarskom skalom do staklene cevčice plazma mlaza, u režimu kada nema plazme. Jedna slika se sastojala od 1024x1024 piksela.



Slika 3.20: (a) Fokus ICCD kamere na vrh elektrode plazma mlaza i tretirani uzorak u sistemu bez plazme (donji kraj slike je 2mm iznad dna kvarcne čašice) i (b) kalibracija dimenzija u sistemu. Vreme ekspozicije 100 ms, dužina perioda snimanja 75 ms, pojačanje 0.

Sve slike koje su prikazane u nastavku su normirane u odnosu na parametre snimanja (dužinu perioda snimanja - eng. Gate pulse width i pojačanje) i oduzeto je pozadinsko zračenje, tako da su slike **3.21** – **3.26** međusobno poredive. Dužina ekspozicije je u svim slučajevima bila 20 ms. U slučaju merenja razloženog po talasnim dužinama urađeno je dodatno normiranje prema krivi efikasnosti fotokatode kamere i transparentnosti optičkih filtera. Na taj način omogućeno je direktno poređenje dobijenih intenziteta zračenja pri različitim podešavanjima parametara kamere. Prilikom definisanja skala za intenzitet zračenja, uzeta je u obzir efikasnost filtera. Takođe, moguće je međusobno poređenje intenziteta zračenja snimljenih prilikom korišćenja različitih optičkih filtera (slike **3.22** – **3.26**). Stoga je moguće poređenje brojčanih vrednosti na različitim skalama, ali ne i boja koje predstavljaju određene opsege intenziteta. Zbog velike razlike u intenzitetima detektovanog zračenja u slučajevima kada se snima sa i bez filtera, nije bilo moguće snimanje sa istim parametrima kamere.

Slika **3.21** prikazuje snimak ukupnog zračenja plazme u opsegu talasnih dužina od 180 nm do 850 nm, za dve različite snage $\langle P_G \rangle$. Strimerski kanal prostirao se duž cele oblasti od vrha elektrode do površine tečnosti. Vertikalna bela linija koja simbolizuje površinu vode uzeta je tako da se nalazi po sredini površine vode neposredno ispod vrha elektrode. Usled kretanja strimera po površini tečnosti i udaljavanja od kamere, na slici postoji prazan prostor između donjeg dela strimerskog kanala i površine tečnosti. Primećena je promena intenziteta zračenja duž ose strimera u slučaju obe snage, što ukazuju na postojanje regija u kojima dolazi do većeg stvaranja ekscitovanih vrsta. Izmerena je i jača emisija iz plazme u donjem delu strimerskog kanala bližem tečnoj meti. Povećanje snage oslobođene u pražnjenju dovodi do radijalnog proširenja provodnog kanala i pojačane emisije zračenja, kao i do širenja kanala u oblasti neposredno uz površinu vode.

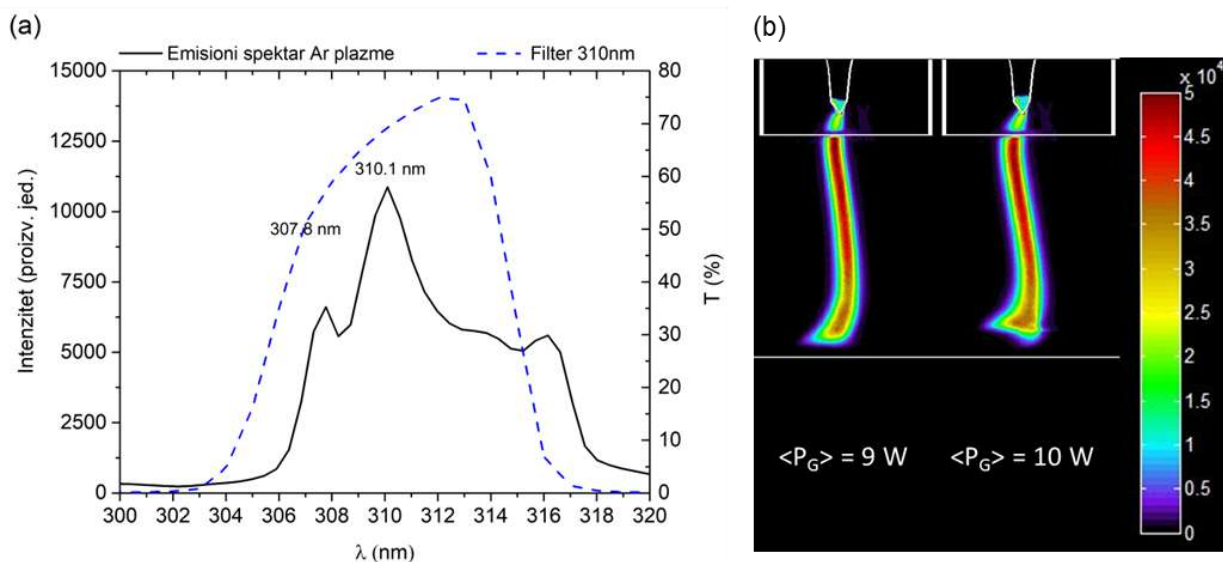


Slika 3.21: Emisija Ar plazme ($q=1\text{slm}$) u kontaktu sa vodom, za dve različite snage pražnjenja. Parametri kamere: dužina perioda snimanja - 1 ms, pojačanje - 0.

Snimanje zračenja iz plazme razloženog po talasnim dužinama

Kako bi se ispitalo zračenje koje potiče od pojedinačnih reaktivnih vrsta korišćeni su uskopojasni optički filteri koji propuštaju zračenje u uskop opsegu talasnih dužina. Za posmatranje emisije koja potiče od pobuđenog molekula OH korišćen je filter koji propušta svetlost u opsegu $310 \text{ nm} \pm 10 \text{ nm}$. Na slici **3.22a** isprekidanom plavom linijom prikazana je zavisnost transparentnosti filtera od talasne dužine svetlosti. U oblasti talasnih dužina svetlosti koje filter propušta, metodom OES detektovane su emisije zračenja koje odgovaraju prelazu OH molekula $A^2\Sigma^+ - X^2\Pi (0,0)$ na $307,8 \text{ nm}$ i $310,1 \text{ nm}$.

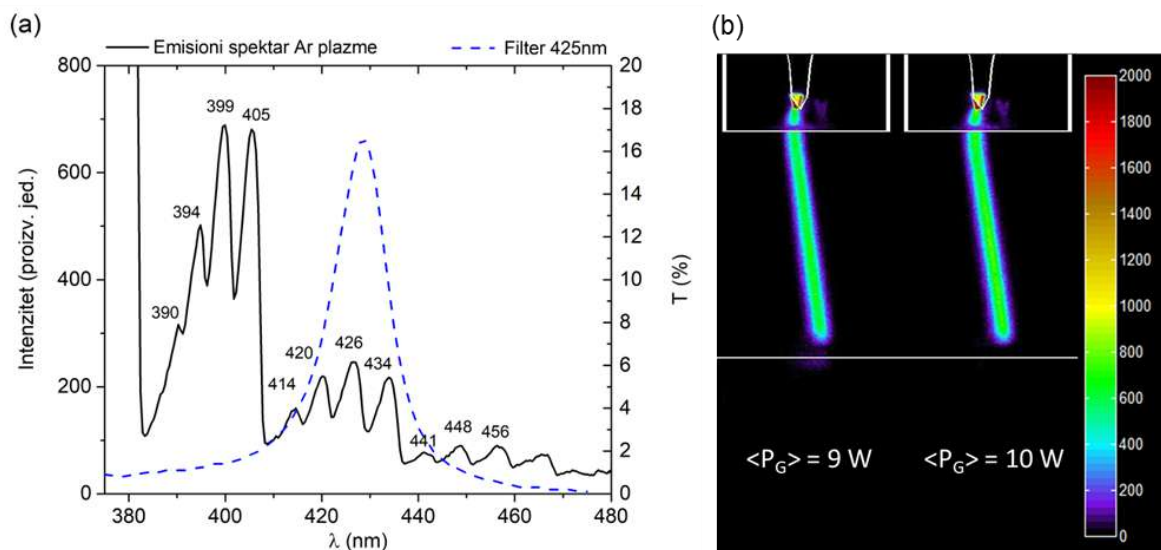
Primećeno je izuzetno intenzivno zračenje u posmatranoj oblasti (videti sliku **3.22b**), koje je intenzivnije u gornjem delu strimerskog kanala, u blizini napajane elektrode i koje po intenzitetu opada ka tečnoj meti. Zračenje iz oblasti između vrha napajane elektrode i donje ivice cevčice bilo je delimično ekranirano zidom cevčice, usled čega je snimljen slabiji intenzitet zračenja od emitovanog. Ovo je posledica toga što je cevčica izrađena od pireksa koji ne propušta zračenje iz UV oblasti. Kao i u slučaju kada je snimano ukupno zračenje iz plazme (slika **3.21**), primećeno je da se sa povećanjem snage oslobođene u pražnjenju strimerski kanal vizuelno širi, naročito u blizini graničnog sloja između gasne i tečne faze.



Slika 3.22: (a) Detektovani emisijski spektar zračenja iz pražnjenja u Ar 1 slm u oblasti talasnih dužina od 300 nm do 320 nm i transparentnost optičkog filtera koji propušta samo svetlost iz ove oblasti i (b) prostorna struktura pražnjenja u ovoj oblasti, za dve različite snage pražnjenja. Posmatrana oblast talasnih dužina odgovara prelazu OH molekula $A^2\Sigma^+ - X^2\Pi (0,0)$ na $307,8 \text{ nm}$ i $310,1 \text{ nm}$. Parametri kamere: dužina perioda snimanja - 5 ms , pojačanje - 50 .

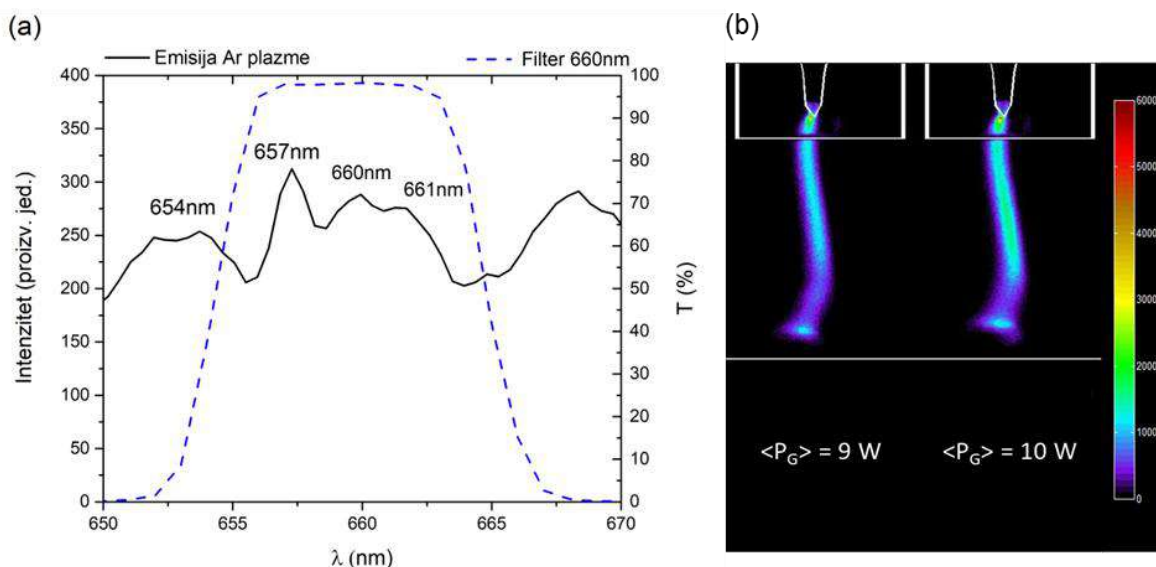
Proučavanje zračenja koje potiče od molekula N_2 izvršeno je sa filterom koji propušta u oblasti talasnih dužina od 370 nm do 470 nm , sa maksimumom transmitivnosti na 428 nm . Ova oblast odgovara prelazima N_2 SPS ($C^3\Pi_u - B^3\Pi_g$) prikazanim na slici **3.23**, čija je emisija niskog intenziteta u odnosu na druge emitovane detektovane prelaze N_2 i prelaze drugih eskcitovanih vrsta. Pored toga, transparentnost korišćenog filtera je izuzetno mala - sa maksimalnom vrednošću od 16% na 428 nm , usled čega je detektovan za red veličine manji intenzitet zračenja nego u slučaju OH molekula.

Dobijena je prilično uniformna raspodela zračenja duž celog kanala strimera, sa najvećim intenzitetom u oblasti oko napajane elektrode, što je prikazano na slici **3.23b**. Prilikom snimanja zračenje u oblasti talasnih dužina od 370 nm do 470 nm nije primećen značajan uticaj promene snage $\langle P_G \rangle$ na intenzitet emisije zračenja i profil pražnjenja.



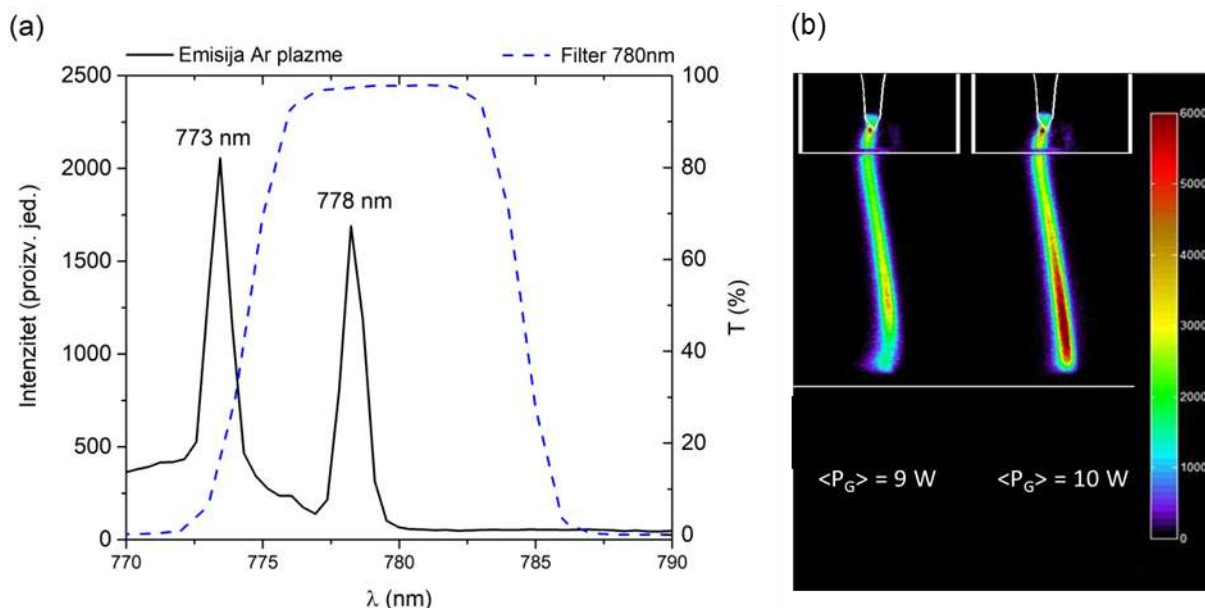
Slika 3.23: (a) Dobijeni emisijski spektar zračenja iz pražnjenja u Ar 1 slm u oblasti talasnih dužina od 370 nm do 470 nm i transparentost korišćenog optičkog filtera i (b) prostorna struktura pražnjenja u ovoj oblasti, za dve različite snage pražnjenja. Emisija zračenja iz ove oblasti odgovara prelazima N_2 SPS ($C^3 \Pi_u - B^3 \Pi_g$). Parametri kamere: dužina perioda snimanja - 5 ms, pojačanje 50.

U oblasti talasnih dužina od 650 nm do 670 nm OES metodom je identifikovano zračenje koje potiče od Balmerove $H\alpha$ linije na 657 nm koje je superponirano sa emisijom N_2 molekula (slika **3.24a**). Međutim, u oblasti koja je obuhvaćena propusnošću korišćenog filtera 660 ± 10 nm, najveći intenzitet ima linija na 657 nm. Struktura pražnjenja emitovanog u ovoj oblasti (slika **3.24b**) se razlikovala od one koja potiče samo od N_2 molekula (**3.23b**). U ovom slučaju nije primećena veća emisija u oblasti elektrode, a intenzitet zračenja opada od elektrode ka tečnosti, a zatim se ponovo povećava pri dodiru strimera i vode. Povećanje snage je uticalo na proširenje kanala i porast intenziteta zračenja.



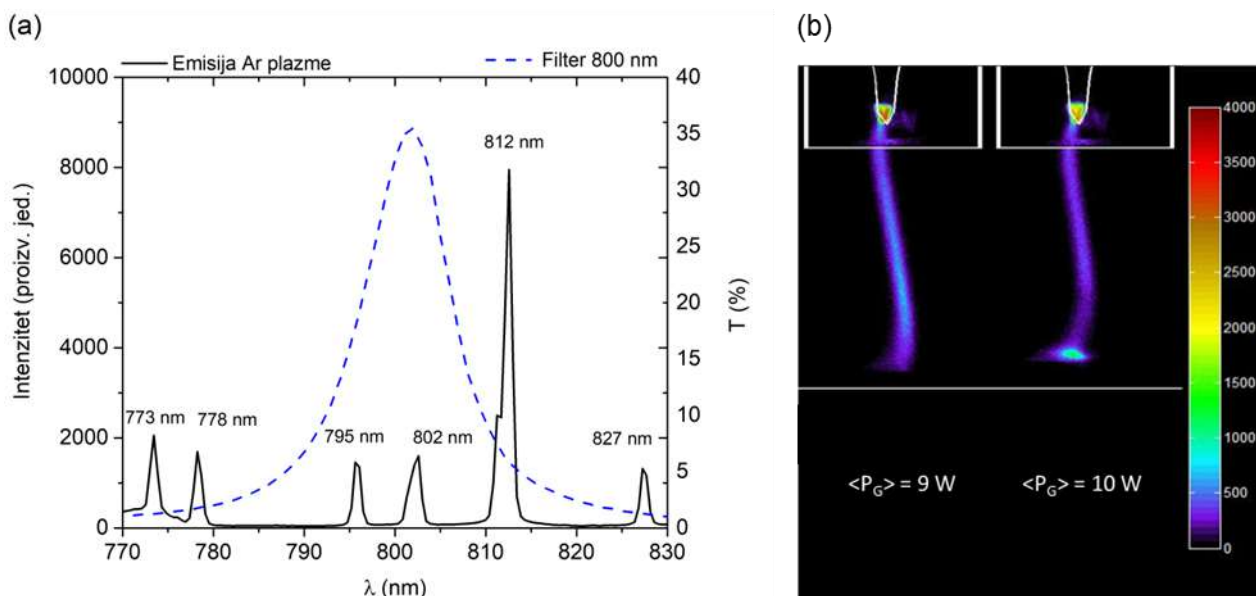
Slika 3.24: (a) Emisija zračenja iz pražnjenja u Ar 1 slm u oblasti na 660 ± 10 nm i transparentnost korišćenog optičkog filtera i (b) prostorna struktura pražnjenja u ovoj oblasti za dve različite snage pražnjenja. Detektovana linija na 657 nm odgovara $H\alpha$ prelazu i zračenje N_2 molekula. Parametri kamere: dužina perioda snimanja - 5 ms, pojačanje 50.

Druga kiseonična vrsta koja je proučavana jeste atomski kisonik O sa prelazom na 780 nm (slika 3.25). Emisija zračenja bila je najintenzivnija u oblasti blizu napajane elektrode i u donjem delu strimerskog kanala u blizini tečnosti. Detekcijom zračenja u datoj oblasti talasnih dužina (780 ± 5 nm), sa povećanjem snage predate u pražnjenju, nije došlo do značajne promene oblika snimljenog profila zračenja, ali je naglo porastao intenzitet zračenja u donjem delu kanala pražnjenja.



Slika 3.25:(a) Emisija zračenja atomskog kiseonika O na 778nm i (b) struktura pražnjenja za dve snage pražnjenja. Parametri kamere: dužina perioda snimanja - 5 ms, pojačanje 50.

U infracrvenom delu spektra od 770 nm do 830 nm posmatrana je i emisija koja potiče od Ar zračenja. Korišćen je optički filter koji je propuštao zračenje koje obuhvata emisiju nekoliko linija Ar (2p-1s), ali i delimično zračenje linije O na 778 nm. deo linije, kao što je prikazano na slici 3.26a. Transmivitnost filtera je bila veoma niska (oko 5%) u oblasti oko 812 nm gde je emitovana najintenzivnija linija Ar, što se veoma odrazilo na snimljenu strukturu pražnjenja koje je prema slici 3.26b veoma slabog intenziteta. Najveći intenzitet je detektovan u oblasti oko napajane elektrode.

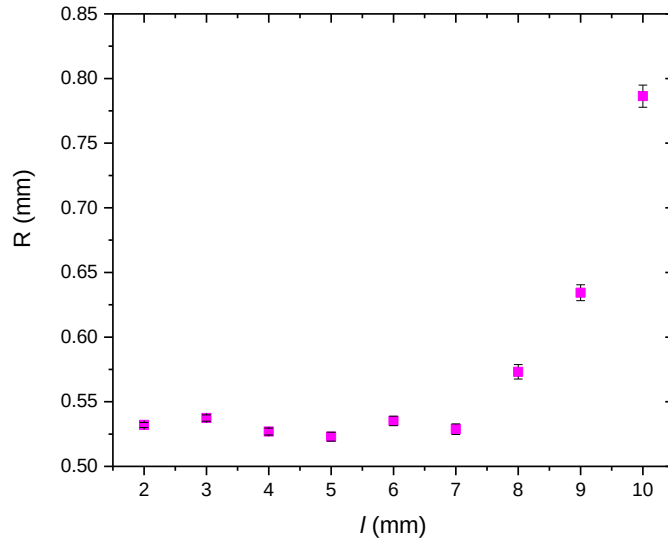


Slika 3.26: (a) Emisija zračenja u infracrvenom delu spektra koja odgovara prelazima Ar i (b) struktura pražnjenja, za dve različite snage pražnjenja. Parametri kamere: dužina perioda snimanja – 5 ms, pojačanje 50.

Određivanje dijametra strimerskog kanala i gustine struje pražnjeja

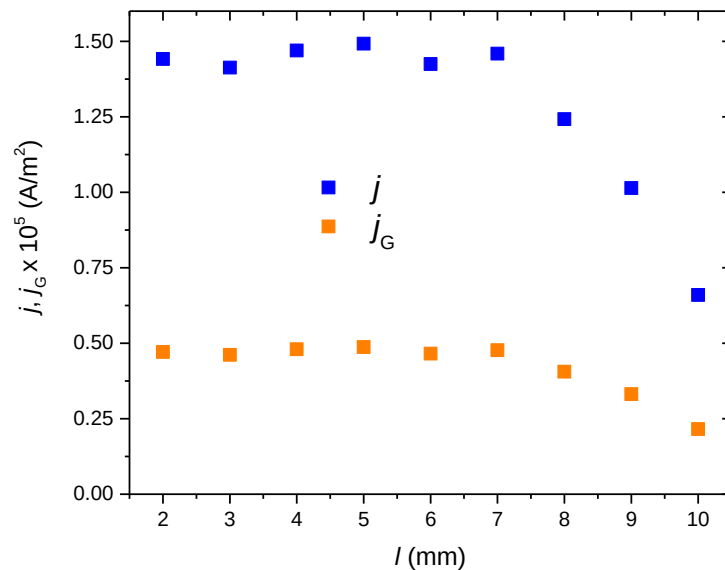
U slučaju kada je snimano ukupno zračenje iz plazme, u opsegu talasnih dužina od 180 nm do 850 nm, za snagu $\langle P_G \rangle = 10$ W (uslovi isti kao kod rezultata sa slike 3.21), sa slika dobijenih sa ICCD kamere izvršeno je merenje dijametra strimerskog kanala i određena je gustina struje pražnjenja. Dijametar je izmeren na devet pozicija na vertikalnoj osi plazma mlaza, između staklene cevi plazma mlaza i površine vode. Merenja nisu izvršena u pozicijama 0 mm i 1 mm jer je zračenje iz tog dela ekranirano staklenom cevčicom.

Korišćena je metoda zasnovana na fitovanju raspodele intenziteta zračenja na datim pozicijama Gausovom funkcijom i određivanjem FWHM – što je uzeto za dijametar strimera (Briels et al., 2008; Chen et al., 2013; Kanmae et al., 2012). Rezultati su prikazani na slici 3.27. Dijametar strimera R imao je gotovo istu vrednost od pozicije 2 mm do 7 mm i iznosio je oko 0,53 mm. Zatim je došlo do naglog širenja strimerskog kanala u blizini površine mete i na površini mete, na rastojanju 10 mm od vrha elektrode, dijametar je bio 0,79 mm.



Slika 3.27: Dijametar strimera R na različitim rastojanjima l od vrha napajane elektrode, mereno na vertikalnoj osi. Određivanje dijametra je urađeno za iste uslove kao za **sliku 3.19** – za ukupno zračenje u celom opsegu talasnih dužina. pri snazi $\langle P_G \rangle = 10$ W. Dijametar strimera dobijen je iz FWHM fitovanjem raspodele intenziteta Gausovom funkcijom, odakle su dobijene i greške merenja.

Pomoću određenog dijametra strimera i izmerenih struja pražnjenja u glavnoj – I_{RMS} i uzemljenoj I_{RMS_G} grani kola, izračunata je gustina struje pražnjenja. U računu je pretpostavljeno da je poprečni presek strimerskog kanala krug, čiji prečnik odgovara dijametru strimera na datoj poziciji. Zavisnosti gustina struja pražnjenja izmerenih u dve grane kola, j i j_G , od rastojanja od napajane elektrode plazma mlaza l su prikazane na slici **3.26**. Vrednosti gustina struja su bile oko $1,45 \cdot 10^5$ A/m² i $0,48 \cdot 10^5$ A/m² pre tačke u kojoj strimerski kanal počinje da se širi ($l=7$ mm), a zatim pri kontaktu pražnjenja i tečnosti padaju na vrednosti od $0,7$ A/m² i $0,2$ A/m², za j i j_G respektivno.



Slika 3.28: Gustina struje pražnjenja na različitim rastojanjima l od vrha napajane elektrode, j – mereno u glavnoj grani kola, j_G – mereno u uzemljenoj grani kola. Apsolutne greške merenja su manje od 1% izmerene vrednosti i nisu prikazane na grafiku.

DISKUSIJA I ZAKLJUČAK

Prikazani su rezultati slikanja vremenski integrisane prostorne raspodele strimerskog pražnjenja u argonu u kondatktu sa destilovanom vodom. Snimljena je ukupna emisija zračenja u opsegu talasnih dužina od 180 nm do 850 nm, za različite snage oslobođene u pražnjenju $\langle P_G \rangle$. Dodatno su korišćeni uskopojasni optički filteru za snimanje emisije zračenja razložene po talasnim dužinama i na taj način su dobijeni profili zračenja za ekscitovane vrste koje su prethodno detektovane OES metod: OH, N₂ SPS, H _{α} , O i Ar.

Dobijeni profil pražnjnja pokazivao je da se strimerski kanal prostire duž čitavog rastojanja $d=10$ mm između napajane elektrode i površine vode. Primećen je pad intenziteta zračenja u od središta strimerskog kanala ka njegovim spoljašnjim delovima (radijalna dimenzija), kao i povećavanje intenziteta zračnja od elektrode ka površini vode (po vertikalnoj osi). Sa povećanjem snage $\langle P_G \rangle$ dolazilo je do radijalnog proširenja kanala, kao i do vizuelnog širenja kanala u neposrednoj blizini vodene mete.

Rezultati dobijeni snimanjem sa optičkim filtrima, pokazali su da je najintenzivnije bilo zračenje koje potiče od OH molekula (oblast oko $\lambda = 310$ nm). Intenziteti drugih reaktivnih vrsta bili su slabiji, ali je kod svih pokazano povećanje intenziteta zračenja sa povećanjem snage $\langle P_G \rangle$. Svi rezultati su uskladu sa rezultatima dobijenim pomoću OES metoda koji su predstavljeni u prthodnom potpoglavlju.

Pomoću dobijenog profila ukupnog pražnjenja strimerskog pražnjenja određen je dijametar provodnog kanala, u različitim pozicijama duž rastojanja d . Primećeno je da je dijametar strimera bio gotovo konstantan od staklene cevčice plazma mlaza do rastojanja od 3 mm iznad površine vode i iznosio je oko 0,53 mm. Od rastojanju 3 mm iznad površine vode do kontakta plazme i vode, dolazi do naglog proširenja kanala čiji dijametar je pri kontaktu sa vodom iznosio 0,79 mm.

Ovi rezultati su upotrebljeni za računanje gustine struje pražnjenja, pomoću izmerenih RMS vrednosti struja u glavnoj i uzemljenog grani kola. U oblasti konstantnog dijametra provodnog kanala, dobijene gustine struja su bile oko vrednosti $j = 1,45 \cdot 10^5$ A/m², mereno u glavnoj grani kola i $j_G = 0,48 \cdot 10^5$ A/m² za merenje u uzemljenoj grani. Sa proširenjem strimerskog kanala, pri kontaktu sa tečnošću, ove vrednosti su efektivno smanjene za više od 50 %.

3.2.3 SNIMANJE KRETANJA FILAMENTARNOG PRAŽNJENJA BRZOM KAMEROM

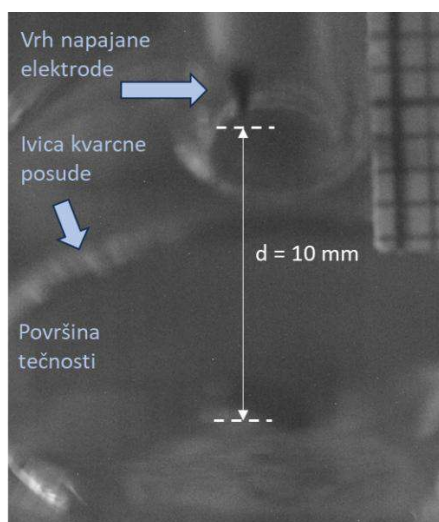
Pored proučavanja prostorne strukture pražnjenja u celoj oblasti između napajane elektrode i tečnosti, korišćena je i metoda snimanja brzom kamerom kako bi se proučavalo kretanje strimera i formiranje filamentarnog pražnjenja u zoni interakcije plazme i tečne mete. Kako se u primenama plazma mlaz koristi pri tretmanu tečnosti različitih električnih provodljivosti - σ , urađeno je snimanje opsegu σ od 2-530 $\mu\text{S/cm}$. Korišćeni su uzorci destilovane vode (DV, $\sigma = 2 \mu\text{S/cm}$), fiziološkog rastvora (FR) provodnosti 100 $\mu\text{S/cm}$ i vode iz gradskog vodovoda (voda sa česme, VČ, $\sigma = 530 \mu\text{S/cm}$).

Eksperimentalna postavka korišćena pri merenju prikazana je u potpoglavlju 2.1.3. Parametri u sistemu su bili isti kao i u slučaju snimanja prostorne evolucije pražnjenja opisane u potpoglavlju 3.2.2.: protok argona je bio 1 slm, uzorak zapremine 4 ml se nalazio u kvarcnoj posudi, na rastojanju $d=10 \text{ mm}$ od površine tečnosti.

Brza kamera bila je postavljena pod uglom od 50° u odnosu na vertikalnu osu plazma mlaza, na rastojanju 20,5 cm od vrha napajanje elektrode. Svaki video zapis se sastojao od 36000 kadrova, a rezolucija je bila 512x600 piksela. Maksimalan broj kadrova po sekundi je bio 10000 (eng record rate = 10000 fps), dok je dužina ekspozicije podešavana u odnosu na intenzitet zračenja plazme za date parametre u sistemu. Time je svaki snimljeni kadar zabeležio emisiju iz pražnjenja koja je efektivno usrednjena iz otprilike 20-33 perioda pobudnog signala.

Za svaku tečnu metu, snimljeni su video zapisi pražnjenja za snagu $\langle P_{G\_NET} \rangle = 10 \text{ W}$. Pražnjenje za druge vrednosti snaga pražnjenja je takođe snimljeno, ali se vizuelno nije značajno razlikovalo (videti objašnjenje u potpoglavlju 3.2.2.) i ovde nije prikazano.

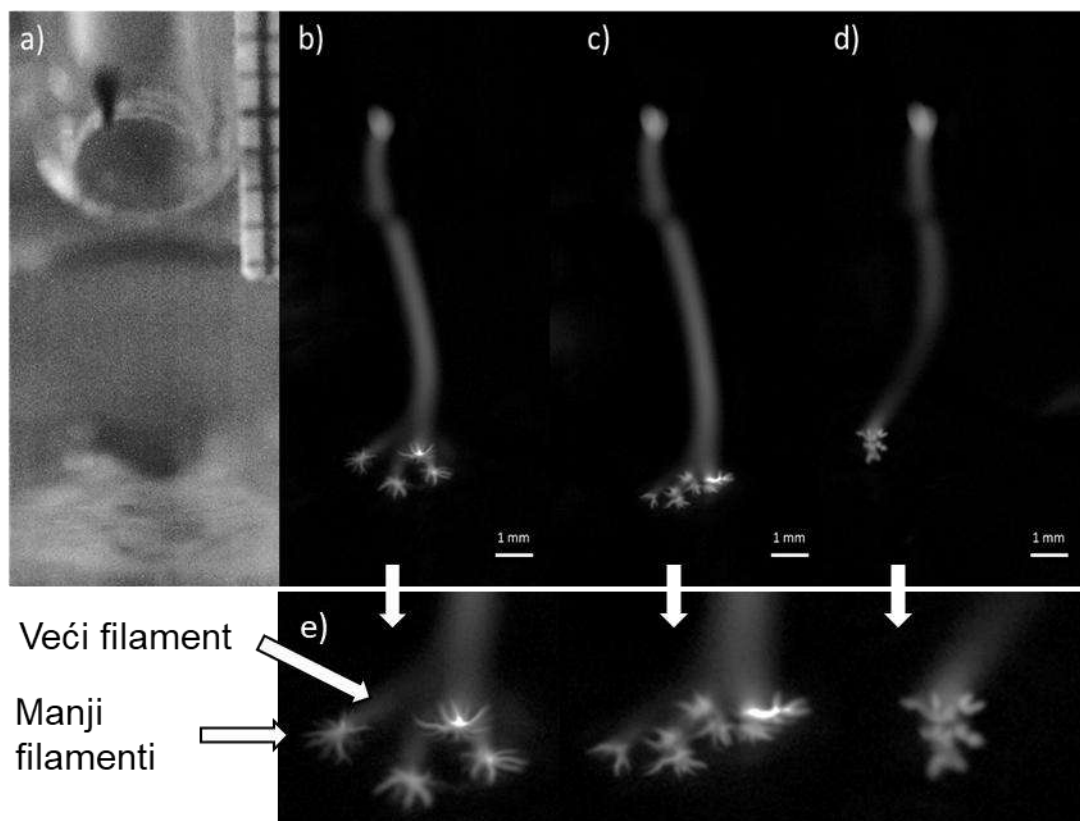
Na slici 3.29 prikazana je fotografija na kojoj se vidi položaj elektrode plazma mlaza u odnosu na površinu tečnosti, sa milimetarskom skalom.



Slika 3.29 Prikaz fokusa brze kamere (pod uglom od 50° u odnosu na vertikalnu osu) na vrh elektrode plazma mlaza i površinu tečnosti u kvarcnoj posudi.

Struktura pražnjenja u zavisnosti od provodnosti mete

Na slici 3.30 prikazana je snimljena struktura pražnjenja, u slučajevima kada su tretirani uzorci bili: b) DV ($\sigma = 2 \mu\text{S/cm}$), c) FR ($\sigma = 100 \mu\text{S/cm}$) i d) VČ ($\sigma = 530 \mu\text{S/cm}$). Svaka slika odgovara jednom kadru napravljenog video snimka. Vreme ekspozicije je prilagođavano intenzitetu emisije zračenja u konkretnom slučaju i iznosilo je $69 \mu\text{s}$, $61 \mu\text{s}$ i $98 \mu\text{s}$, u slučaju DV, FR i VČ respektivno. Jedan period oscilovanja VN signala kojim je napajan plazma mlaz bilo je $3 \mu\text{s}$, tako da jedan kadar predstavlja vremenski integrisano pražnjenje u toku 20-33 perioda oscilovanja VN signala. Slike pražnjenja na slici 3.30 prikazane su podešavanjem kontrasta i osvetljenosi originalnih kadrova, kako bi se jasnije prikazala struktura filamentarnog pražnjenja.

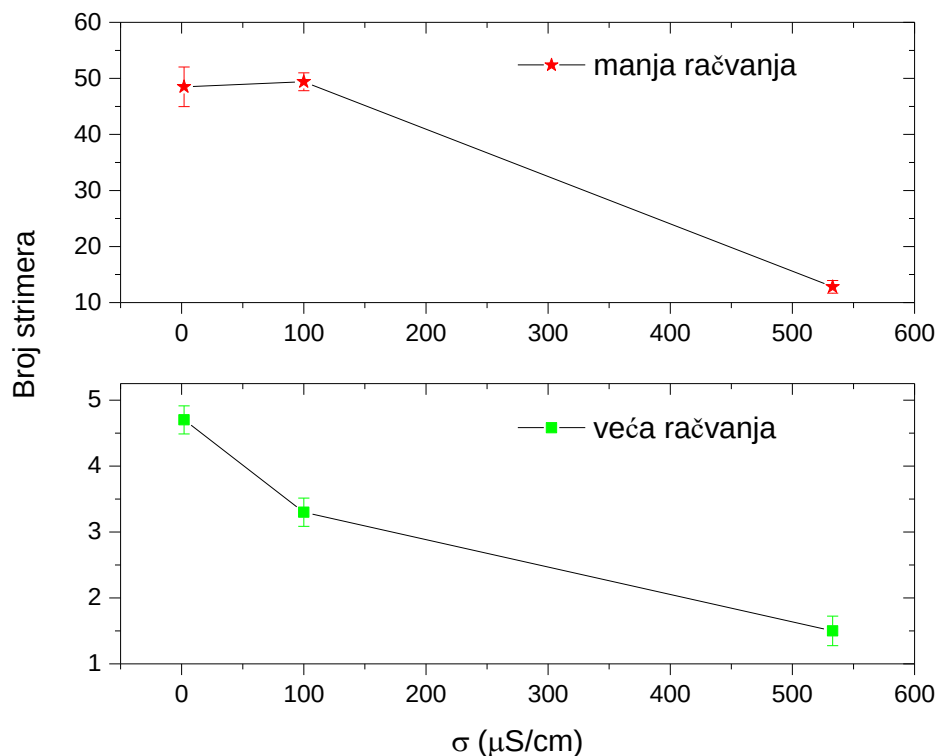


Slika 3.30: a) Prikaz položaja igle plazma mlaza tokom simanja. Prikaz strukture strimerskog pražnjenja sa jednog kadra video zapisa kada je tečna meta bila: b) DV, c) FR ($100 \mu\text{S/cm}$), d) VČ i e) uvećan prikaz pražnjenja pri kontaktu sa tečnom metom. Vreme ekspozicije: b) $69 \mu\text{s}$, c) $61 \mu\text{s}$, d) $98 \mu\text{s}$, rezolucija nakon obrade slika a)-d) 308×600 piksela.

U sva tri slučaja strimersko pražnjenje se prostiralo od vrha napajane elektrode do površine tečnosti. Kada je meta bila DV (slika 3.30b), sa snimaka se videlo da se glavni strimer pri kontaktu sa tečnošću račvao prvo na 3-6 većih filamenata, koji su se zatim račvali u veći broj manjih filamenata (videti uvećan prikaz). Širina većih filamenata bila je manja od širine početnog strimerskog kanala, koji se vertikalno prostirao od vrha igle ka površini tečnosti, dok su manji filamenti bili veoma tanki i kratki. Položaj većih filamenata je bio pod uglom u odnosu na vertikalni položaj glavnog strimera, dok je položaj manjih strimera bio gotovo radijalan, duž površine tečnosti.

Sa povećanjem σ na 100 $\mu\text{S}/\text{cm}$ (**3.30c**), dolazi do smanjenja broja većih filamenata (račvanja) i skraćivanja svih filamenata. Veći filamenti su kraći u odnosu na slučaj kod DV i bilo ih je teže razlikovati. Dalje povećanje σ na 530 $\mu\text{S}/\text{cm}$ (**3.30d**), dovelo je do smanjivanja površine kontakta strimera i vode, a filamenati su bili manje uočljivi nego kod prethodna dva slučaja.

Pojava većih i manjih filamenata na pojedinačnim kadrovima, nastaje usled promena pozicija strimera tokom trajanja ekspozicije kamere. Zavisnost broja filamenata na jednom snimljenom kadru od σ prikazana je na slici **3.31**. Prikazani rezultati predstavljaju srednju vrednost broja filamenata za deset različitih kadrova, za datu tečnu metu, a prikazana je i standardna greška za deset nezavisnih merenja.

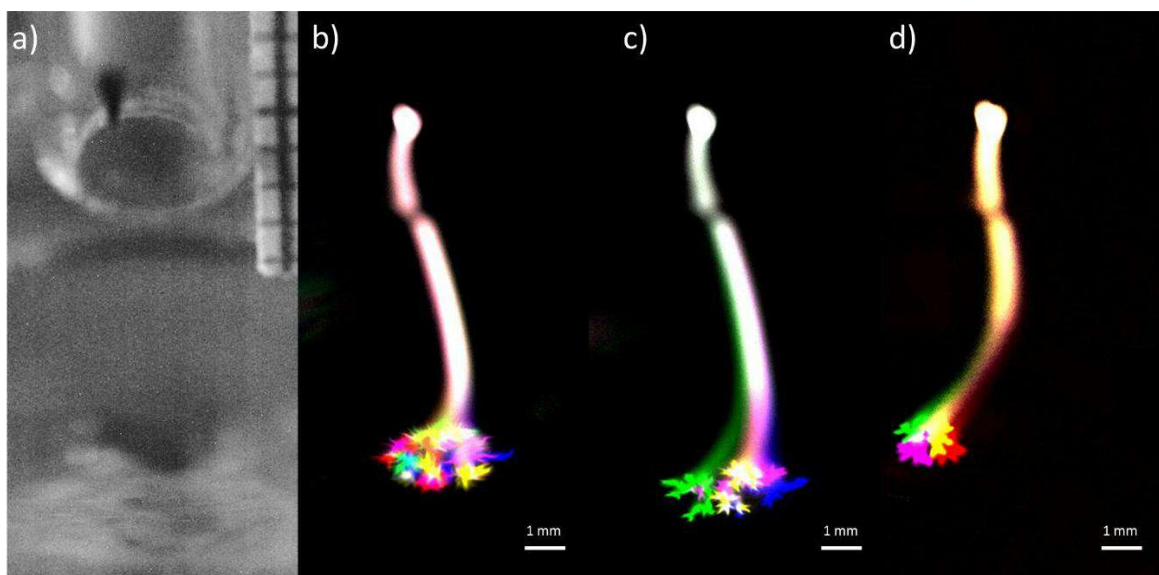


Slika 3.31: Srednji broj manjih i većih filamenata snimljenih u toku 100 μs u zavisnosti od električne provodnosti tečne mete. Prikazane su standardne greške merenja dobijene iz deset nezavisnih merenja. U toku 100 μs snimanja detektovano je zračenje emitovano tokom 23 (DV), 20 (FR 100 $\mu\text{S}/\text{cm}$) odnosno 33 (VČ) perioda oscilovanja pobudnog signala.

Povećanje σ tečne mete, dovelo je do smanjenja broja većih račvanja strimera, odnosno do lokalizacije centralnog kanala. Povećanje σ sa 2 $\mu\text{S}/\text{cm}$ na 100 $\mu\text{S}/\text{cm}$ dovelo je do blagog povećanja broja manjih filamenata, ali u okviru greške merenja može se reći da nije došlo do značajne promene. Smanjenje broja i većih i manjih filamenata bilo je veoma izraženo pri povećanju σ sa 100 $\mu\text{S}/\text{cm}$ na 530 $\mu\text{S}/\text{cm}$.

Efektivna kontaktna površina strimerskog pražnjenja

Promene pozicija strimera uočene su kako na pojedinačnim kadrovima (videti sliku 3.30), tako i tokom čitavog trajanja snimanja (0,9 s za jednu tečnu metu). U svim slučajevima je primećeno da pored lokalnog grananja centralnog kanala (promena pozicije donjeg dela strimerskog kanala neposredno pre kontakta sa tečnošću) dolazi i do njegovog pomeranja u odnosu na horizontalnu osu plazma mlaza. Na slici 3.32 prikazane su različite pozicije strimerskog pražnjenja, dobijene sa različitih kadrova, za svaku tečnu metu. Urađeno je preklapanje snimljenih kadrova, tako da svaka boja predstavlja pražnjenje sa jednog kadra, za datu metu. Pozicija centralnog strimera se nije menjala značajno između uzastopnih kadrova, stoga su za prikaz izabrani oni kadrovi koji ilustruju najveće promene pozicija.

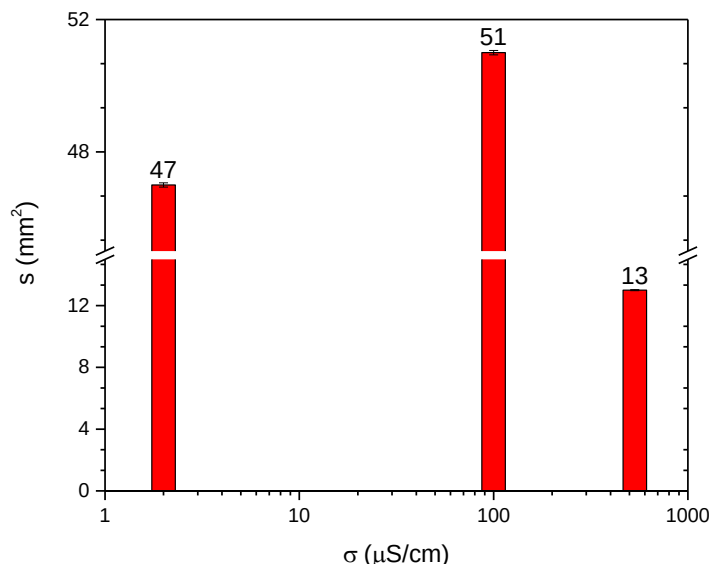


Slika 3.32: Različite pozicije strimera tokom tretmana tečnosti u trajanju od 0,9s snimanja (b-d), za DV, FR (100 $\mu\text{S}/\text{cm}$) i VČ, respektivno.

Iako se sa pojedinačnih kadrova uočava da je najveće širenje pražnjenja u slučaju DV, do većih promena položaja centralnog strimera dolazi u slučaju FR (100 $\mu\text{S}/\text{cm}$) (slika 3.32b), što za posledicu ima da je veća površina mete izložena dejstvu plazme tokom trajanja tretmana. Najveća lokalizovanost strimerskog kanala bila je i u ovom slučaju kod mete sa najvećom vrednošću σ – VČ.

Izvršeno je merenje kontaktne površine plazme i tečnosti za svaku metu, tako što je pretpostavljeno da je širenje strimera konusno i aproksimativno je uzeto da je kontaktna površina plazme i tečnosti jednaka površini kruga koju strimer prebriše tokom trajanja snimanja. Prečnik kruga je određen tako što su u vremenskom intervalu od 0,9 s, koliko je trajalo snimanje, izabrana dva kadra na kojima je najudaljeniji međusobni položaj strimera u odnosu na osu plazma mlaza. Zatim je na oba kadra određena pozicija tačke najudaljenije od vertikalne ose plazma mlaza i izračunato je rastojanje između te dve tačke.

Rezultati merenja prikazani su na slici 3.33. Prikazana je i procenjena apsolutna greška merenja površine dobijena merenjem rastojanja na kadru pomoću milimerarske skale (videti sliku 3.32a). U slučaju vrednosti σ od 2 $\mu\text{S}/\text{cm}$ i 100 $\mu\text{S}/\text{cm}$, kontaktna površina iznosila je 47 mm^2 i 50 mm^2 , dok je u slučaju VČ (533 $\mu\text{S}/\text{cm}$) bila značajno manja i iznosila je 13 mm^2 . Porastom vrednosti σ sa 100 $\mu\text{S}/\text{cm}$ na 533 $\mu\text{S}/\text{cm}$, došlo je do smanjenja kontaktne površine za oko 75%.



Slika 3.33: Izmerena efektivna kontaktna površina strimerskog pražnjenja i tečnosti – s , u zavisnosti od provodnosti mete - σ , za slučajeve DV, FR(100 $\mu\text{S/cm}$) i VČ.

DISKUSIJA I ZAKLJUČAK

U okviru ovog potpoglavlja predstavljeni su rezultati proučavanja strimerskog pražnjenja snimanjem kretanja i grananja-filamenata po površini tečnosti. Posmatrana je vremenski integrisana (za 20-33 perioda oscilovanja pobudnog signala) struktura pražnjenja u zavisnosti od σ mete. Pored toga određen je broj račvanja strimerskog kanala i procenjena je kontaktna površina pražnjenja i tečnosti za tri mete DV, FR i VČ u opsegu σ od 2 – 530 $\mu\text{S/cm}$.

Račvanje strimera u blizini površine tečne mete primećeno je u celom opsegu σ tečnosti. Sa dobijenih snimaka (u oblasti talasnih dužina od 400 nm do 1000 nm) se može videti da sa povećanjem σ tečne mete dolazi do:

- skraćivanja dužine većih filamenata, dok dužina manjih filamenata ostaje nepromenjena u oblasti od 2 - 100 $\mu\text{S/cm}$, a zatim dolazi do značajnog skraćivanja za 530 $\mu\text{S/cm}$
- smanjenja broja filamenata,
- smanjenja intenziteta detektovanog zračenja.

Izmerene su karakteristične dimenzije kontakta plazme i tečnosti za sve tri vrednosti σ mete. Pretpostavljajući da je kontaktna površina pražnjenja i mete kružnog oblika, izmereni su (sa vremenski integrisanih snimaka, u toku ~ 1 s tretmana) prečnici kontaktne površine u opsegu od 2 mm do 4 mm.

Dobijeni rezultati su u skladu sa rezultatima prikazanim u drugim istraživanjima plazma mlazeva u kontaktu sa različitim metama: 1- 2 mm kod kiNPen uređaja (Reuter et al. 2018), 2-5 mm kod plazmam mlaza u helijumu (Begum et al., 2023) i oko 4 mm kod argonskog plazma mlaza sa centralnom napajanom elektrodom (Onyshchenko et al., 2016).

U slučaju DV, gde se može smatrati da se tečna meta ponaša kao dielektrik s obzirom na nisku vrednost σ , uočen je najveći broj filamenata na površini tečnosti. Intenzitet i dužina filamenata su bili značajno veći nego kod VČ. U drugim istraživanjima gde je eksperimentalno i u modelu (Herrmann et al., 2023, 2025; Zhang et al., 2021) proučavano strimersko pražnjenje u kontaktu sa dielektričnom metom, pokazano je da kako se strimerski kanal približava površini tretirane dielektrične mete, dolazi do promene u pravcu prostiranja pražnjenja iz vertikalnog u horizontalno. Kako je za širenje strimerskog kanala neophodno prostorno naelektrisanje u glavi strimera i formiranje elektronskih lavina oko nje (videti poglavlje 1.1), smatra se da kada se strimer približi dielektričnoj meti rastojanje postane previše malo što dovodi do smanjenja učestanosti odvijanja procesa koji daju slobodne elektrone (uglavnom fotojonizacije) za nastajanje elektronskih lavina. Zbog toga dolazi do nagomilavanja slobodnog prostornog naelektrisanja po površini dielektrične mete. Kada električno polje ovih naelektrisanja postane dovoljno visoko, strimer prateći gde je intenzitet polja najveći – počinje da se kreće radijalno - po površini mete. Da bi se strimersko pražnjenje održalo u ovakvom sistemu, strimer mora da se raširi po većoj površini mete.

Kod tečnosti sa većom vrednošću σ (slučaj VČ), prisustvo jona u tečnosti utiče na svojstva strimera i njegovu dinamiku širenja usled njihovih interakcija sa primenjenim električnim poljem i sa naelektrisanim vrstama iz glave strimera. Pod dejstvom spoljašnjeg polja dolazi do preraspodele jona u tečnosti, a oni učestvuju i u procesima neutralizacije naelektrisanih vrsta koje pražnjenje deponuje na površinu tečnosti. Pored toga, naelektrisanja iz pražnjenja se brže gube jer struja efikasnije teče kroz metu. Usled ovih procesa linije polja su koncentrisane lokalno na manjoj površini, strimer postaje lokalizovan i fokusiran na manju poveršinu (Akishev et al., 2019; Herrmann et al., 2025). Dolazi do smanjenja kontaktne površine i energija pražnjenja se oslobađa na manjoj površini, odnosno tretira se manja površina mete.

Razumevanje prostiranja strimerskog pražnjenja kako u gasnoj fazi tako i na površini tečnosti i određivanje kontaktne površine pražnjenja i mete, veoma je važno sa gledišta primene plazma mlaza. Osobine tečnosti kao što je σ i prisustvo jona u tretiranoj tečnosti, utiču na naelektrisane čestice i električno polje fronta strimera, a samim tim i na dinamiku i morfologiju pražnjenja. Sa druge strane, u tretmanima zagađene vode plazmom, gde su često dominantni površinski zagađivači, veličine kontaktne površine u velikoj meri utiče na efekat tretmana, a u sprezi je sa σ tečne mete (videti potpoglavljje 4.2.1) (Khan et al., 2022).

3.2.4 ZAKLJUČAK

U okviru ovog poglavlja urađena je dijagnostika gasne faze pri čemu su korišćene tri optičke metode:

- Optička emisiona spektroskopija (OES)
- Slikanje prostorne evolucije strimerskog praznjenja u kontaktu sa vodom iCCD kamerom
- Snimanje razvoja i kretanja filamentarnog praznjenja brzom kamerom

Pomoću OES metode identifikovane su pobuđene vrste koje nastaju u gasnoj fazi praznjenja. Ispitivano je kako vrsta i protok radnog gasa utiču na režim praznjenja i ekscitovanje reaktivnih azotnih i kiseoničnih vrsta. Kod strimerskog praznjenja u kontaktu sa vodom pokazano je prisustvo NO, OH, N₂, N₂⁺, H_α, O, pored pobuđenih vrsta korišćenog radnog gasa (Ar ili He). Kada je kao radni gas korišćen Ar dobijeni su značajno intenzivniji spektri zračenja nego u slučaju He. Dodavanje sintetičkog vazduha u smešu radnog gasa kod Ar dovelo je do povećanja intenziteta zračenja identifikovanih pobuđenih vrsta, ali ne i do pojave novih linija u spektru.

Na osnovu dobijenih rezultata, zaključeno je da je u pogledu daljih primena ovog tipa praznjenja u tretmanu tečnih uzoraka korisnija upotreba argonskog praznjenja. Jači intenziteti emitovanih linija ukazivali su na veće koncentracije reaktivnih vrsta, što bi predstavljalo veću verovatnoću deponovanja reaktivnih vrsta u tečnost. Zbog toga je u daljem proučavanju gasne faze praznjenja korišćen isključivo argon kao radni gas.

Upotrebom iCCD kamere snimljena su profili argonskog praznjenja. Pokazano je da se praznjenje prostire duž celog međuelektrodnog rastojanja i da intenzitet opada od središta ka obodima strimera i da raste od vrha napajanje elektrode ga površini tečnosti. Povećanje snage oslobodene u praznjenju dovelo je do širenja provodnog kanala. Snimanje zračenja razloženog po talasnim dužinama pomoću uskopropusnih optičkih filtera, pokazalo je da je najintenzivnije zračenje koje je poticalo od OH molekula, što je u skladu i sa rezultatima OES. Sa dobijenih iCCD snimaka određen je i dijametar praznjenja (0,5-0,8 mm) i procenjena je gustina struje praznjenja ($j = 0,5 - 1,5 \cdot 10^5 \text{ A/m}^2$).

Na kraju, korišćenjem brze kamere snimljeno je kretanje i račvanje strimerskog kanala pri kontaktu sa površinom vode. Dobijeni su snimci vremenski integrisani snimci strukture praznjenja za približno 20-30 perioda oscilovanja primenjenog visokonaponskog signala. Urađena su merenja za vodene uzorke u opsegu σ od 2-533 $\mu\text{S/cm}$. Pokazano je da provodnost mete utiče na dužinu i broj filamenata i intenzitet ukupnog detektovanog zračenja (sve tri veličine su opadale sa porastom σ). Procenjena je površina kontakta plazme i tečnosti. Uz pretpostavku da je ona kružnog oblika, procenjeno je da je njen prečnik od 2-4 mm. Kao i kod ostalih veličina, primećeno je da kontaktna površina opada sa povećanjem σ . Dobijeni rezultati su naročito značajni u oblasti trtmana tečnih uzoraka plazmom, jer će efekat tretmana u velikoj meri zavisiti od početnih osobina tečnosti.

4. PRIMENA PLAZMA MLAZA SA ŠILJASTOM ELEKTRODOM U BIOTEHNOLOGIJI I TEHNOLOGIJI PREČIŠĆAVANJA VODE

U okviru ovog dela disertacije urađena su istraživanja u oblasti novih primena neravnotežne plazme u okviru aktuelnih interdisciplinarnih pravaca istraživanja. Prva oblast primene je podrazumevala **tretmane čiste vode plazmom i pravljenje PAV-a kojim su dalje tretirni biološki uzorci** (cele biljke, semena, biljna tkiva) i ispitivani su procesi koji su prouzrokovani u biljnim ćelijama (potpoglavlje 4.1). Druga oblast primene obuhvatala je **prečišćavanje vode zagađene organskim zagađivačima uz pomoć plazme** (potpoglavlje 4.2).

Eksperimentalna postavka korišćena za tretmane čiste i zagađene vode ranije je prikazana i detaljno opisana u poglavlju 2.1. Plazma mlaz napajan je kontinualnim VN sinusnim signalom frekvencije 340 ± 10 kHz u svim eksperimentima. U okviru svakog narednog potpoglavlja date su konkretne vrednosti parametara korišćene za odgovarajuće tretmane tečnosti. U potpoglavlju 4.3 dati su opšti zaključci o uspešnosti upotrebe hladne plazme na atmosferskom pritisku u opisanim oblastima primene.

4.1 TRETMANI BILJNOG MATERIJALA PAV-OM

U ovom delu istraživanja korišćena su dva biljna modela za ispitivanje uticaja PAV-a na biljni svet. Cilj je bio da se ispita toksičnost PAV-a kao i da se pokaže da je primena plazme efikasan metod za podsticanje procesa u biljkama koji su od značaja za različite oblasti u industriji hrane i biotehnologiji.

Prvi eksperiment je obuhvatao tretmane biljnog tkiva - kalusa šargarepe (lat. *Daucus carota subsp. sativus*) PAV-om kako bi se oksidativnim stresom podstakla proizvodnja sekundarnih metabolita koji se koriste u industriji hrane i kozmetici (potpoglavlje 4.1.1).

U drugom eksperimentu, biljka sočivica (*Lemna Minor*) je korišćena pri ispitivanju toksičnosti PAV-a (poglavlje 4.1.2). Proučavan je moguć uticaj PAV na živi svet ukoliko bi takva tečnost dospela u vodene sisteme. Korišćene su dve različite matrice za razblaživanje PAV-a - voda sa česme (laboratorijski sistem) i rečna voda (realan sistem), kako bi se ispitalo koliki udeo PAV u vodi je toksičan. Ispitivano je i da li postoji pozitivnih efekata PAV – a na biljke.

4.1.1 EFEKAT TRETMANA NA KALUSE ŠARGAREPE (*DAUCUS CAROTA* SUBSP *SATIVUS*)

UVOD

Šargarepa (lat. *Daucus carota subsp. sativus*) je veoma cenjena povrtarska kultura sa dobro poznatom fiziologijom i sekvenciranim genomom, a danas se u velikoj meri proizvodi i široko koristi kao model-biljka u biologiji i biotehnologiji. Pored toga, to je bila jedna od nekoliko vrsta korišćenih za razvoj tehnika uzgoja biljnog tkiva, uključujući indukciju kalusa i njegovo održavanje *in vitro*.

Kultura kalusa šargarepe *in vitro* je uspostavljena i gajena u laboratoriji za kulturu tkiva u *Institutu za biološka istraživanja "Siniša Stanković"*, Odeljenje za fiziologiju biljaka. Uz saradnju sa kolegicama dr Milicom Milutinović i dr Suzanom Živković obezbeđen je pristup biološkom materijalu za tretmane PAV-om i planirani su i sprovedeni eksperimenti vezani za biološke uzorke.

Kalusi su tretirani sa dva tipa PAV kako bi se ispitao uticaj osobina PAV na kaluse. U eksperimentu su korišćeni kalusi dve sorte šargarepe – narandžaste i crne. Praćene su morfološke promene (parametri rasta) i metabolički status kalusa tretiranih PAV-om, koji je uključivao promene u profilu specijalizovanih (sekundarnih) metabolita i aktivnosti antioksidativnih enzima kao što su peroksidaze (POX), katalaze (CAT) i superoksid dismutaze (SOD).

EKSPERIMENTALNA POSTAVKA I METODE

Tretmani destilovane vode plazmom

U eksperimentu, rastojanje između napajane elektrode i površine vode bilo je $d = 10$ mm, a ukupni protok radnog gasa 1 slm. U okviru ovog eksperimenta urađena su dva različita tretmana destilovane vode ($\text{pH} = 6,9 \pm 0,4$; $\sigma = 16,0 \pm 0,8 \mu\text{S/cm}$), gde je početna zapremina uzorka bila 15 ml. Parametri prilikom tretmana dati su u tabeli 4.1. Prvi tretman je trajao 5 min i kao radni gas korišćena je smeša Ar+3%Vazduh. U drugom tretmanu vreme tretiranja je povećano na 20 min i korišćen je čist Ar. Kako bi se obezbedilo da tokom celog perioda tretiranja čitava zapremina vode bude izložena dejstvu plazme, korišćena je magnetna mešalica (BOECO MSH130). Srednje vrednosti visokog napona primenjenog na napajanu elektrodu, struje kroz uzemljenu granu kola i snage deponovane u tretirani uzorak bile su $V_{\text{RMS}} = 1,3$ kV, $I_{\text{RMS}_G} = 10\text{mA}$ i $\langle P_{\text{NET}_G} \rangle = 10$ W, respektivno.

Tabela 4.1: Parametri prilikom tretmana za pravljenje PAV-a.

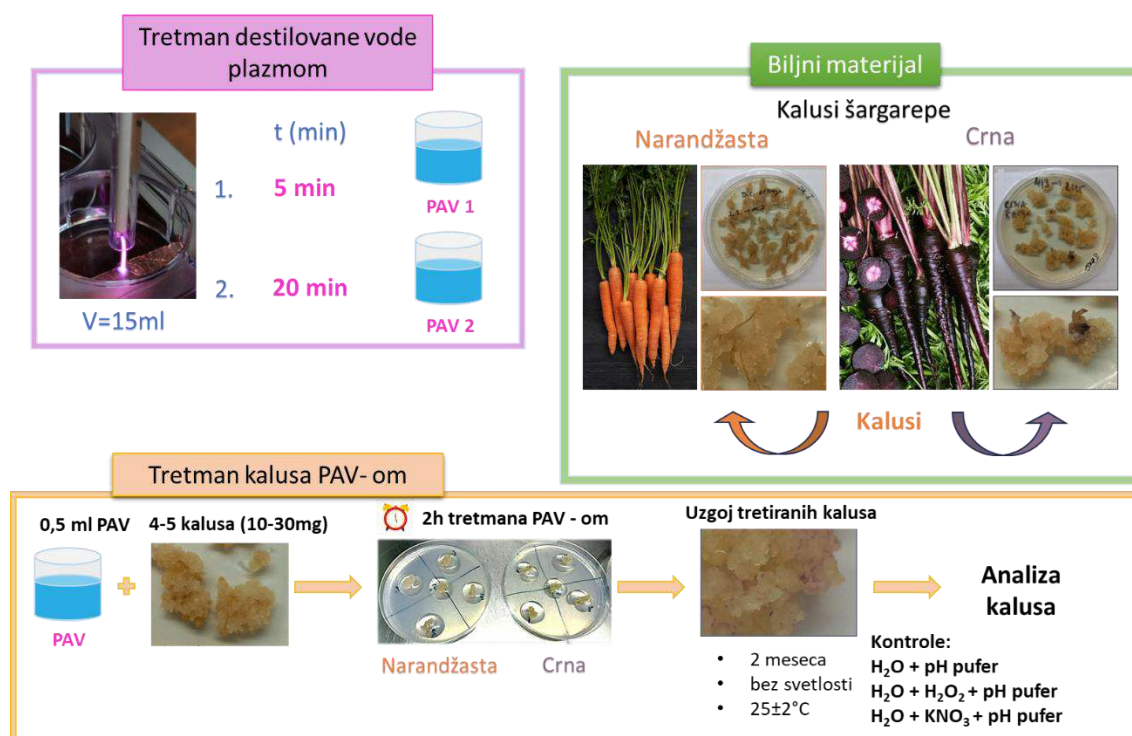
Redni broj	Uzorak	t	Radni gas	Protok Ar	Protok sintetičkog vazduha	Magnetna mešalica
		min		slm	sccm	
1	PAV5	5	Ar	1,00	0	ne
2	PAV20	20	Ar + Sintetički vazduh	0,97	30	da

Kultura kalusa *in vitro*

Za uspostavljanje aseptične kulture kalusa korišćeni su odsečki korenova biljaka šargarepe (1 cm) koje su gajene *in vitro* na MS čvrstoj hranljivoj podlozi sa 3% saharoze i 100 mg l⁻¹ mio – inozitola (Murashige & Skoog, 1962). Formirani kalusi dalje su gajeni u mraku, na 25±2°C, na istoj hranljivoj podlozi koja je sadržala ekvimolarne koncentracije (1 mg l⁻¹) biljnih regulatora rasta 2,4-dihlorfenoksi sirćetne kiseline (2.4-D) i kinetina (KIN).

Tretman kalusa PAV-om

Grupa od 4 - 5 kalusa (10 mg - 30 mg) stavljena je u petri posudu (Ø 90 mm) i prelivena sa 0,5 ml PAV-a ili kontrolnim tečnim uzorkom, tako da im je cela površina pokrivena tečnošću. Kontrolni uzorci bili su destilovana voda sa prilagođenom pH vrednošću, sa ili bez odgovarajuće količine KNO₃ ili H₂O₂ (videti sliku 4.1). Nakon 2 h tretmana, tečnost je uklonjena sa kalusa, a oni su dalje uzgajani tri meseca u klima komori u mraku na 25±2°C. Porast sveže mase je određivan u intervalima od po deset dana.



Slika 4.1: Šematski prikaz koraka u eksperimentu sa kalusima.

Detalji o metodama izolacije ukupnih proteina, spektrofotometrijske analize aktivnosti enzima i određivanja sadržaja fenola i flavonoida mogu se naći u **Prilogu 1** ove disertacije.

REZULTATI I DISKUSIJA

Karakteristike PAV-a

Kako bi se odredila efektivnost tretmana plazmom, odmah nakon tretmana urađena su merenja pH vrednosti uzoraka i koncentracije reaktivnih vrsta. Rezultati merenja fizičko-hemijskih karakteristika PAV uzoraka prikazani su u tabeli 4.2.

Tabela 4.2: Karakteristike PAV uzoraka. V – zapremina nakon tretmana, c – koncentracija. Prikazane su srednje vrednosti i odgovarajuće standardne greške merenja. Merenja su urađena odmah nakon tretmana.

Redni broj	Uzorak	V	pH	c(H ₂ O ₂)	c(NO ₃ ⁻)
		ml		mg/l	mg/l
1	PAV1	14	3,48±0,04	42±4	33±3
2	PAV2	12	2,82±0,08	93±7	104±3

Kod oba tretirana uzorka primećen je značajan pad pH vrednosti, sa početne vrednosti 6,90 na 3,48 nakon 5 min tretmana (PAV1) i 2,82 nakon 20 min tretmana (PAV2). Za niske pH vrednosti tretiranih vodenih uzoraka, odovorne su dugoživeće vrste kiseonika i azota koje ostaju u tečnosti i nakon prestanka dejstva plazme (Oehmigen et al., 2010). Do naglog pada pH vrednosti dolazi već u prvim minutima tretmana, a zatim se opadanje usporava. U oba tretirana uzorka primećeno je prisustvo reaktivnih vrsta - nitrata i vodonik peroksida.

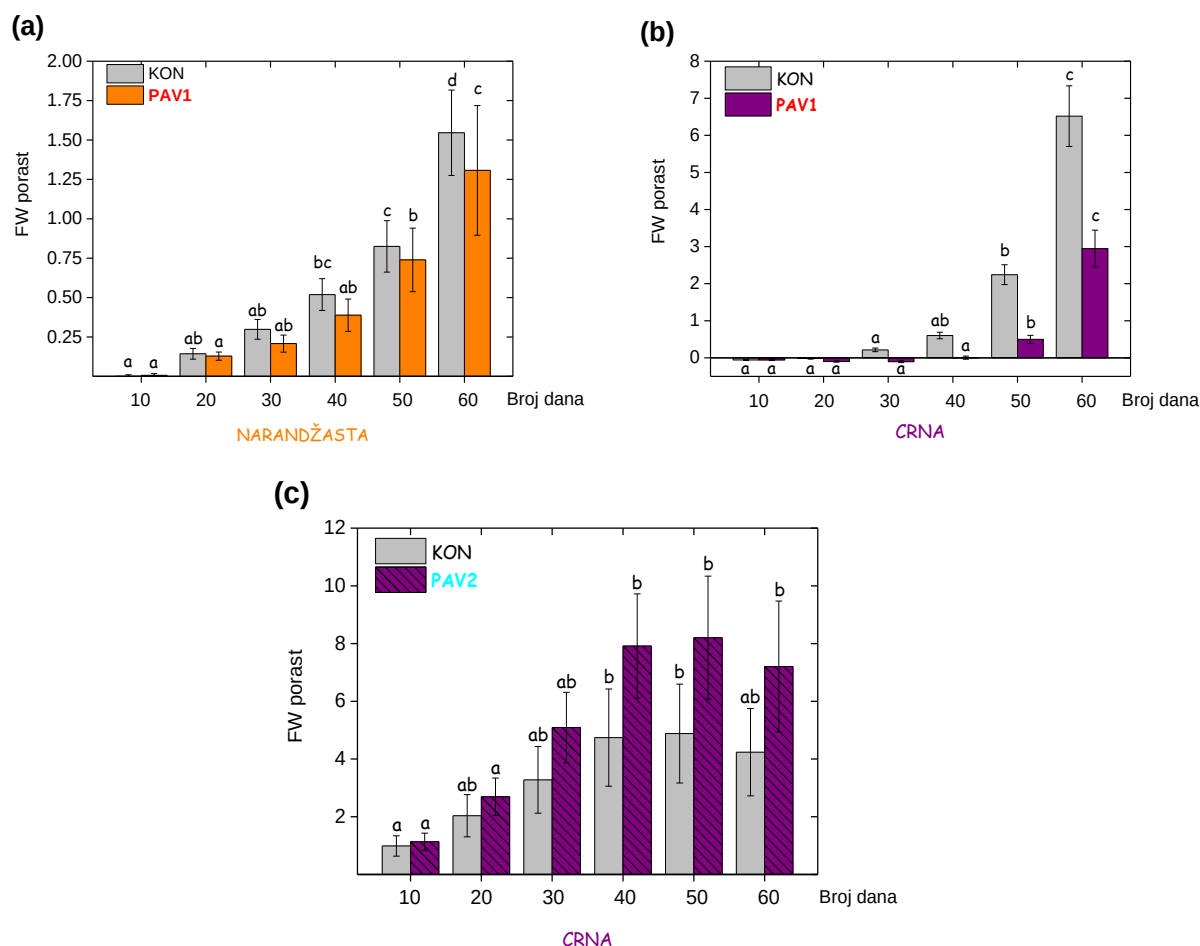
Pokazano je da vreme trajanja tretmana utiče na količinu reaktivnih vrsta koje će se deponovati u vodi. Osim merenja NO₃⁻ i H₂O₂, merena je i koncentracija NO₂⁻, ali su rezultati pokazali da nema nitrita u uzorcima nakon tretmana. U kiseloj sredini (pH < 3,5) nitriti su nestabilno jedinjenje koje prelazi u nitrata, a u reakciji sa H₂O₂, dovode do formiranja peroksinitrita (peroksiazotaste kiseline), što objašnjava nepostojanje nitrita u uzorcima tretirane destilovane vode (Tarabová et al., 2018).

PROMENE U MORFOLOGIJI I FIZIOLOGIJI KALUSA ŠARGAREPE

Efekat PAV na rast kalusa

Sveža masa (eng. fresh weight, FW) kalusa šargarepe koji su 2h tretirani PAV uzorkom, merena je na svakih deset dana u naredna dva meseca. Na slici 4.2 prikazana je izmerena sveža masa za (a) kalusa narandžaste šargarepe tretiranih uzorkom PAV1, (b) kalusa crne šargarepe tretiranih sa PAV1 i (c) kalusa crne šargarepe tretiranih uzorkom PAV2. Kontrolni uzorak bila je destilovana voda. Kod obe vrste kalusa koji su tretirani uzorkom PAV1 pokazan je sporiji porast ili čak smanjenje sveže mase sa vremenom u odnosu na kontrolni uzorak.

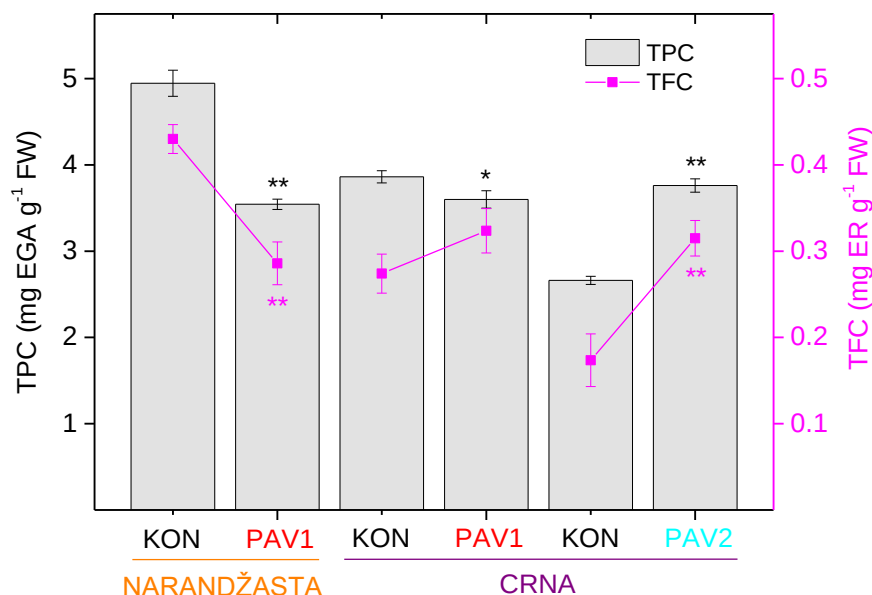
Sa druge strane, kod kalusa tretiranih uzorkom PAV2, primećen je značajan porast sveže mase u odnosu na kontrolni uzorak. Razlika u promeni sveže mase kod uzoraka tretiranih različitim PAV, može biti povezana sa različitom koncentracijom reaktivnih vrsta u ta dva uzorka. Pretpostavlja se da aktivne vrste azota iz plazme koje dolaze u kontakt sa kalusima mogu u njima da izazovu isti efekat kao i konvencionalna đubriva na bazi azota (Rathore & Nema, 2024; Robinson & Stapelmann, 2024).



Slika 4.2: Stopa rasta sveže mase kalusa šargarepe tretiranih: (a) i (b) uzorkom PAV1 i (c) uzorkom PAV2 u poređenju sa merenjima iz kontrolnog uzorka (KON). Prikazane su srednje vrednosti i standardna greška merenja za tri nezavisna eksperimenta. Podaci su podvrgnuti jednosmernoj analizi varijanse (ANOVA). Vrednosti sa istim slovima pripadaju statistički homogenoj grupi, prema Fisher-ovom LSD testu ($p < 0.05$).

Sadržaj fenola i flavonoida

Promene u sadržaju specijalizovanih metabolita - ukupnih fenola (TPC) i flavonoida (TFC) izazvane različitim PAV uzorcima prikazane su na slici 4.3. Kontrolni uzorak bila je destilovana voda. Tretman sa PAV1 je prouzrokovao pad TPC kod obe sorte šargarepe, dok je PAV2 doveo do suprotnog efekta. Sadržaj TFC kod narandžaste sorte tretirane sa PAV1 je opao u odnosu na kontrolu, dok je kod crne sorte šargarepe primećen rast TFC u odnosu na kontrolu, bez obzira na vrstu korišćenog PAV uzorka.



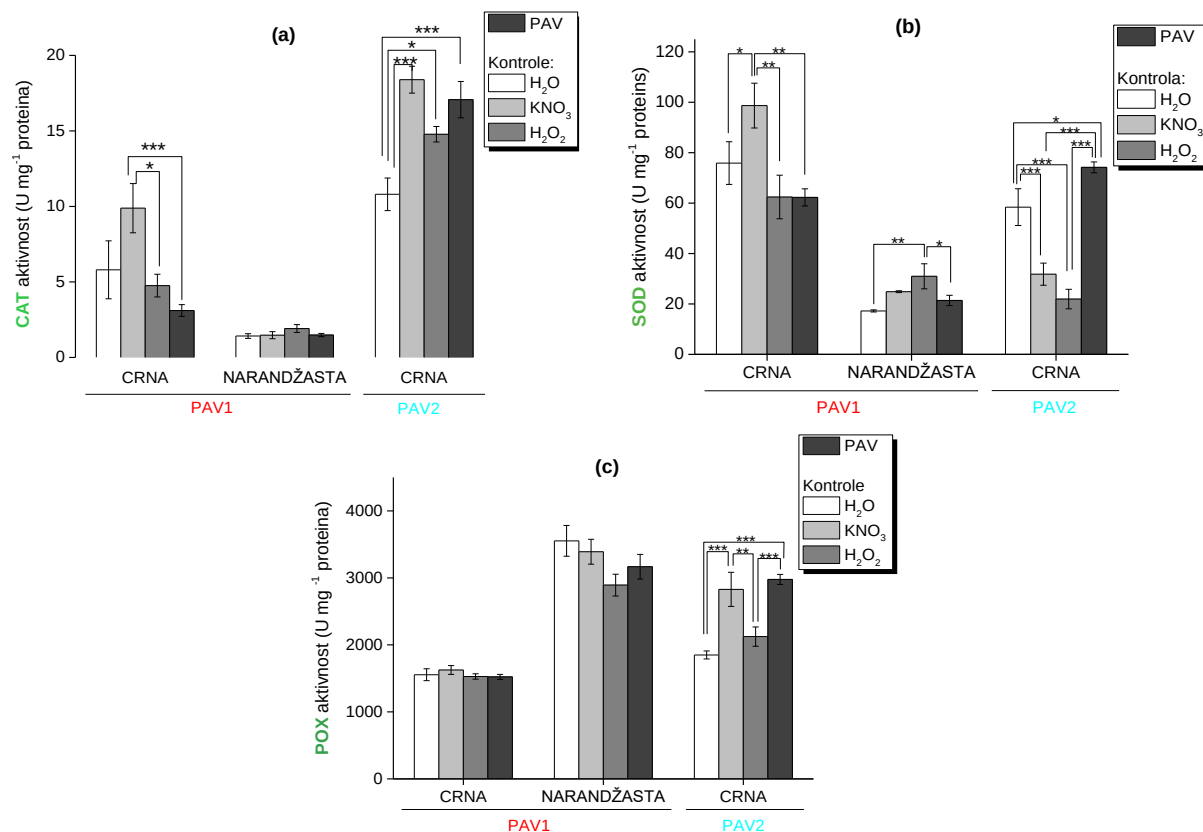
Slika 4.3: Promene u ukupnom sadržaju specijalizovanih metabolita u kalusima šargarepe izazvane PAV tretmanima. Rezultati su izraženi kao miligram ekvivalenta galne kiseline po gramu sveže mase ekstrakta (mg EGA g⁻¹ FW), odnosno kao miligram ekvivalenta rutina po gramu sveže mase ekstrakta (mg ER g⁻¹ FW). Podaci predstavljaju srednje vrednosti i standardne greške merenja za tri ponovljena eksperimenta. Simbol zvezdice predstavlja statističku značajnost u poređenju sa kontrolnim vrednostima (*p<0.05 i **p<0.01).

Aktivnost antioksidativnih enzima

Interakcija plazme sa tečnostima dovodi do transporta reaktivnih vrsta kiseonika i azota u tečnost i formiranja sekundarnih reaktivnih vrsta. U vodi, glavne reaktivne vrste nastale dejstvom plazme su hidroksilni radikali, ozon, vodonik peroksid, nitriti, nitrati, peroksinitriti i peroksinitrati. Ove vrste su biološki relevantni molekuli, koji imaju važnu ulogu u metaboličkim procesima u biljnim ćelijama. Tokom evolucije biljke su razvile brojne mehanizme da se zaštite od oštećenja uzrokovanim ovim vrstama, uključujući upotrebu antioksidativnih enzima (npr. katalaze-CAT, peroksidaze-POX i/ili superoksid dismutaze-SOD) koji su direktno uključeni u zaštitu biljaka od štetnog dejstva slobodnih radikala i oksidativnog stresa. U kalusima šargarepe CAT i SOD imaju primarnu ulogu u antioksidativnoj odbrani protiv reaktivnih kiseoničnih vrsta koje nastaju kao rezultat tretmana biljaka plazmom.

Rezultati tretmana kalusa uzorkom PAV1 su pokazali da je aktivnost enzima CAT (videti Sliku 4.4a) smanjena (crna sorta) ili je na istim nivou (narandžasta sorta) kao kod kontrolnih uzoraka. Gotovo isti uticaj PAV1 je primećen i kod enzima SOD (Slika 4.4b), pri čemu je ukupna aktivnost enzima SOD za red veličine veća u odnosu na aktivnost CAT. PAV2 uzorak je delovao stimulatивно na rast aktivnosti CAT kod crne sorte (4.4a) u odnosu na kontrolni ozorak H₂O, kao i na rast aktivnosti SOD (4.4b) u odnosu na sve kontrolne uzorke.

S druge strane, POX pokazuje najveću ukupnu aktivnost kod oba varijeteta (crni i narandžasti) šargarepe, bez obzira na tretman PAV (Slika 4.4c). Značajan uticaj na porast aktivnosti POX u odnosu na kontrolne uzorke H₂O i H₂O₂ je primećen kod tretmana sa PAV2.



Slika 4.4: Aktivnost antioksidativnih enzima u kalusima šargarepe tretiranim PAV-om. Ukupne aktivnosti katalaze (a), superoksid dismutaze (b) i peroksidaze (c) su izmerene spektrofotometrijski. Prikazani rezultati su predstavljeni kao srednja vrednost $\pm$ standardna greška za tri nezavisna merenja. Podaci su podvrgnuti jednosmernoj analizi varijanse (ANOVA), korišćenjem Fisher-ovog LSD testa (* $p < 0.05$, ** $p < 0.01$ i *** $p < 0.001$).

ZAKLJUČAK

Urađeni su tretmani destilovane vode sa ciljem dobijanja dva različita PAV uzorka (PAV1 i PAV2), sa različitim fizičko-hemijskim osobinama i sadržajem dugoživećih reaktivnih vrsta kiseonika, kako bi se utvrdio njihov uticaj na kaluse šargarepe.

Tretman PAV-om izazvao je značajne fiziološke i metaboličke promene u kalusima dve sorte šargarepe (crne i narandžaste). Uzorak PAV1 je uticao pozitivno na rast sveže mase narandžastih kalusa, koji je gotovo isti kao kod kontrole - H_2O . Sa druge strane, kod crne sorte je pokazano inhibitorsko dejstvo PAV1, pa je rast kalusa primećen tek nakon 50 dana od početka merenja i bio je manji u poređenju sa kontrolnim uzorkom. Rezultati dobijeni sa uzorkom PAV1 su pokazali da na dinamiku rasta kalusa utiče sama sorta biljke. Konkretno, tretman sa PAV2 (sa većim sadržajem NO_3^- i H_2O_2) pokazao je da je moguće stimulirati rast kalusa crne šargarepe – razlika u stopama rasta između PAV2 i kontrole bila je izražena nakon 20 dana i stabilisala se nakon 40 dana. Time je pokazano da se promenom karakteristike PAV-a može preokrenuti inhibicioni efekat i postaći rast biljnog tkiva.

Oksidativni stres kod kalusa šargarepe tretiranih PAV-om rezultirao je povećanjem aktivnosti antioksidativnih enzima CAT i SOD u periodima kada to nije primećeno kod kontrolnih uzorka, a koji se uz POX smatraju prvim linijama odbrane kalusa šargarepe od reaktivnih kiseoničnih vrsta proizvedenih tokom tretmana vode plazmom. Profil generisanja specijalizovanih metabolita (fenola i flavanoida) tretiranih kalusa razlikuje se između sorti i izmenjen je nakon tretmana plazmom. Ove promene se odnose na ukupan sadržaj dominantnih fenolnih jedinjenja u kalusima šargarepe.

4.1.2 ISPITIVANJE TOKSIČNOSTI PAV NA BILJCI SOČIVICA (*LEMNA MINOR*)

UVOD

U okviru ovog dela disertacije, urađena su ispitivanja toksičnosti PAV-a (Adhikari et al., 2019; Thirumdas et al., 2018) i uticaja na biljku sočivicu (lat. *Lemna Minor*), u saradnji sa kolegom dr Arianom Morinom, ekologom, sa kojim je urađeno planiranje biološkog dela eksperimenta kao i primena svih korišćenih bioloških protokola.

Zbog svoje rasprostranjenosti širom sveta, *Lemna minor* se našla kao ključna vrsta u različitim oblastima biologije i ekologije, kao dodatak ishrani ljudi i životinja (Appenroth et al., 2015; Khan et al., 2014; Sosa et al., 2025; Xu et al., 2022) gde utiče na smanjenje troškova proizvodnje i obezbeđuje kvalitet proizvoda, kao i u proizvodnji biogasa (Kaur et al., 2019; Soñta et al., 2019; Vyas et al., 2025). U pogledu ogromnih problema koje prouzrokuju različiti zagađivači vode, *Lemna minor* se pokazala kao skoro idealan modelni sistem za njihovo praćenje, rešavanje problema toksičnosti i fitoremedijaciju (Ahmed et al., 2025; Bokhari et al., 2016; Sasmaz et al., 2015; Sharma, 2017; Singh, 2012).

Sočivica je dobro proučena slatkovodna plutajuća vrsta makrofita koja se obično nalazi u obliku debelog pokrivača u sporim vodenim staništima bogatim hranljivim materijama (Ekperusi et al., 2019). Sama biljka se sastoji od dva karakteristična dela, nekoliko listova koji čine jedan složeni list (eng. frond) i jednog korena. Ova biljka se razmnožava vegetativno, što je čini skoro savršenom vrstom za laboratorijske eksperimente, jer može brzo da raste u medijumima bogatim hranljivim materijama, a uz odgovarajuću negu kolonija sočivica se može održavati gotovo zauvek (Niklas & Kutschera, 2014).

EKSPERIMENTALNA POSTAVKA I METODI

Proizvodnja i merenje fizičko-hemijskih parametara PAV

U eksperimentu je kao radni gas korišćen argon, a protok gasa bio je 1 slm. Vertikalno ispod napajane elektrode postavljen je uzorak sa vodom. Napon primenjen na napajanje elektrode, struja kroz uzemljenu granu kola i snaga deponovana u tretirani uzorak bile su $V_{RMS} = 1,2$ kV, $I_{RMS\_G} = 10,5$ mA i $P_{MEAN\_G} = 10$ W, respektivno. Površina vode bila je na udaljenosti $d = 10$ mm od vrha napajane elektrode. Plazmom je tretirano 15 ml vode iz gradskog vodovoda (u nastavku: voda sa česme, Voda Č), vreme tretmana bilo je 10 min.

Fizičko-hemijski parametri vode – pH vrednost, električna provodljivost i zapremina su mereni pre i posle tretmana. Nakon tretmana je merena i koncentracija reaktivnih vrsta – nitrita, nitrata i vodonik-peroksida. Metode korišćene za dijagnostiku tečnih uzoraka detaljno su opisane u poglavlju 2.2. Uzorci PAV su nakon tretmana držani u tečnom azotu do postavke eksperimenta sa biljkama. Pred korišćenje uzorci su odleživani, a u trenutka postavljanja eksperimenta sa biljnim uzorcima voda je bila na sobnoj temperaturi.

U delu eksperimenta u kome je ispitivan uticaj PAV na biljne uzorke, korišćene su dve različite matrice za pravljenje različitih razblaženja PAV – voda sa česme i rečna voda. Rečna voda je uzorkovana iz Dunava (Voda D) sa područja u Beogradu, Zemun, na dan postavke eksperimenta. U ove dve matrice dodavana je tretirana voda sa česme - PAV i napravljena su četiri razblažena rastvora sa udelom PAV u rastvoru od 10 %, 30 %, 60 % i 100 %. Kontrolni uzorci bili su Voda Č i Voda D. U tabeli 4.3 navedeni su svi korišćeni vodeni uzorci, odgovarajuće zapremine PAV i rastvarača, kao i skraćenice koje će biti korišćene za oznake uzoraka u daljem tekstu.

Tabela 4.3: Tečni uzorci korišćeni u eksperimentu sa biljkama.

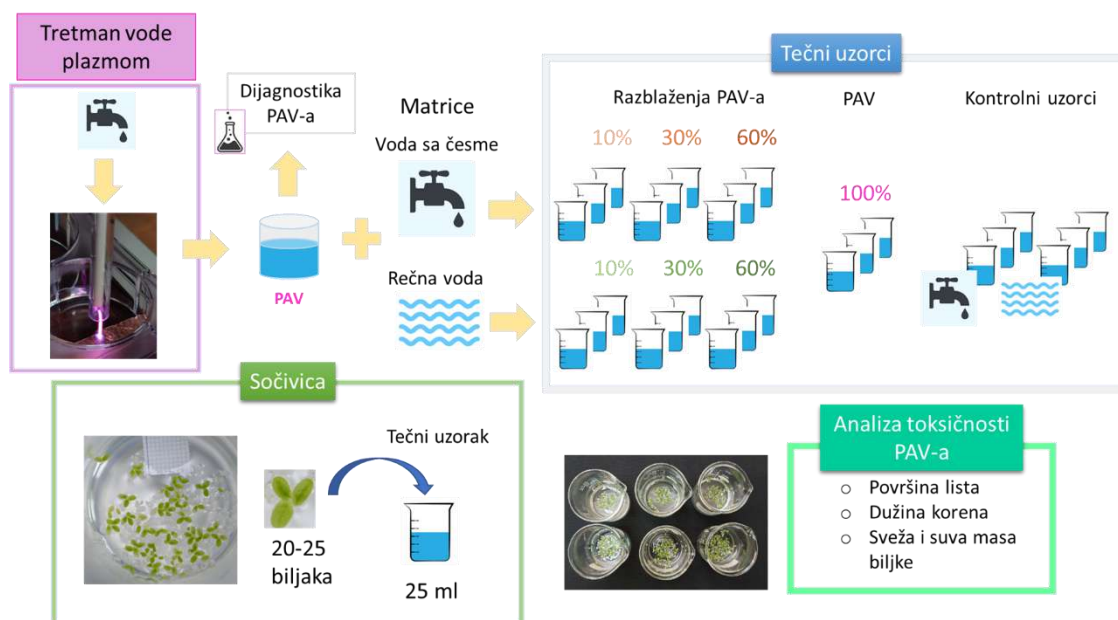
Tečni uzorci			
Skraćenica	Opis	Zapremina (ml)	
		PAV	Voda
Voda Č	Voda sa česme	0,0	25,0
10% PAV	10% PAV rastvor u vodi sa česme	2,5	22,5
30% PAV	30% PAV rastvor u vodi sa česme	7,5	17,5
60% PAV	60% PAV rastvor u vodi sa česme	15,0	10,0
100% PAV	PAV 10 min tretirana voda sa česme	25,0	0,0
Voda D	Voda uzorkovana iz Dunava* (rečna voda)	0,0	25,0
10%P u D	10% PAV rastvor u rečnoj vodi	2,5	22,5
30%P u D	30% PAV rastvor u rečnoj vodi	7,5	17,5
60%P u D	60% PAV rastvor u rečnoj vodi	15,0	10,0

*Voda uzorkovana iz Dunava, na lokaciji u Beogradu, Zemun 2023. godine.

Biološki uzorci

Pre početka eksperimenta, biljke su uzgajane u staklenoj posudi sa vodom sa česme u koju je redovno dodavana prehrana – komercijalni proizvod *Easy-Life Profito*, prema uputstvu proizvođača (100 µl hrane na 1 l vode). Pre eksperimenta posuda sa biljkama se nalazila u klima-komori u kojoj je podešen ciklus dan-noć 12 h-12 h (osvetljenje u komori je bilo uključeno od 6 h do 18 h). Temperatura je bila 20-21°C, a vlažnost vazduha 80 % ± 5 %.

Na slici 4.5 ilustrativno su prikazani delovi eksperimenta – proizvodnja PAV-a, priprema tečnih uzoraka, tretman i analiza biljnih uzoraka. Vodeni rastvori (videti Tabelu 4.3) ukupne zapremine 25 ml sipani su u laboratorijske čaše (ukupna zapremina čaše bila je 50 ml). Biljni uzorci - 20-25 pojedinačnih biljaka su stavljeni u čaše sa vodom, u koje je dodato i po 2,5 µl prehrane. Čaše su prekrivene aluminijumskom folijom i stavljene u klima komoru. Svaki uzorak postavljen je u triplicatu (po 3 čaše sa biljkama za svaki tečni uzorak). Uzorci su stajali u komori 17 dana, pod istim uslovima u kojima su biljke bile pre eksperimenta.



Slika 4.5: Ilustrativni prikaz eksperimentalne procedure proizvodnje PAV-a, pravljenja razblaženih rastvora PAV sa dva tipa vode (Voda Č i Voda D) i postavljanja biljaka u rastvore.

Određivanje površine lista i dužine korena

Nakon 17 dana, fotografisane su biljke sa ciljem da se izmere površine listova, odredi sadržaj hlorofila u biljkama i izmeri dužina korenova. U te svrhe korišćen je fotoaparat *Nikon D200* sa objektivom *Micro-NIKKOR 105mm f/2.8*. Slike su obrađivane u softverskom paketu *ImageJ 1.54d*. Svaka čaša sa uzorkom je slikana iz dva ugla. Slikanje površine listova urađeno je odgore, pod uglom od 90° u odnosu na površinu vode. Korenovi su fotografisani kroz staklo čaše, pod uglom od 90° u odnosu na vertikalnu osu čaše. Dobijene su srednje vrednosti površina listova i dužina korena i izračunate su standardne greške merenja, za tri nezavisna merenja.

Merenje sveže mase uzoraka

Sveža masa uzoraka merena je odmah nakon što su biljke izvađene iz tečnosti. Prvo su isečeni listići aluminijumske folije, kvadrati stranice 5-6 cm, koji su numerisani i izmereni svaki pojedinačno na analitičkoj vagi (*Gramma Libero*, opseg merenja od 0,0001 g do 100 g). Zatim su na jedan listić folije stavljene biljke iz dve (od ukupno tri) čaše, za svaki tip tečnog uzorak ponaosob. Izmerena je zbirna masa jednog listića folije i biljaka koje su na njemu. Iz dobijenih podataka je preračunata sveža masa biljaka (eng. fresh weight, FW). Ove biljke su u nastavku eksperimenta iskorišćene za merenje količine hlorofila u biljkama tretiranim PAV-om.

Statistička metoda

Za statističku obradu podataka korišćena je Studentova t-test metoda, sa nivoom značajnosti postavljenim na $p < 0,05$. Analiza je sprovedena u programu *OriginLab*.

Merenje hlorofila u tretiranim uzorcima²

Za merenje hlorofila u biljnim uzorcima korišćena je metoda adaptirana iz doktorske teze (Vuleta, 2013). Biljni uzorci (iz dve od tri čaše, za svaki tip tečnog uzorka ponaosob), stavljeni su u ependorf od 1,5 ml. Prema izmerenoj svežoj masi $FW(g)$, u ependorf je dodavana odgovarajuća količina - $V_{DMSO}(ml)$, dimetil sulfoksidnog rastvora (hemijska formula $(CH_3)_2SO$, u daljem tekstu DMSO), u sledećoj razmeri:

$$FW (g) : V_{DMSO} (ml) = 1:30 \quad (4.1),$$

$$V_{DMSO} (ml) = FW(g) \cdot 30 \quad (4.2).$$

Za ove potrebe korišćen je rastvor *Dimethylsulfoxid* C_2H_6OS , Merck (99.5%, dried, max. 0.03% water, Mol.-Gew. 78.13, 1l=1.1kg).

Nakon sipanja rastvora i biljaka u ependorfe, oni su vorteksovani (VELP SCIENTIFICA ZX3 Napredni vorteks mešać) po 10 s. Tako dobijeni homogenati bili su inkubirani na 60°C u ultrazvučnoj kadi, u trajanju od 6 h. Posle toga su uzrci prebačeni u plastične tube od 15 ml i stavljeni na centrifugiranje (Cole Parmer #17250-10 CENTRIFUGE) u trajanju od 30 min. Maksimalni broj obrtaja centrifuge bio je 3400 X g, ali stvarni broj obrtaja zavisi od mase uzoraka koji se nalaze u centrifugi i ne može se tačno odrediti. Na kraju je izmeren apsorpcioni spektar supernatanta (tečni sadržaj koji se izdvoji nakon centrifugiranja, iznad taloga koji ostane na dnu tube) na spektrofotometru u opsegu talasnih dužina od 200-900 nm. Blank uzorak bio je DMSO rastvor. Praćeni su glavni (najizraženiji) apsorpcioni pikovi na 649 nm i 665 nm, čiji intenziteti su (linearno) srazmerni sadržaju hlorofila u biljci (Caesar et al., 2018; Egan & Schiff, 1974; Minocha et al., 2009).

Sadržaj hlorofila a i hlorofila b izračunat je prema sledećim izrazima (Wellburn, 1994):

$$C_a = 12,19A_{665} - 3,45A_{649} \quad (4.3),$$

$$C_b = 21,99A_{649} - 5,32A_{665} \quad (4.4).$$

REZULTATI I DISKUSIJA

Osobine dobijenog PAV-a

Kako bi se ispitala efektivnost tretmana vode plazmom, neposredno nakon tretmana izmerena je pH vrednost tretirane tečnosti, električna provodljivost, zapremina preostalog uzorka i određene su koncentracije reaktivnih vrsta. Karakteristike Vode Č i PAV uzorka dobijenog tretiranjem Vode Č, prikazane su u Tabeli 4.4.

Među brojnim azotnim i kiseoničnim vrstama koje se formiraju u ovoj zoni, ranijim snimanjem optickog emisionog spektra u sistemu plazma mlaza (videti potpoglavlje 3.2), detektovano je prisustvo dva značajna radikala - hidroksila OH i azot monoksida NO, koji učestvuju u reakcijama formiranja dugoživećih vrsta u vodi (videti potpoglavlje 1.3.2).

Tabela 4.4: Fizičko-hemijske osobine Vode Č i PAV-a: V - zapremina uzorka, σ – el. provodljivost, c - koncentracija.

Uzorak	V [ml]	pH	σ [μ S/cm]	c(H ₂ O ₂) [mg/l]	c(NO ₃ ⁻) [mg/l]	c(NO ₂ ⁻) [mg/l]
Voda Č	15,0	7,8±0,1	583±30	0	0	0
PAV	13,5±0,1	8,1±0,2	457±3	36±1	39±1	29±1

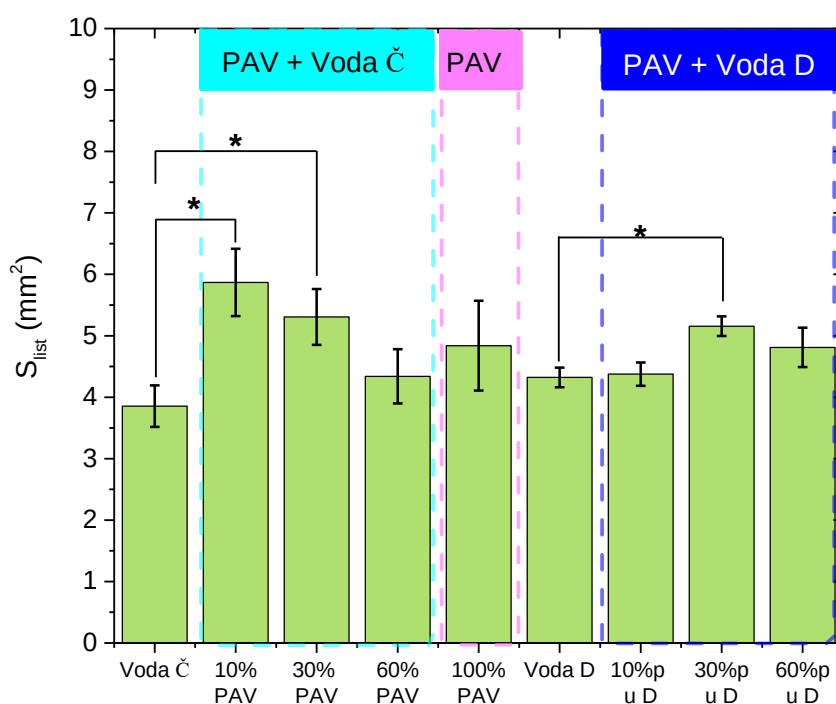
pH vrednost Vode Č ostaje nepromenjena nakon tretmana, što se može objasniti njenim puferskim osobinama usled prisustva različitih primesa. Prvenstveno su to karbonati i bikarbonati, koji redukuju stvaranje kiselina i time onemogućuju zakišeljavanje sredine (Judée & Dufour, 2019; Schmidt et al., 2019; Simon et al., 2022; Wartel et al., 2021).

Rezultati merenja električne provodljivosti pokazuju da je došlo do smanjenja od oko 20%, sa vrednosti 583 μ S/cm na 457 μ S/cm. Pad vrednosti el. provodljivosti za kratke tretmane (do 10 min), pokazan je u slučaju tretmana vode DBD izvorom (Judée et al., 2018), nakon čega je usledeo rast provodljivosti sa vremenom trajanja tretmana. Varijacija u električnoj provodljivosti tokom tretmana je objašnjena procesima povećavanja i smanjivanja koncentracija dugoživećih jonskih vrsta, koje utiču na ukupnu provodljivost vodenog uzorka. Među njima je najznačajnije povećanje koncentracije nitrata (pogledati poglavlje 1.3.2) i smanjenje koncentracija (bi)karbonata u vodi, usled lokalnog zagrevanja vode u kontaktu sa plazmom, pri čemu dolazi do stvaranja gasnog CO₂. Sličan trend promene el. provodnosti sa vremenom tretmana dobijen je i u slučaju tretmana vode sa pražnjenjem kliznog luka (eng. gliding arc discharge) (Chiappim et al., 2021).

Uticaj PAV-a na biljke

Rezultate eksperimenata sa bojkama podelili smo u dve grupe. Prva grupa odnosi se na morfološke promene kod sočivice (ukupna površina lista i dužina korena), dok druga grupa obuhvata fiziološko praćenje sadržaja hlorofila (hlorofil a, hlorofil b i ukupni hlorofil) pri različitim kombinacijama PAV, Voda Č i Voda D.

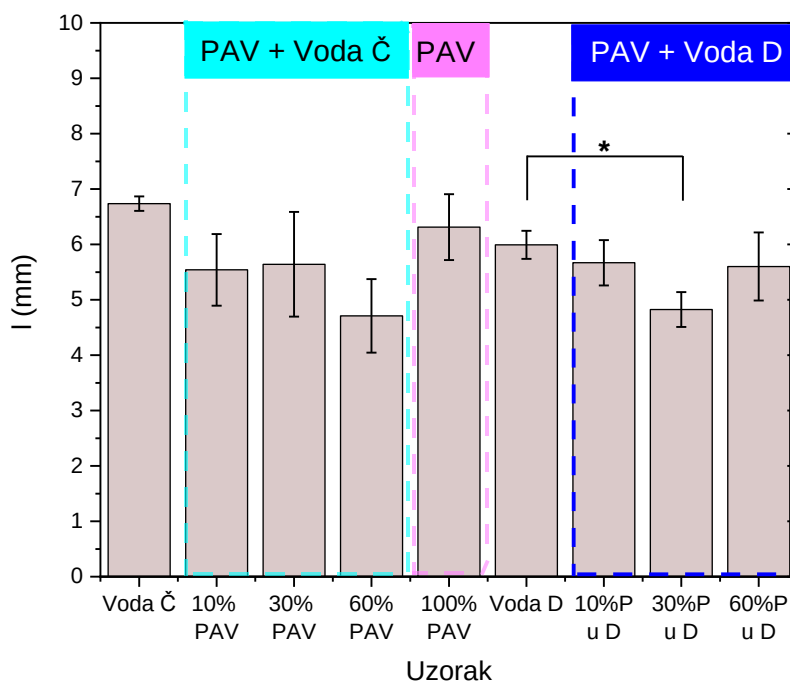
Slika 4.6 prikazuje srednju površinu listova jedne biljke S_{list} (mm^2), za različite tečne uzorke u kojima su biljke uzgajane. Primećen je porast u površini lisova, kod većine uzoraka koji sadrže PAV u odnosu na odgovarajuće kontrolne uzorke – Vodu Č i Vodu D. Kada je kao matrica za razblaživanje korišćena Voda Č, primećeno je da površina lista dostiže maksimum za razblaženje 10% PAV, gde je izmeren povećanje površine od 50% u odnosu na kontrolni uzorak Voda Č. Veći udeo PAV u smeši ($\geq 60\%$) je usporio rast. Kod druge matrice – Voda D, tek udeo PAV od 30% dovodio je do porasta površine lista, dok je sa većim udelom PAV došlo do usporavanja povećanja površine.



Slika 4.6: Izmerena srednja površina zelenih listova biljaka S_{list} nakon 17 dana uzgajanja u tečnim uzorcima sa različitim razblaženjima PAV (10%-100%) i kontrolnim uzorcima, za dve korišćene matrice Voda Č i Voda D. Simbol zvezdice predstavlja statističku značajnost u poređenju sa kontrolnim vrednostima (* $p < 0.05$).

Dobijeni rezultati odgovaraju različitim eksperimentima na biljkama u kojima je korišćen PAV, kao što je povećana površina listova i prečnik listova kod *Arabidopsis thaliana* (Bafail et al., 2018). Pozitivni efekti PAV su takođe primećeni u eksperimentima sa *Solanum lycopersicum* gde su maksimalna dužina stabljike i folijarna pokrivenost pronađeni kod biljaka tretiranih PAV-om (Aceto et al., 2024). Pored toga, dužina biljke *Zea mays* i *Hordeum vulgare* takođe je povećana nakon tretmana sa PAV (Ndiffo Yemeli et al., 2021). Svi ovi rezultati su u skladu sa našim merenjima porasta površine listova sočivice.

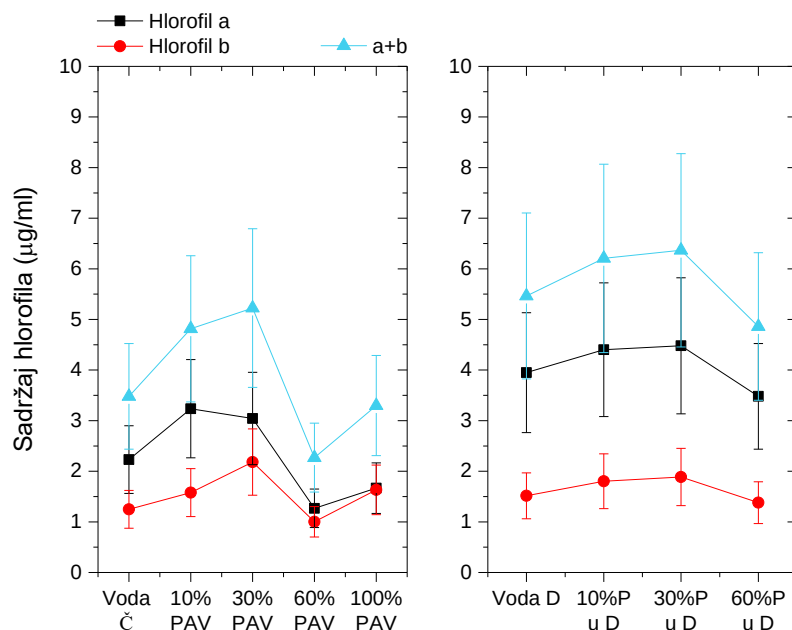
Na slici 4.7 prikazane su srednje dužine korena l (mm) biljaka, koje su za različite matrice pokazivale različite obrasce. U slučaju rastvora u Vodi Č, primećen je pad dužine korena sa povećem procenta PAVu rastvori, u odnosu na kontrolni uzorak, s tim da je kod razblaženja 30% PAV došlo do blagog povećanja dužine u odnosu na druga dva razblaženja. U slučaju kada je matrica bila Voda D, primećen je pad dužine u odnosu na uzorak Voda Č i 100%PAV. Pored toga, primećen je pad dužine korena kod uzorka 30%P u D u odnosu na kontrolni uzorak, dok kod uzoraka 10%P u D i 60%P u D nije bilo značajne promene.



Slika 4.7: Dužina korena biljaka izmerena nakon tretmana 17 dana tretmana PAV-om, za različita razblaženja PAV (10 - 60%) i kontrolne uzorke Voda Č i Voda D. Simbol zvezdice predstavlja statisticku znacajnost u poredenju sa kontrolnim vrednostima (* $p < 0.05$).

Dobijeni rezultati o dužini korena sočivice tretirane razblaženjima PAV u Vodi Č, podudaraju se sa istraživanjem efekata nanočestica cink oksida na istu biljku, pri čemu se izduženje korena povećava sa koncentracijom nanočestica cink oksida od 0,3 do 1 mg/l i smanjuje za viših koncentracija ove nanočestice (Chen et al., 2018). Studija koju su sprovedli Xue i saradnici (2018) istražujući efekte kadmijuma na sočivicu istakla je da se izduženje korena i broj listova smanjuju nakon povećanja rastvora kadmijuma u medijumu. Druga studija koja je istraživala efekte fluorida na sočivicu (Kamiński et al., 2024) primetila je postepeno smanjenje površine listova sa povećanjem fluorida (2; 4; 6 mg/l). Slični efekti na površinu listova primećeni su nakon izlaganja sočivice nanočesticama cink selenida, pri čemu se veličina listova postepeno smanjivala sa povećanjem koncentracije ovih nanočestica od 1 do 80 mg/l, a najveća površina listova primećena je tretiranjem biljke koncentracijom od 5 mg/l (Tarrahi et al., 2018).

Na slici 4.8 prikazane su izmerene vrednosti koncentracija hlorofila a, hlorofila b i ukupnog hlorofila ($C_{\text{ukupni}} = C_a + C_b$) u biljkama nakon tretmana PAV-om, dobijene korišćenjem izraza 4.3 i 4.4. Kod matrice Voda Č, ukupan sadržaj hlorofila je porastao kod uzoraka 10%PAV i 30%PAV, a zatim je usledilo smanjenje sa povećanjem koncentracije PAV. Sličan trend je primećen i kod matrice Voda D, iako su ovde ukupni nivoi hlorofila bili viši u poredenju sa rastvorima sa matricom Voda Č.



Slika 4.8: Koncentracija hlorofila a, hlorofila b i ukupnog (a+b) u biljkama tretiranim različitim tečnim uzorcima. Tečni uzorci su bili PAW i rastvori PAW (10 – 60 %) pripremljeni sa dve različite vodene matrice: Voda Č i Voda D. Kontrolni uzorci su bili ne tretirani uzorci Voda Č i Voda D. Procenjena je greška merenja od 30%.

Smanjenje sadržaja hlorofila za koncentracije PAV iznad 30% je mnogo izraženije nego smanjenje površine lista. Sadržaj hlorofila pruža tačniju indicaciju kvaliteta lišća i ukupnog stanja biljke nego sama merenja površine lista. Međutim, oba parametra ukazuju na to da niže koncentracije PAV u vodi ne samo da nisu toksične već podstiču poboljšanje biljaka, pri čemu azotne vrste iz PAV deluju kao đubrivo.

U studiji koju su sprovedli Chen i kolege (2018), zabeleženo je postepeno smanjenje sadržaja hlorofila sa povećanjem koncentracije nanočestica cink oksida primenjenih na sočivicu (0,03; 0,30; i 3,00 mg/l). Slični rezultati u vezi sa hlorofilom a, objavljeni su u studiji koja je istraživala efekte fluorida na sočivicu, gde je pokazano smanjenje koncentracije hlorofila sa povećanjem koncentracije fluora, sa odabranim koncentracijama (2,10; 4,19; i 4,90 mg/l) (Kamiński et al., 2024). U istraživanju koje se bavilo efektima nanočestica cink selenida na sočivicu, takođe je primećeno postepeno smanjenje sadržaja hlorofila sa povećanjem koncentracije ovih nanočestica (Tarrahi et al., 2018), a slični rezultati su pronađeni i prilikom istraživanja efekata herbicida flurohloridona na sočivicu (Zhou et al., 2020).

Sadržaj hlorofila se smatra indikatorom aktivnosti proteina Fotosistema II u biljkama koje su direktno ili indirektno izložene određenom stresu (Percival, 2005). Povećanje sadržaja hlorofila u ovom istraživanju može se objasniti na dva načina. Prvo, formiranjem hloroplasta u listovima u senci (eng. shade-type chloroplasts), koji imaju ultrastrukturne modifikacije kako bi primili više pigmenata, sa povećanjem sadržaja pigmenata u biljci izloženoj PAV-u. S druge strane, veći porast sadržaja hlorofila pri nižim koncentracijama PAV može biti povezan sa hormetskim odgovorima, gde biljke izložene uslovima niskog stresa povećavaju rast i aktivnost enzima. Takva hormeza je ranije primećena kod sočivice tretirane različitim koncentracijama različitih pesticida (Cedergreen et al., 2007).

ZAKLJUČAK

U ovom potpoglavlju, ispitana je toksičnost PAV-a na biljku sočivicu. PAV je dobijen tretmanom vode sa česme, čime je dobijen vodeni uzorak koji ima neutralnu pH vrednost, ali sadrži reaktivne vrste proizvedene pod dejstvom plazme. Pored PAV uzorka, za uzgoj biljke su korišćena još tri različita razblaženja (udeo PAV od 10%, 30% ili 60%), a kao matrice su korišćene dve različite vode – voda sa česme i rečna voda. Praćene su promene na biljkama – površina lista, dužina korena, masa i udeo hlorofila kako bi se proučio uticaj vodenih rastvora na morfološke i fiziološke osobine biljake.

Pokazano je da uzorci sa PAV-om dovode do povećanja površine lista u odnosu na kontrolne uzorke (Voda Č i Voda D), za obe grupe uzoraka. Međutim, dobijeno je da uzorci sa manjom količinom PAV u vodi dovode do većeg rasta lista nego uzorci sa većim procentom PAV-a, odnosno veći udeo PAV-a u vodi dovodi do usporavanja rasta lista. Sa druge strane, pokazano je da efekti PAV na druge merene parametre, zavise kako od procenta PAV u vodi tako i od korišćene matrice. Određivanje sadržaja hlorofila pokazalo je kod obe grupe uzoraka da dolazi do rasta sadržaja hlorofila (u odnosu na kontrolne uzorke) sa porastom udela PAV u vodi, do procenta PAV od 30%. Količine PAV veće od 30% deluju negativno na sadržaj hlorofila u biljkama. U pogledu dužine korena biljaka, negativan uticaj PAV-a se pokazao kod obe korišćene matrice.

Pristupi koji su ovde korišćeni za eksperimente toksičnosti bili su u skladu sa metodama koje su se ranije koristile za istraživanje različitih potencijalno toksičnih hemikalija. U tom smislu, ovo je omogućilo poređenje uticaja PAV sa studijama gde su efekti temeljno ispitani na biohemijskom nivou, a takođe i povezivanje sa mehanizmima indukovanim u biljkama. Naši rezultati testiranja toksičnosti pokazali su da, generalno, uticaj PAV na sočivicu može biti i pozitivan i negativan, u zavisnosti od koncentracije PAV, a takođe i od drugih nečistoća u vodi. Stoga, trebalo bi testirati širi opseg svojstava PAV kako bi se u potpunosti razumeli efekti reaktivnih vrsta iz PAV na fiziološke mehanizme biljaka.

4.2 TRETMANI ZAGAĐENE VODE PLAZMOM

Druga oblast primene u kojoj je iskorišćen potencijal hladne plazme bila je tehnologija prečišćavanja otpadnih voda, u kojoj je plazma upotrebljena kao NOP. Primarni zadatak bio je da se pokaže da je primena plazme efikasan i ekološki prihvatljiv metod za uklanjanje zagađivača koji se ne uklanjaju lako iz vode konvencionalnim tehnikama. Cilj je bio da se ispita efikasnost korišćenog sistema za tretmane vode zagađene organskim materijama, načini razgradnje i da li razgradnja dovodi do stvaranja drugih štetnih produkata raspada.

U prvom eksperimentu je ispitivana mogućnost korišćenja tehnologije plazme za prečišćavanje otpadnih voda nastalih pri poljoprivrednim aktivnostima (poglavlje 4.2.1) kako bi se generalno ispitala mogućnost ponovnog korišćenja prečišćene vode za primene u poljoprivredi. Plazmom je tretirana voda u kojoj je bila rastvorena jedna vrsta pesticida - malation ili acetamiprid. Analiza uzoraka urađena je merenjem fizičko-hemijskih osobina pre i nakon tretmana. U saradnji sa kolegionicama sa Tehnološko-metalurškog fakulteta Univerziteta u Beogradu, urađena je i analiza tretiranih uzoraka HPLC metodom.

U saradnji sa kolegionicama iz instituta ICRA u Ćirioni, Španija, ispitani su efekti tretmana na degradaciju organskih jedinjenja PFAS u vodi (poglavlje 4.2.2). Rezultati ovog istraživanja vezani za procese dekontaminacije predstavljeni su u radu (Topolovec et al. 2024) i u doktorskoj tezi (Topolovec, 2025) i ovde će biti prikazani u kontekstu kompletnog prikaza primene plazma mlaza.

4.2.1 EFEKTI TRETMANA NA DEGRADACIJU PESTICIDA RASTVORENIH U VODI

UVOD

Dejstvu plazme je izložena voda u kojoj je rastvorena organska supstanca – pesticid malation ili pesticid acetamiprid. Početne koncentracija vodenog rastvora bila je 500 ppm. Izvršeno je prilagođavanje parametara sistema tako da za datu električnu provodnost uzorka bude najefikasniji prenos snage od napajanja do sistema (videti poglavlje 3.1), za svaki korišćeni radni gas.

Nakon tretmana uzorci su analizirani metodom tečne hromatografije visokih performansi (HPLC-MS/MS) u saradnji sa kolegionicama sa Tehnološko-metalurškog fakulteta Univerziteta u Beogradu. Proučavano je kako vreme trajanja tretmana utiče na efikasnost razgradnje zagađivača. Pored toga, identifikovani su produkti raspada malationa i predložen je put razgradnje malationa. Dodatno su urađeni tretmani čiste (destilovane) vode kako bi se proučile promene fizičko-hemijskih osobina tečnosti u zavisnosti od sastava mete.

EKSPERIMENTALNA POSTAVKA I METODI

Tretmani čiste i zagađene vode

Urađena su dva tipa tretmana, gde su korišćeni He i Ar kao radni gasovi, a protoci su bili 2 slm i 1 slm, respektivno. Plazma mlaz napajan je VN signalom rms vrednosti $V_{rms}=1850$ V, dok je srednja snaga oslobođena u uzorku bila $P_{G\_NET}=1$ W, u slučaju pražnjenja u helijumu, a vrednosti za argon su iznosile $V_{rms}=1200$ V i $P_{G\_NET}=10$ W.

Tokom tretmana uzorci su bili postavljeni u jedan od otvora mikrotiter posude sa 6 otvora (*TC- Ploča sa 6 otvora, Srandard, F, Sarstedt, Nimbreh, Nemačka*). Početna zapremina uzorka bila je 4 ml. Rastojanje između napajane elektrode i površine uzorka bilo je $d=10$ mm. Urađeni su tretmani od 5 min, 10 min i 20 min.

Tretirana su dva tipa uzorka - destilovana voda i destilovana voda u kojoj je rastvorena jedna vrsta pesticida (kontaminirani uzorak). Korišćeni su sledeći komercijalni proizvodi pesticida: *Etiol tečni* (*Galenika -Fitofarmacija*) - aktivna supstanca malation, malation analitičke čistoće (*Sigma Aldrich 36143*), *Tonus* (*Galenika -Fitofarmacija*) - aktivna supstanca acetamiprid. Lista svih uzoraka i skraćenica koje će biti korišćene u nastavku ovog poglavlja, data je u tabli 4.5

Tabela 4.5: Lista korišćenih tečnih uzoraka i odgovarajućih skraćenica.

Tip uzorka	Zagađivač	Skraćenica	Vreme tretiranja [min]
Tretiran plazmom	nema	PAV5	5
	nema	PAV10	10
	nema	PAV20	20
Kontrola	nema	DV	0
Tretiran plazmom	Malation 500 ppm	MD5	5
	Malation 500 ppm	MD10	10
Kontrola	Malation 500 ppm	M500ppm	0
Tretiran plazmom	Acetamiprid 500 ppm	AD10	10
	Acetamiprid 500 ppm	AD20	20
Kontrola	Acetamiprid 500 ppm	A500ppm	0

Analiza fizičko-hemijskih osobina uzorka

Kako bi se ispitao uticaj tretmana plazmom na osobine meta različitog sastava, urađena su merenja zapremine i pH vrednosti uzoraka pre i nakon tretmana plazmom. Određena je i koncentracija dugoživećih reaktivnih vrsta (NO_3^- , NO_2^- i H_2O_2) korišćenjem semi-kvantitativnog metoda uz pomoć komercijalnih kolorimetrisjkih traka.

Za sve uzorke, merenja trakama su obavljena tri ili više puta na skupu uzoraka dobijenih nezavisnim tretmanima plazmom, za iste uslove tretmana – vrstu uzorka i vreme tretiranja. Pošto su merenja davala konzistentne rezultate unutar datog skupa, grešku merenja je određena kao preciznost metode za razlikovanje koncentracija. Za merenje H_2O_2 uzeta je procenjena greška merenja od 15%.

U cilju sistematskog praćenja razgradnje organofosfata, polazni rastvor malationa i uzorci tretirani plazmom su analizirani metodom HPLC, gde je kao maseni spektrometar (detektor) korišćen linearni jonski trap (LC-MS). Detalji korišćene metode mogu se naći u **Prilogu 2** ove disertacije.

Zbog osetljivosti instrumenata, za potrebe ovih merenja urađeni su tretmani vode zagađene malationom analitičke čistoće, a početna koncentracija zagađivača bila je 10 ppm. Da bi se dobili skupovi podataka relevantni za hemijsku kinetičku studiju, za ova merenja uvedeno je dodatno vreme tretmana od 20 minuta.

REZULTATI I DISKUSIJA

Merenje isparavanja tretiranih uzoraka

Usled dejstva plazme na tretirani tečni uzorak, tokom tretmana je dolazilo do njegovog isparavanja. Kako bi se ispitalo da li prisustvo zagađivača utiče na isparavanje, nakon svakog tretmana je merena zapremina uzorka (videti tabelu 4.6). Urađeni su tretmani čiste destilovane vode, kao i vode zagađene jednom vrstom pesticida. Kao radni gasovi korišćeni su He i Ar. U tabeli su prikazane srednje vrednosti i standardne greške merenja zapremina uzoraka nakon tretmana u *ml* i u procentima u odnosu na početnu zapreminu od 4ml. Svaki eksperiment je urađen u triplicatu.

Tabela 4.6: Isparavanje tečnih uzoraka tokom tretmana plazmom: *t* – vreme trajanja tretmana, *V_t* – srednja vrednost zapremine uzorka nakon tretmana, / - tretmani koji nisu urađeni. Početna zapremina svih uzoraka bila je 4 ml.

		Radni gas			
		He 2slm		Ar 1 slm	
Uzorak	t	V _t			
	min	ml	%	ml	%
PAV5	5	3,73±0,03	93	3,14±0,05	78
PAV10	10	3,51±0,01	88	2,35±0,04	59
PAV20	20	3,20±0,03	80	/	/
MD5	5	3,76±0,03	94	3,13±0,08	78
MD10	10	3,53±0,03	88	2,33±0,05	58
AD10	10	3,52±0,03	88	/	/
AD20	20	3,20±0,03	80	/	/

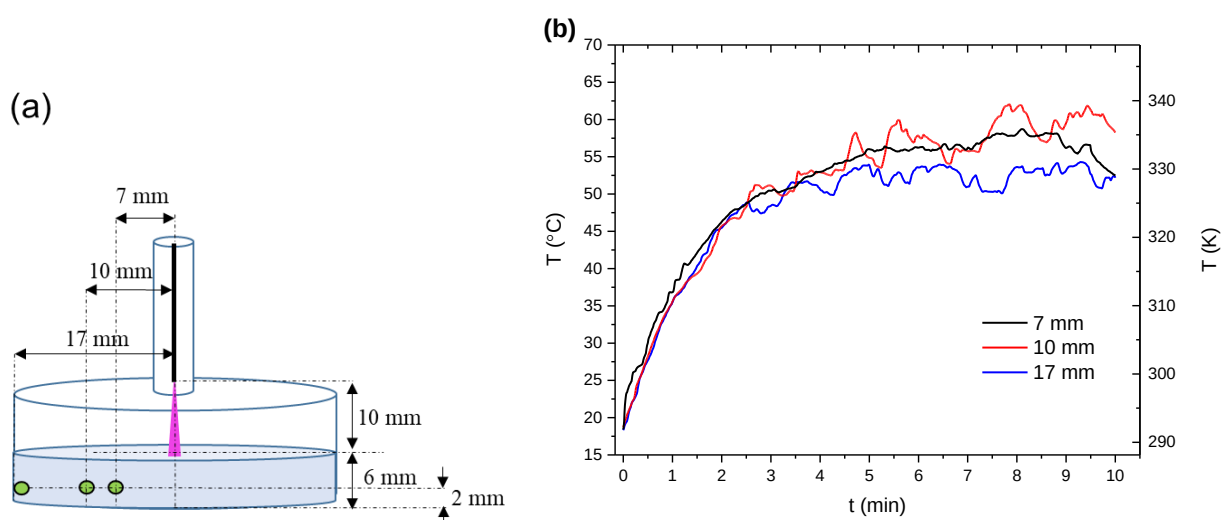
Kada je kao radni gas korišćen He, urađeni su tretmani u trajanju od 5, 10 i 20 minuta. Tretirani uzorci bili su DV, M500ppm i A500ppm. Rezultati pokazuju da bez obzira na prisustvo i vrstu zagađivača, isparavanje zavisi samo od vremena trajanja tretmana. Pokazalo se da zapremina uzorka eksponencijalno opada sa vremenom trajanja tretmana. Nakon 5 min tretmana ispari 6-7%, nakon 10 min 12% i nakon 20 min 20% početne zapremine uzorka.

U slučaju kada je kao radni gas korišćen argon, takođe je dobijeno da isparavanje vodenih uzoraka zavisi isključivo od vremena trajanja tretmana. Isparavanje uzorka je bilo veće za 15% i 30%, nakon 5 min i 10 min tretmana, respektivno, u odnosu na tretmane iste dužine kada je radni gas bio helijum. Tretmani od 20 min nisu urađeni kada je u sistemu korišćen argon, jer bi to dovelo do prekomernog isparavanja uzorka, što nije opravdano sa stanovišta primene ove tehnologije.

Kako bi se utvrdilo da li do isparavanja uzoraka dolazi usled termalnog zagrevanja, izvršena su merenja temperature tokom 10 min tretmana DV. Merenje je izvršeno na rastojanju 2 mm iznad dna posude, na tri različita rastojanja od vertikalne ose plazma mlaza: 7 mm, 10 mm i 17 mm (videti sliku 4.9a). Na slici 4.9b prikazana je zavisnost izmerene temperature od vremena trajanja tretmana, za tri različite pozicije u tečnom uzorku.

U sva tri slučaja, primećen je brz rast temperature sa početnih 18,5 °C na približno 50 °C tokom prva 3 minuta, praćem sporijim rastom u sledeća dva minuta. Nakon toga, temperatura se stabilisala i ostala gotovo konstantna do kraja tretmana. Primećen je temperaturni gradijent unutar zapremine uzorka, pri čemu su vrednosti opadale od ose plazma mlaza ka zidu posude i nakon stabilizacije su iznosile oko 56°C na rastojanjima 7 mm i 10 mm i oko 52 °C na 17 mm.

Prenos toplote sa gasa na tečnost odvija se pre svega putem toplotne provodnosti, što se manifestuje kroz brz porast temperature. Kako radni gas nije zasićen vodenom parom, ovaj proces je praćen i isparavanjem. Istovremeni prenos toplote i mase zasniva se na ravnoteži između energije koja se prenosi u tečnost i brzine prelaska vodene pare u gasnu fazu, pri čemu se temperatura vode prilagođava tako da uravnoteži brzine ovih procesa (Bruggeman et al., 2016). Nakon uspostavljanja ravnoteže, temperatura se stabilise, ne menjajući značajno svoju vrednost tokom preostalog vremena tretmana.



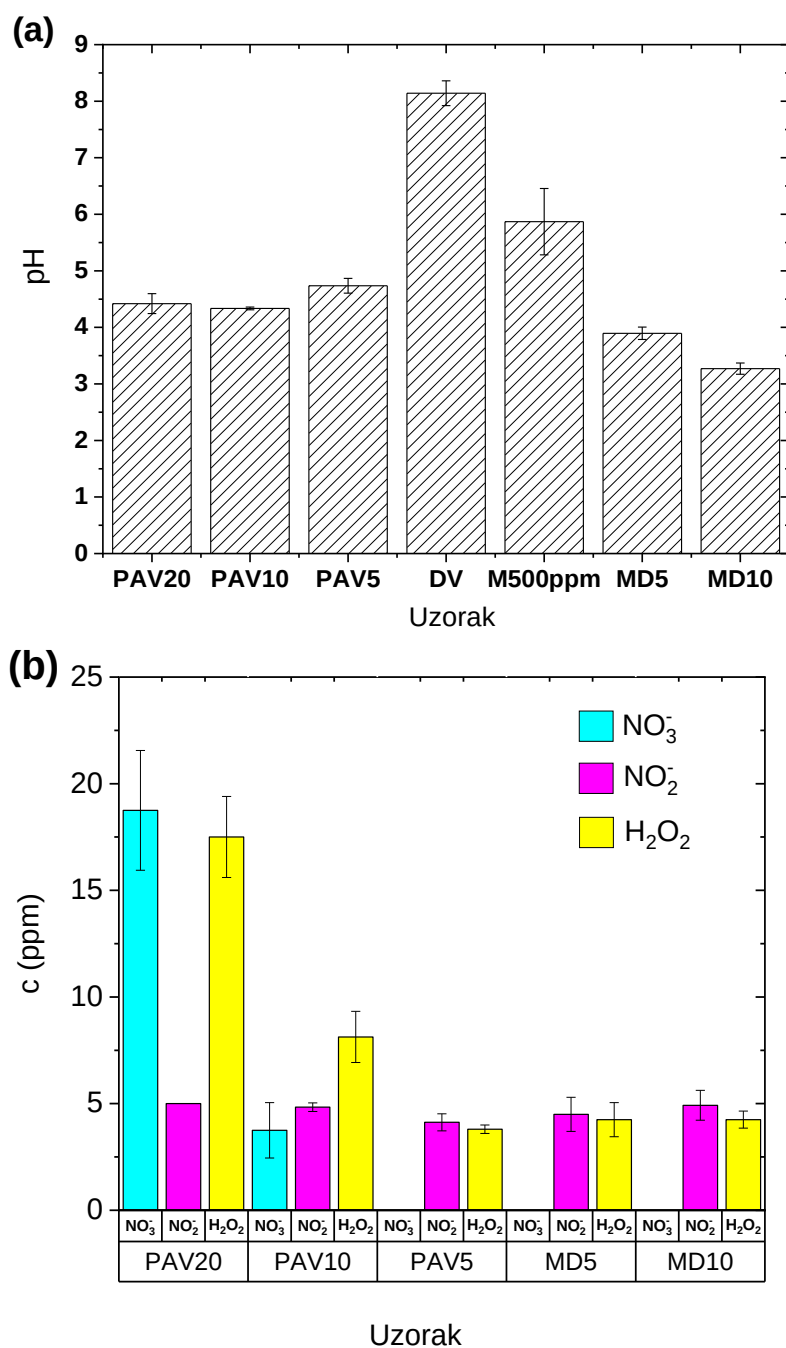
Slika 4.9: Promena temperatura vodenog uzorka (DV) tokom tretmana plazmom (Ar 1slm) u trajanju od 10 min, za tri različite pozicije (7 mm, 10 mm i 17 mm) od vertikalne ose plazma mlaza.

Uprkos stabilizaciji temperature, značajno isparavanje se nastavilo do kraja tretmana. Tokom tretmana, zapremina uzorka se smanjila sa početnih 4 ml na $3,14 \pm 0,05$ ml nakon 5 minuta i na $2,35 \pm 0,04$ ml nakon 10 minuta, što odgovara približno 80% i 60% početne zapremine.

Ukupno zagrevanje nije bilo dovoljno da bi njime objasnilo ovoliko smanjenje zapremine, ali izmerene vrednosti temperature sugerišu da se porast temperature može javiti lokalno u neposrednoj zoni interakcije plazme i površine tečnosti. Stoga se može zaključiti da je isparavanje tečnosti određeno procesima prenosa mase usled direktne interakcije plazme i tečnosti, a ne isključivo termalnim efektima. Pored toga, stalni protok gasa (1 slm) usmeren ka površini tečnosti verovatno je dodatno pospešio isparavanje, uklanjanjem vodene pare iz oblasti neposredno iznad površine tečnosti, što dodatno potvrđuje zaključak da je prenos mase dominantni mehanizam u ovom sistemu. Usled značajnog isparavanja uzorka malih zapremina tokom tretmana argonskom plazmom, u nastavku eksperimenta je kao radni gas korišćen helijum.

Analiza tretiranih tečnih uzoraka – pH vrednost i dugoživeće vrste

Promene hemijskih svojstava tretiranih uzoraka DV i M500ppm, su ispitivane merenjem pH vrednosti i koncentracije kiseoničnih i azotih vrsta nakon tretmana plazmom. Dobijeni rezultati za pražnjenje u helijumu su prikazani na slici 4.10.



Slika 4.10: Karakteristike tretiranih uzoraka, za različito vreme trajanja tretmana i kontrolnih uzoraka: (a) pH vrednost i (b) izmerene koncentracije nitrata (plavi stubići), nitrita (rozi stubići) i vodonik peroksida (žuti stubići) u uzorcima. Prema (Jovanović, 2018).

Rezultati za izmerene pH vrednosti su deo master rada (Jovanović, 2018). i ovde su predstavljeni (na slici **4.10a**) kako bi se dobio potpuni prikaz promena koje nastaju u uzorcima usled tretmana plazmom. Tretmani DV početne pH vrednosti 8,1, doveli su do smanjenja pH vrednosti uzorka koja je bila gotovo identična u okviru eksperimentalne greške merenja za sve dužine trajanja tretmana i iznosila je između 4,3 i 4,7.

Dodavanjem malationa u DV, dobija se uzorak M500ppm čija je izmerena pH vrednost bila 5,87. U ovom slučaju dužina trajanja tretmana je uticala na pad pH vrednosti, koja je nakon 5 min tretmana iznosila 3,9, a nakon 10 min je pala na 3,3.

Koncentracija vodonik-peroksida u uzorku destilovane vode je imala stalni porast sa dužim vremenima tretmana, sa 4 ppm na 17 ppm (videti sliku **4.10b**). Ovo ponašanje je ranije primećeno i povezano je sa stvaranjem OH reaktivnih vrsta tokom tretmana plazmom (Murakami et al., 2013; Yue et al., 2016). Sa druge strane, u kontaminiranim uzorcima koncentracija H_2O_2 je ostala na oko 4 ppm nakon oba vremena tretmana. Takav rezultat može proizaći iz činjenice da su OH reaktivne vrste potrošene za interakciju sa molekulom zagađivača čime se smanjuje stvaranje H_2O_2 .

Koncentracije nitrita proizvedenih u svim uzorcima su slične – oko 5 ppm. Uzimajući u obzir smanjenje pH vrednosti, na konačnu koncentraciju nitrata može uticati reakcija peroksinitridizacije jer niže pH vrednosti favorizuju reakciju NO_2^- (Lukes et al., 2014). Prisustvo niskih koncentracija nitrita pri sniženim vrednostima pH ($pH < 4$), primećena je u istraživanjima degradacije pesticida (Khan et al., 2021; Limkoey et al., 2025). U radu Khan et al., 2020 pokazano je da koncentracija azotnih oksida NO_x ostaje konstantna i do sedam dana nakon tretmana plazmom, ali da dolazi do prelaska iz jednog oblika u drugi. Takođe, pokazano je da kod pesticida koji pripadaju grupi organofosfata, azotni oksidi imaju značajniji doprinos u degradaciji u odnosu na kiseonične vrste OH i O_3 .

Koncentracija nitrata nakon tretmana destilovane vode takođe je postepeno rasla sa najvišom vrednošću od 19 ppm, dok kod vodenog uzorka malationa uopšte nije detektovano prisustvo nitrata. Kako je za određivanje koncentracije reaktivnih vrsta korišćena metoda merenja kolorimetrijskim trakama, koja daje rezultate u okviru definisanih opsega koncentracija, urađena su dodatna merenja preciznijom spektrofotometrijskom metodom. Urađena su merenja za PAV uzorke i dobijeni rezultati za koncentracije nitrita i vodonik peroksid se podudaraju sa onima prikazanim na slici **4.10b**. U slučaju nitrata, dobijen je isti rezultat za uzorak PAV20, dok su kod uzorka PAV10 i PAV5 dobijene vrednosti 14 ± 2 ppm i 10 ± 1 ppm, respektivno. Ovi rezultati ukazuju da metodom mernih traka nije moguće detektovati koncentracije ispod 14 ppm.

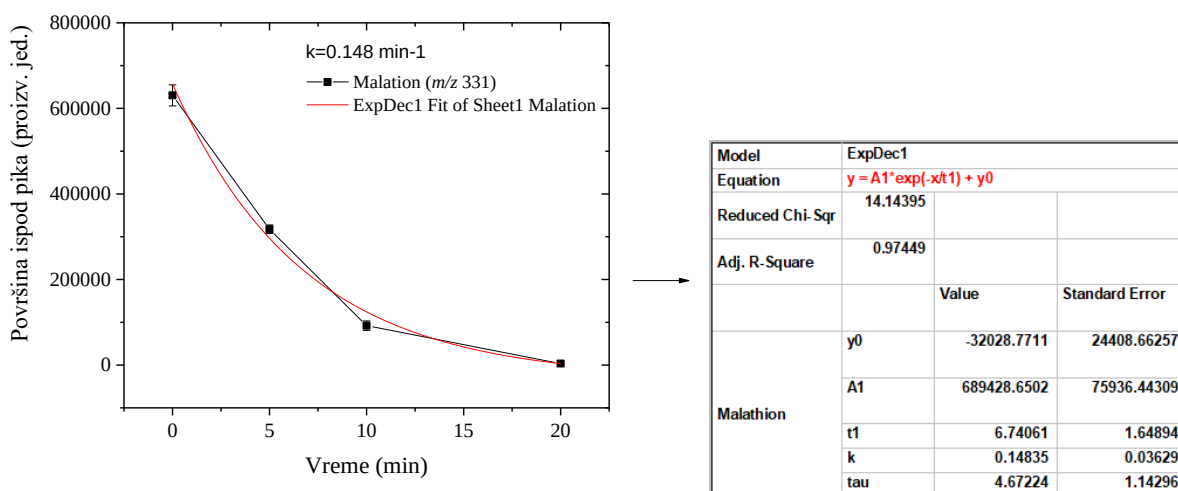
U slučaju kontaminiranih uzoraka MD5 i MD10, postojala je mogućnost da je došlo do stvaranja nitrata ali da njihovo prisustvo nije detektovano usled ograničenja primenjene metode. Primena spektrofotometrijske metode u ovom slučaju nije bila moguća jer može doći do reakcije produkata raspada, nastali tokom razgradnje, sa reagensima koji se koriste za kolorimetrijska merenja. Ipak, izostanak formiranja nitrata može biti i reakcija nusproizvoda malationa sa nitratom što dovodi do formiranja azotne kiseline, izazivajući rastvor nižeg pH nego kod odgovarajućih PAV uzoraka.

Analiza produkata raspada pesticida malationa

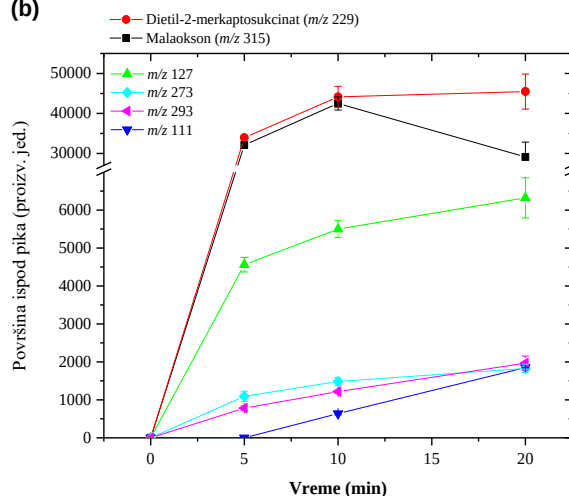
Istraživanje degradacije malationa i hemijske kinetike produkata razgradnje u tečnim uzorcima izvršeno je u LC-MS merenjima. Nakon izvršenih tretmana uzorci su razblaženi 20 puta (kao i kontrolni uzorak) zbog osetljivosti mernih instrumenta. Iz rezultata tečne-hromatografske analize početnog uzorka malationa (koncentracije 10 ppm) i tretiranih uzoraka, određene su površine ispod pikova na hromatogramu koje odgovaraju malationu i odgovarajućim proizvodima raspada, čime je omogućeno relativno poređenje koncentracija.

Na slici **4.11a** prikazano je opadanje površine ispod pika za malation, što odgovara promeni koncentracije nakon tretmana u odnosu na početnu koncentraciju. Uočeni trend se može fitovati reakcionom funkcijom prvog reda.

(a)



(b)

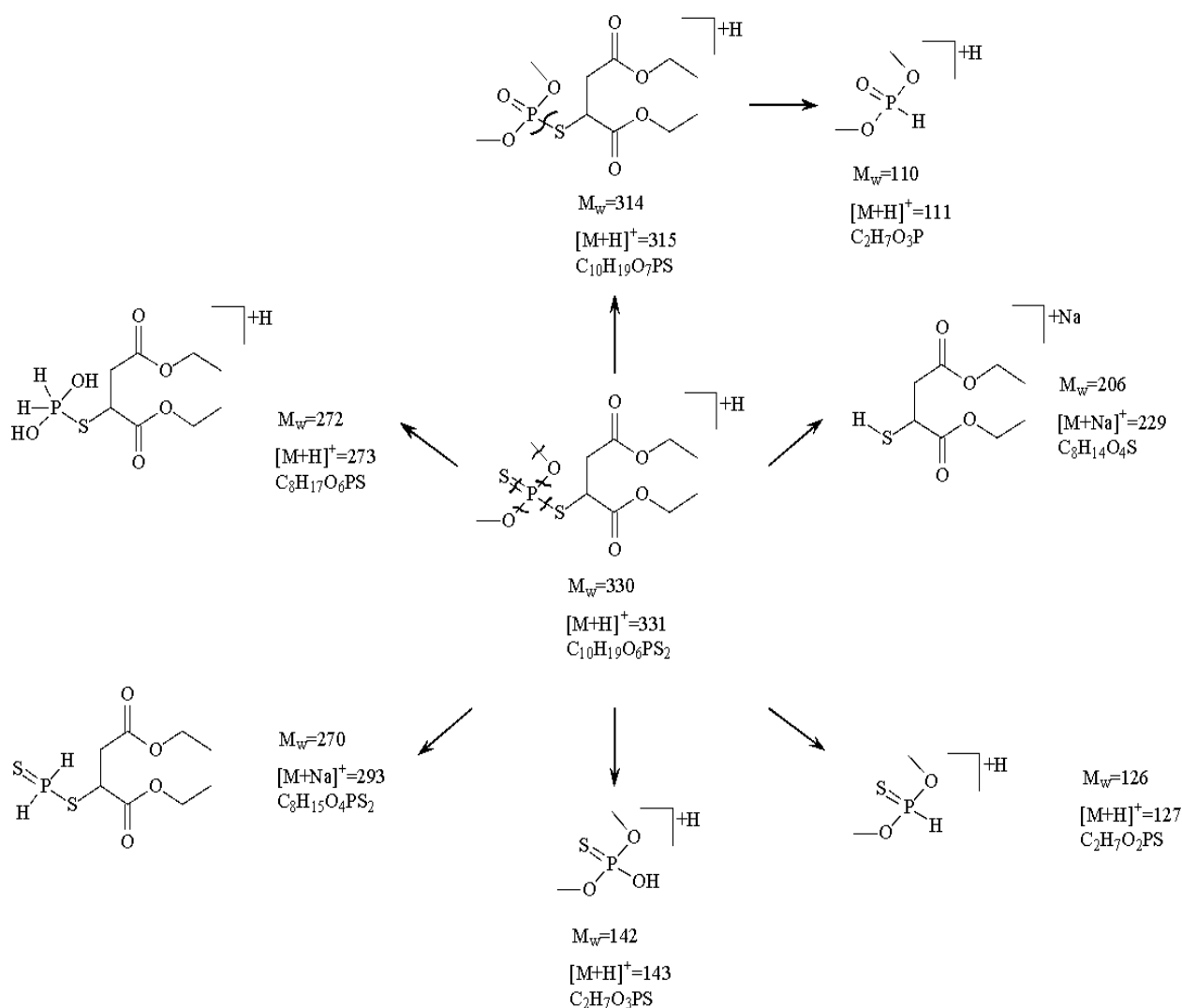


Slika 4.11: Rezultati LC-MS analize tretiranih uzoraka za tri vremena tretiranja plazmom 5 min, 10 min i 20 min. a) Smanjenje koncentracije malationa i stopa degradacije, b) hemijska kinetika produkata raspada.

Merenja su dala i informacije o masama proizvoda razgradnje malationa. Među najzastupljenijim m/z odnosima su identifikovani: malaokson na 315 amu i dietil-2-merkaptosukcinat na 229 amu koji se dalje razlaže u dimetilfosfonat (m/z 111). Takođe je utvrđeno postojanje degradacionih proizvoda (m/z 127, 273 i 293) koji su prisutni u manjem obimu. Slični odnosi m/z proizvoda razgradnje su ranije uočeni (Noradoun et al., 2005; Škoro et al., 2018; Zhu et al., 2010).

Kinetika odabranih masa praćena je u uzorcima tretiranim 5, 10 i 20 minuta i prikazana je na slici **4.11b**. Uočeno je povećanje površine ispod pika, odnosno koncentracije masa koje odgovaraju produktima razgradnje, nakon 5 i 10 minuta tretmana. Ovaj trend se nastavlja i za tretmane od 20 minuta osim u slučaju m/z=315 amu što odgovara malaoksonu. Uočeno smanjenje površine pika može se pripisati razgradnji produkata raspada koji se razlažu na molekule niže mase.

Na osnovu dobijenih rezultata predložen je put razgradnje malationa i nastanak degradacionih proizvoda, koji je prikazan na slici 4.12.



Slika 4.12: Predloženi put degradacije malationa.

ZAKLJUČAK

U okviru ovog dela istraživanja pokazana je uspešnost korišćenja plazma tehnologije za prečišćavanje vode zagađene pesticidima malationom i acetamipridom. Proučeno je i kako reaktivne vrste deponovane u tečnom uzorku utiču na razgradnju zagađivača, a u tu svrhu su urađeni i dodatni tretmanai čiste vode iste dužine trajanja.

Urađeni su tretmani vodenih uzoraka u trajanju od 5, 10 i 20 min, pri čemu su kao radni gasovi korišćeni He ili Ar. Pokazano je da oba tipa pražnjenja dovode do isparavanja uzorka tokom tretmana, ali je isparavanje 20-30% veće u slučaju argonskog pražnjenja, za istu dužinu trajanja tretmana. Sa druge strane, rezultati su pokazali da bez obzira da li u vodi ima zagađivača ili ne, za dati radni gas, ispari ista količina tečnosti za isto vreme trajanja tretmana.

Imajući u vidu ove rezultate, kao i da je temperatura ključanja pesticida malationa 156–157 °C (Budavari et al., 1996), a kod acetamiprida dolazi do raspadanja pre postizanja temperature ključanja (od 200 °C) (Lewis et al., 2016), može se zaključiti da plazma ne utiče na isparavanje zagađujućih supstanci tokom trajanja tretmana i njihovo ispuštanje u okolni vazduh.

Iz merenja degradacije matičnog molekula ustanovljena je zavisnost efikasnosti dekontaminacije od vremena tretmana. Prikazano je da tretman plazmom dovodi do efikasne i značajne degradacija pesticida malationa, za 50 % nakon 5 min, 85 % nakon 10 min i više od 99% nakon 20 min tretmana. Dodatno je uočena i pojava produkata razgradnje u tečnom uzorku i praćena je njihova koncentracija tokom tretmana. Dalja istraživanja su potrebna kako bi se procenila toksičnost nastalih nusproizvoda.

Ipak, u prethodnom istraživanju (Jovanović, 2018) urađena je provera da li tretirana voda može ponovo da se upotrebi u oblasti poljoprivrede – za imbibiranje semena rotkvice i kukuruza. Rezultati klijanja semena zalivenih plazmom dekontaminiranom vodom su pokazali povećanje procenta klijavosti u poređenju sa slučajem imbibicije semena sa vodom zagađenom malationom, za oba korišćena semena. U slučaju pesticida acetamiprida, pokazano je da nakon tretmana od 20 min tretirani tečni uzorak (AD20) dovodi do iste klijavosti semena kao i DV.

Time je pokazano da se tehnologija niskotemperaturske plazme može uspešno upotrebiti za prečišćavanje zagađene vode do nivoa da se ta voda može ponovo koristiti u oblasti poljoprivrede. U istraživanju je takođe pokazano da PAV pozitivno utiče na proces klijanja semena, a na uspešnost primene utiče kako sadržaj PAV-a (vrste i količine reaktivnih jedinjenja), tako i vrsta biljke koja se tretira.

4.2.2 EFEKTI TRETMANA NA DEGRADACIJU ORGANSKIH JEDINJENJA PFAS U VODI

Efikasnost tretmana plazmom za uklanjanje šest PFAS supstanci, koje su odabrane kao predstavnici različitih grupa perfluorisanih jedinjenja, ispitana je u saradnji sa kolegama sa instituta ICRA u Đironi, Španija. Istraživanje samih mehanizama degradacije bilo je deo doktorske teze Topolovec 2025. Rezultati iz tog istraživanja koji su predstavljeni u ovom poglavlju su pre svega fokusirani na osobine tečnih uzoraka (temperatura, pH vrednost, električna provodljivost) nakon tretmana plazmom, u zavisnosti od toga da li je korišćena destilovana voda ili voda sa složenom matricom zagađivača. Rezultati u okviru ovog dela disertacije su prikazani u radu Topolovec et al. 2024. Uspešnost plazme za uklanjanje zagađivača procenjena je u 3 različite matrice: destilovanoj vodi (DV), vodi sa česme (Voda Č) i otpadnoj vodi koja je prethodno podvrgnuta nekoj metodi prečišćavanja - sekundarnom efluentu (SE).

Eksperimentalna postavka i metodi merenja

U svim eksperimentima za uspostavljanje pražnjenja korišćen je argon kao radni gas, sa protokom 1 slm. RMS vrednost napona kojim je napajana elektroda bila je u opsegu od 600 ± 8 V do 810 ± 32 V, a snaga oslobođena u pražnjenju od $8,1 \pm 0,1$ W do $9,3 \pm 0,2$ W, u zavisnosti od sastava mete. Rastojanje između elektrode i tečnog uzorka bilo je $d=10$ mm. Tretirano je 10 ml uzorka koji se nalazio u staklenoj petri posudi (ϕ 55 mm). Urađeni su tretmani u intervalima dužine 3, 5, 7 i 10 min.

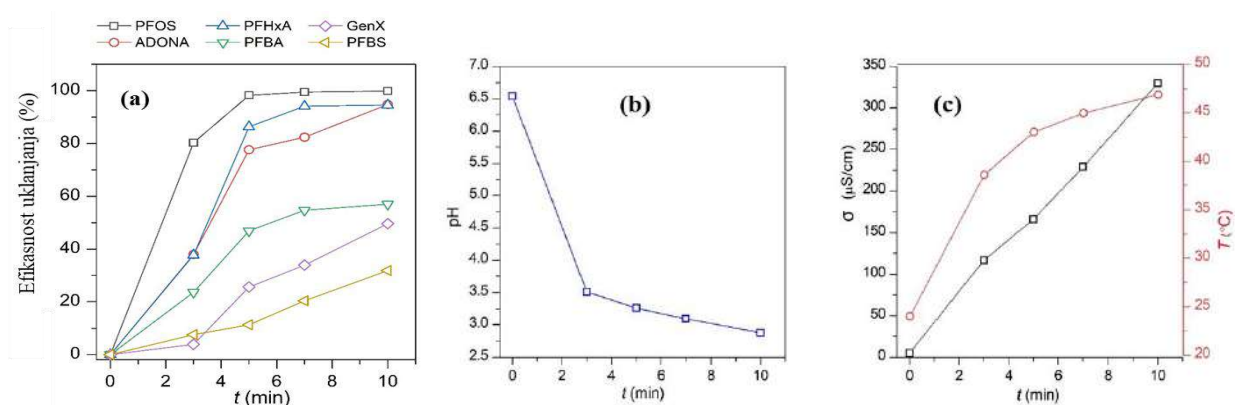
Pripremljeni su pojedinačni vodeni rastvori jedinjenja PFOS, PFBS, PFBA, PFHxA, GenX, ADONA. Početna koncentracija svakog rastvora bila je $100 \mu\text{g/l}$. Rastvori su pravljani u tri različite vodene matrice: DV, Voda Č i SE. Merene su fizičko-hemijske osobine uzoraka pre i nakon tretmana, pH i električna provodljivost, a kontrolni uzorci su bile netretirane matrice DV, Voda Č i SE. Temperatura uzorka je merena termo-optičkom sondom, sa ciljem da se isprati da li se tokom tretmana dostižu temperature isparavanja tretiranih jedinjenja. DV je služila kao model da se istraže optimalni uslovi za uklanjanje PFAS iz vode, a da nema uticaja rastvarača (matrice). Voda Č i SE su omogućili da se istraži ponašanje i efikasnost degradacije PFAS u složenijim okruženjima.

Za proučavanje ciljanih PFAS jedinjenja korišćen je hibridni maseni spektrometar sa trostrukim kvadrupolno-linearnim jonskim trapom (QqLIT-MS), opremljenim sa turbo jonskim raspršivačem i spojenim na tečni hromatograf (UPLC). Za (neselektivnu) identifikaciju produkata raspada svih komponenti korišćen je metoda tečne hromatografije sa masenim spektrometrom visoke rezolucije (LC-HRMS). Detalji o korišćenoj aparaturi i metodama mogu se naći u radu (Topolovec et al. 2024).

REZULTATI I DISKUSIJA

Tretmani zagađivača u DV

Efikasnost uklanjanja proučavanih PFAS rastvorenih u DV, tokom deset minuta tretmana plazmom, prikazana je na slici 4.13a. Pokazalo se da što je veća dužina ugljenikovih lanaca PFAS jedinjenja, postignuta je veća efikasnost uklanjanja i dostigla vrednost od 90% za jedinjenja PFOS(C8), PFHxA(C6) i ADONA(C7). Pošto površinska aktivnost ovih jedinjenja raste sa dužinom lanca, kao rezultat same strukture jedinjenja dugolančani PFAS su ostvarili veći kontakt sa plazmom na graničnom sloju interakcije plazme i vode, što je dovelo do brže degradacije. Kratkolančani PFAS (PFBA, GenX, PFBS) na slici 4.13a imaju manju tendenciju da se akumuliraju na površini, zbog čega je potrebno duže vreme da bi se razgradili. Kod svih jedinjenja je pokazano da nakon 6 min tretmana dolazi do usporavanja rasta efikasnosti degradacije. To može objasniti kao posledicom stvaranja produkata raspada koji u zavisnosti od vrste jedinjenja nastaju u različitim trenucima tokom tretmana.



Slika 4.13: (a) Efikasnost uklanjanja PFAS jedinjenja u DV, (b) pH vrednost uzoraka, (c) električna provodljivost i temperatura uzoraka, tokom 10 min tretmana. Prema (Topolovec et al., 2024).

Tokom tretmana praćene su promene pH vrednosti, električne provodljivosti, zapremina i temperature uzorka. Pošto su za sva jedinjenja izmerene približno iste vrednosti parametara, rezultati su prikazani kao srednje vrednosti parametara merenih tokom nezavisnih tretmana različitih supstanci. Početna pH vrednost uzoraka (vrednost između 6,5 i 7) tokom prva tri minuta tretmana padne na vrednost oko 3 (slika 4.13b). Ovakvo ponašanje je već primećeno kod tretmana čiste DV usled stvaranja dugoživećih reaktivnih vrsta (pogledati potpoglavlje 4.1.1). Pored toga, degradacijom jedinjenja iz rastvora nastaju produkti raspada, kao što su sumporna kiselina, H^+ jon i vodonik peroksid koji takođe utiču na kiselost tečnosti (Marotta et al., 2011).

Početna vrednost električne provodljivosti uzoraka (slika 4.13c) bila je oko 4 $\mu\text{S/cm}$, a provodljivost je linearno rasla tokom tretmana, dostižući finalnu vrednost od 325 $\mu\text{S/cm}$. Ovakav trend porasta provodljivosti sa vremenom tretmana primećen je u ranijim istraživanjima (R. K. Singh et al., 2021; R. K. Singh, Multari, et al., 2019; Wang et al., 2019), gde je pretpostavljeno da elektroni iz pražnjenja interaguju sa molekulima vode, razdvajajući ih na jone. Na taj način se povećava provodnost vode, a tome doprinose i jonske vrste koje u vodu dospevaju iz plazme u gasnoj fazi. Neki autori ukazuju na negativnu korelaciju između veoma visoke provodljivosti (iznad 20 mS/cm) i degradacije zagađivača u vodi, pretpostavljajući da veća koncentracija jona smanjuje kontaktnu površinu plazme i tretiranog jedinjenja (R. K. Singh, Multari, et al., 2019).

Drugi autori smatraju da provodljivost uzorka ne utiče na degradaciju ili čak ima pozitivan uticaj (Wang et al., 2019). U slučaju ovog eksperimenta, maksimalna vrednost električne provodljivosti (325 $\mu\text{S/cm}$) je neznatna (u odnosu na 20 mS/cm) i došlo je do smanjenja degradacije tokom tretmana.

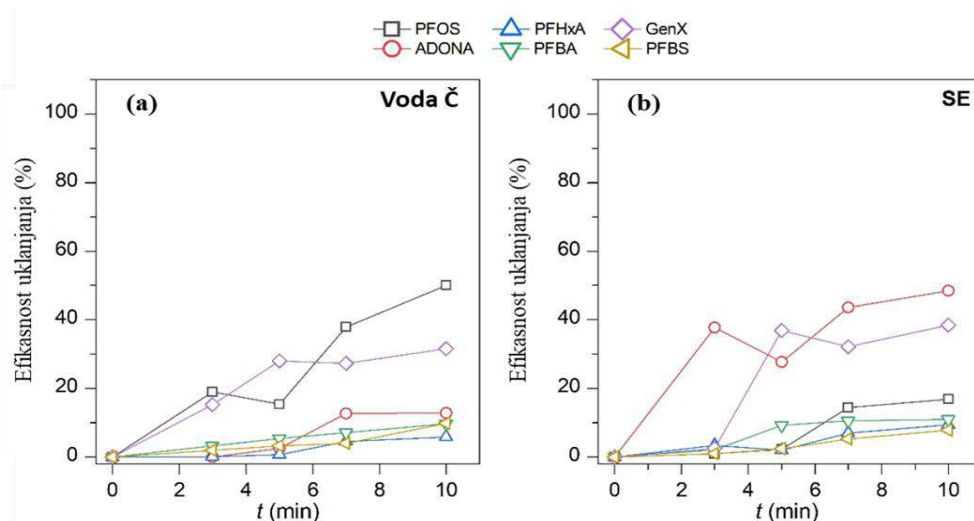
Uzrok tome može se naći u nadmetanju između početnog jedinjenja koje se tretira i njegovih produkata raspada u interakciji sa vrstama iz plazme, a ne u promeni provodnosti uzorka.

Zapremina uzoraka nakon tretmana merena je kako bi se utvrdili potencijalni gubici uzorka usled isparavanja. Dobijeni rezultati su pokazali da se nakon 10 min tretmana zapremina smanjila za oko 10 %. Ovaj podatak je uzet prilikom normiranja drugih merenja. Kako bi se ispitala isparljivost uzorka, tokom tretmana je merena temperatura uzorka (u zapremini uzorka). Rezultati pokazuju da rast temperature uzoraka nije linearan sa porastom vremena tretiranja, da usporava na vrednosti od oko 40°C i maksimalna vrednost nakon 10 min tretmana ne prelazi vrednost od 50°C (slika 4.13c).

Izmerene vrednosti temperature, zajedno sa prethodna merenjima temperature, na različitim pozicijama unutar zapremine uzorka (videti sliku 4.10), ukazuju da do termalnih isparavanja tečnosti usled dejstva plazme može doći samo lokalno u tački interakcije strimera i površine vode, ali je i na ovoj poziciji izmerena temperatura i dalje bila ispod 100°C. Stoga se zaključuje da je dominantan mehanizam isparavanja uzorka promena parcijalnog pritiska u zoni interakcije protoka radnog gasa i tečnosti. Takođe, izmerene temperature dostignute tokom tetmana pokazuju da je degradacija zagađivača potpuno netermalni proces.

Tretmani zagađivača u složenima matricama

Pored ispitivanja efikasnosti uklanjanja PFAS jedinjenja iz DV, što je uglavnom bio slučaj u prethodnim istraživanjima gde je korišćena tehnologija niskotemperaturne plazme, ovde je urađeno i istraživanje sa dve složene matrice. Pronađeno je ukupno šest različitih produkata rasapada od kojih je samo jedan do sada pronađen u prethodnim studijama (Singh et al., 2019). Efikasnost uklanjanja šest PFAS jedinjenja u zavisnosti od vremena prikazana je na slici 4.14, za (a) Vodu Č i (b) SE. U oba slučaja dobijena je značajno manja efikasnost u poređenju sa DV. U slučaju tri kratkolančana jedinjenja, ova zavisnost je gotovo linearna i maksimalna degradacija ne prelazi 10%. Kod dugolančanih jedinjenja efikasnost je oko 40%, što je više nego dvostruko manje u poređenju sa degradacijom istih jedinjenja u destilovanoj vodi.

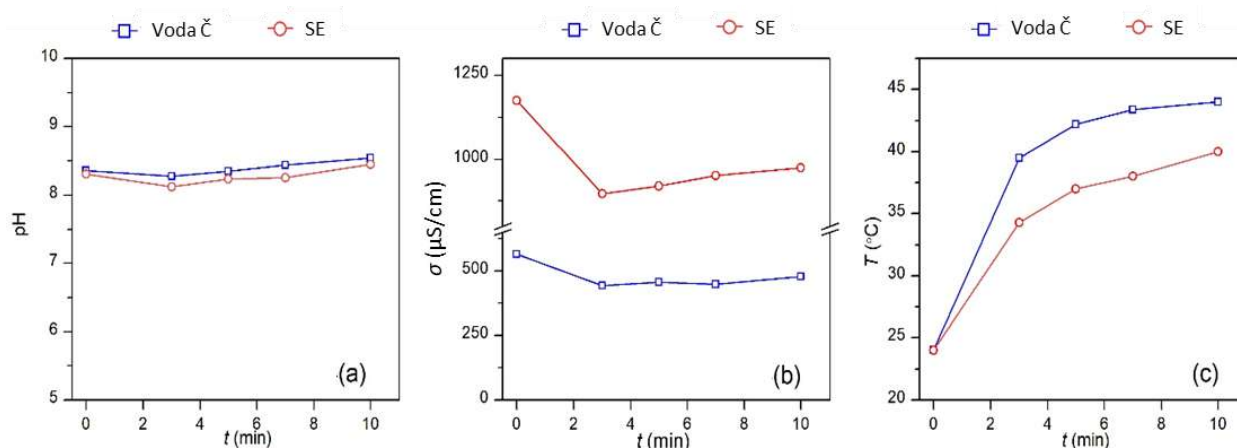


Slika 4.14: Efikasnost uklanjanja PFAS jedinjenja u (a) Vodu Č i (b) SE u zavisnosti od vremena trajanja tretmana. Prema (Topolovec et al., 2024).

Merenje pH vrednosti (slika 4.15a) je pokazalo da u oba slučaja ne dolazi do značajne promene početne vrednosti od 8,3. Početna pH vrednost Vode Č i SE se nije značajno promenila dodavanjem PFAS jedinjenja (pre tretmana), što ukazuje da ona ni tokom tretmana ne utiču na promenu pH vrednosti rastvora. Nepromenljivost pH vrednosti kod Vode Č razmatrano je u potpoglavlju 4.1.2.

Početne vrednosti provodljivosti Vode Č i SE bile su 500 $\mu\text{S}/\text{cm}$ i 1200 $\mu\text{S}/\text{cm}$, respektivno. U oba slučaja zapažen je pad ove vrednosti na početku tretmana, a zatim dolazi do blagog rasta (slika 4.15b).

Ovaj trend je mnogo izražajniiji kod SE, ukazujući da kompleksnost samog rastvarača utiče na provodljivost uzorka. Porast temperature sa dužinom trajanja tretmana (slika 4.15c) je veoma sličan onom u slučaju DV, a smanjenje zapremine nakon 10 min tretmana je bilo oko 7%.



Slika 4.15: Zavisnost (a) pH vrednosti, (b) σ – električne provodljivosti i (c) T – temperature od vremena trajanja tretmana, za slozene matrice Voda Č i SE. Prema (Topolovec et al., 2024).

ZAKLJUČAK

U okviru ovog dela istraživanja ispitana je mogućnost primene (u laboratorijskim razmerama) netermalne plazme u tretmanima vodenih uzoraka kontaminiranih PFAS jedinjenjima. Proučeni su efekti tretmana u zavisnosti od korišćene vodene matrice – DV ili slozenih matrica (Voda Č i SE).

Količine rastvorenih PFAS jedinjenja u DV nisu dovele do promene pH vrednosti (ostala je oko 7) i električne provodnosti tečnog uzorka (4 $\mu\text{S}/\text{cm}$). Tretmani su doveli do zakišeljavanja uzorka već u prvim minutima tretmana, a nakon 10 min tretmana pH vrednost je bila ispod 3. Dobijena je zavisnost električne provodljivosti uzoraka od vremena trajanja tretmana koja je pokazala rast el. provodnosti do vrednosti od 325 $\mu\text{S}/\text{cm}$ nakon 10 min.

Nasuprot ovim rezultatima, kod slozenih matrica ni dodavanje PFAS jedinjenja ni tretmani vode nisu doveli do promene pH vrednosti uzoraka. Merenja su pokazala i da u prvim minutima dolazi do pada električne provodnosti, koja zatim blago raste sa porastom vremena tretiranja, ali ni nakon 10 min ne dostiže početnu vrednost. Ovi rezultati su u skladu sa rezultatima dobijenim pri tretmanu (čiste) Vode Č prikazanim u potpoglavlju 4.1.2 (tabela 4.4) i drugim istraživanjima (Chiappim et al., 2021; Judée et al., 2018), gde je obajšnjeno da sastav početne matrice određuje dinamiku promene ovih veličina.

Kako bi se ispitalo da li plazma utiče na isparavanje PFAS jedinjenja merena je temperatura unutar zapremine uzorka tokom tretmana. Za sve korišćene matrice je pokazano da iako temperatura raste sa vremenom trajanja tretmana, ona nije prelazila vrednost od 50 $^{\circ}\text{C}$, iz čega se može zaključiti da do degradacije PFAS jedinjenja nije moglo da dođe usled termalnih efekata.

Pokazana je uspešnost tretmana u uklanjanju PFAS jedinjenja iz kontaminirane vode, za tri različite vodene matrice. Visoka efikasnost (>90%) je postignuta u DV za dugolančane PFAS: PFOS, PFHkA i ADONA za 10 minuta vremena tretmana, dok su druga jedinjenja pokazala manju efikasnost uklanjanja, ali i dalje visoku za kratko vreme tretmana. U slozenim matricama (Voda Č i SE) koje predstavljaju realne vodene uzorke, u celini je procenat degradacije bio manji, između 8% i 50%, u zavisnosti od jedinjenja i matrice. Iz toga se može zaključiti da degradacija zavisi od samog jedinjenja i da je pod uticajem različitih efekata korišćene matrice. Dalja istraživanja u ovoj oblasti su potrebna da bi se istražila toksičnost produkata raspada nastalih u tretmanu plazmom.

5. ZAKLJUČAK

U ovoj disertaciji sprovedena su eksperimentalna istraživanja u okviru kojih su proučavane osobine neravnotežnog pražnjenja u kontaktu sa tečnostima. Glavni cilj bila je detaljna dijagnostika plazma mlaza sa šiljastom elektrodom koji radi na atmosferskom pritisku sa napajanjem u oblasti kHz frekvencija i u kontaktu sa tečnošću. Dijagnostika je obuhvatala električnu karakterizaciju uređaja i primenu optičkih metoda za karakterizaciju pražnjenja u gasnoj fazi. Ispitivanjem uticaja različitih parametara sistema na osobine strimerskog pražnjenja definisani su relevantni parametri plazme, razjašnjeni fizičko-hemijski mehanizmi stvaranja reaktivnih vrsta u gasnoj fazi i procesi na prelazu iz gasa u tečnost, važni za formiranje reaktivnih vrsta u tečnoj fazi. Utvrđena je metoda za njihovu detekciju u tečnostima i izvršena dijagnostika tečne faze, kako bi se ispunio i drugi cilj - da se uređaj uspešno primeni u oblastima biotehnologije i procesima prečišćavanja zagađenih voda.

OSNOVNA ISTRAŽIVANJA U FIZICI NERAVNOTEŽNE PLAZME

Električne osobine pražnjenja

Rezultati fundamentalnih istraživanja predstavljeni su u okviru trećeg poglavlja ove disertacije. Prvi deo ovog poglavlja obuhvata rezultate električne karakterizacije plazma mlaza napajanog visokonaponskim sinusoidnim signalom u oblasti kHz frekvencija. Uticaj različitih parametara u sistemu – vrsta i protok radnog gasa, međuelektrodno rastojanje i vrsta tečne mete, na I - V karakteristike gasnog pražnjenja proučavani su kako bi se odredili najpogodniji uslovi rada plazma mlaza za konkretne primene. Analiza I - V karakteristika pokazala je da impedansa sistema ima kapacitivnu prirodu kada nema protoka radnog gasa. Kod pražnjenja u helijumu, fazna razlika između struje i napona ostaje gotovo nepromenjena, dok suprotno tome, kod argonskog pražnjenja pojava plazme uvodi rezistivnu komponentu koja menja impedansu sistema i dovodi do nelinearnosti I - V karakteristika.

U slučaju helijuma, za uspostavljanje pražnjeja na manjem međuelektrodnom rastojanju $d = 5$ mm, potrebno je primeniti značajno viši napon ($V_{rms}=1200$ V), nego kod većih rastojanja (10 mm i 15 mm, $V_{rms} = 700$ V). Nakon pojave pražnjenja kod većih rastojanja I - V karakteristika zadržava linearni oblik, dok na rastojanju 5 mm postaje nelinearna, što je praćeno naglim skokom struje, intenzivnim zračenjem i širenjem strimerskog kanala.

Sa druge strane, kod pražnjenja u argonu, stabilno pražnjenje se uspostavlja u veoma uskom opsegu rastojanja d ($d=10\pm 2$ mm) i protoka radnog gasa ($q=1-1,5$ slm). Pri tim uslovima, I - V karakteristika pre proboja u gasu ima linearni oblik, do vrednosti napona od $V_{rms} = 1900$ V. Nakon proboja dolazi do pada napona na vrednost od oko 1200 V, a sa povećanjem snage na napajanju plazma mlaza dolazi samo do porasta struje koji se manifestuje kroz širenje strimerskog kanala.

Snaga oslobođena u pražnjenju je od ključnog značaja jer određuje efikasnost tehnologije zasnovane na neravnotežnoj plazmi, zbog čega je njeno precizno merenje tokom tretmana neophodno. Merenje oslobođene snage sprovedeno je i u odsustvu radnog gasa i pražnjenja, kako bi se odredili gubici na elementima električnog kola. Određivana u dve tačke sistema – pre plazma mlaza i u uzemljenoj grani. U slučaju helijuma poređeni su rezultati korišćenja dve standardne merode (I - V i Q - V). Pokazano je da obe metode daju istu zavisnost $\langle P_G \rangle$ od V_{rms} , ali i da izbor kapacitivnosti i vrste kondenzatora značajno utiče na apsolutne vrednosti. Pri korišćenju kondenzatora od 0,8 nF i 10,3 nF dobijena je razlika od oko 23%, dok su film kondenzatori davali nefizičke rezultate na frekvencijama reda stotina kHz. Zbog toga je utvrđeno da kapacitivnost mernog kondenzatora treba biti barem jedan red veličine veća od kapacitivnosti izvora plazme. U slučaju I - V metode, izbor otpornika treba da bude takav da ograniči rast struje pražnjenja i time promenu režima pražnjenja, naročito kod provodnih meta male otpornosti. Upoređivanjem gasova uočena je jasna razlika u energetske efikasnosti: u helijumu je u pražnjenju oslobođeno manje od 30% ukupne snage prenete plazma mlazu ($\langle P_G \rangle$ do 1,7 W), dok je u argonu taj procenat bio 76%, sa znatno većim izmenjenim vrednostima ($\langle P_G \rangle$ do 12 W).

Na kraju, ispitivan je uticaj električne provodnosti tečne mete na pražnjenje i poređeni su rezultati za destilovanu vodu (2 μ S/cm) i vodu sa dodatkom malationa (500 ppm, 10 μ S/cm). Pokazano je da i pored mogućeg ponašanja organskog jedinjenja kao surfaktanta, čime lokalno utiče naelektričnu provodljivost tečnosti, koncentracija od 500 ppm ne menja režim pražnjenja u odnosu na čistu destilovanu vodu.

Optičke osobine pražnjenja

Za karakterizaciju gasne faze pražnjenja korišćena je OES metoda (He i Ar) sa slikanjem vremenski integrisane prostorne raspodele strimerskog pražnjenja (Ar) u kontaktu sa destilovanom vodom iCCD kamerom. Dodatno je snimljeno kretanje, širenje i grananje strimerskog pražnjenja u argonu u kontaktu sa metama različite električne provodljivosti.

Snimanjem emisionog spektra u argonu, usled veće struje pražnjenja i koncentracije elektrona, dolazi do intenzivnije emisije primarnih (N, N₂, O i O₂) i sekundarnih vrsta (OH i NO), nego u slučaju pražnjenja u helijumu. Širi strimer i veća kontaktna površina sa vodom pospešuju emisiju OH. Dodavanje sintetičkog vazduha (3%) u protok argona, povećava emisiju azotnih i kiseoničnih vrsta za oko 30%, bez pobuđivanja novih vrsta.

Imajući u vidu rezultate OES metode i električne karakterizacije pražnjenja, pokazalo se da su prenos energije u pražnjenje i efikasnost u pobuđivanju reaktivnih vrsta daleko efikasniji u argonu nego u helijumu. Iz tog razloga je u daljoj dijagnostici korišćen samo argon kao radni gas.

iCCD snimci pokazali su da se strimersko pražnjenje prostire duž celog međuelektrodnog rastojanja, pri čemu intenzitet zračenja opada radijalno od centra kanala, a raste od elektrode ka površini vode. Sa povećanjem snage oslobođene u pražnjenju dolazi do širenja provodnog kanala, čiji je izmereni dijametar bio u opsegu 0,5–0,8 mm, a procenjena gustina struje iznosila je $j = (0,5–1,5) \cdot 10^5$ A/m². Spektralno razlaganje pomoću optičkih filtera pokazalo je da najintenzivnije zračenje potiče od OH molekula ($\lambda = 310$ nm), dok je intenzitet drugih vrsta (N₂, O, Ar) manji. Intenziteti zračenja svih vrsta rasli sa povećanjem snage pražnjenja, što je u skladu sa rezultatima analize spektra zračenja.

Snimanjem brzom kamerom praćena je dinamika i račvanje strimerskog kanala na površini vodenih uzoraka različite provodljivosti (2–530 μ S/cm). Pokazano je da sa porastom σ opadaju dužina i broj filamenata, intenzitet emisije i kontaktna površina (procenjena na 10 – 50 mm²). Kod dielektrične mete (DV, niska σ) filamenti su najbrojniji i najduži i šire se radijalno po površini usled nagomilavanja prostornog naelektrisanja.

Nasuprot tome, kod provodljivijih tečnosti (FR i VČ) prisustvo jona u tečnosti ubrzava neutralizaciju na površini, pa se strimer lokalizuje, a energija pražnjenja oslobađa na manjoj oblasti. Ovi rezultati naglašavaju značaj provodljivosti i kontaktne površine u efikasnosti tretmana tečnosti plazmom, posebno pri uklanjanju površinskih zagađivača.

PRIMENE PLAZMA MLAZA

Proizvodnja PAV i primena u biotehnologiji

Prva oblast primene plazma mlaza obuhvatila je tretmane biljnog materijala PAV-om i ispitivanje potencijalnog toksičnog uticaja PAV-a na živi svet u vodi. U prvom eksperimentu je biljni materijal, kalusi dve sorte šargarepe (narandžaste i crne), tretiran PAV-om kako bi se ispitala mogućnost podsticanja oksidativnog stresa i proizvodnje sekundarnih metabolita. Dobijeni rezultati pokazali su da tretman PAV-om izaziva značajne fiziološke i metaboličke promene, pri čemu je uočena zavisnost od sorte i sastava PAV-a: PAV sa manjim sadržajem NO_3^- i H_2O_2 je stimulisao rast sveže mase kod narandžaste sorte, dok je kod crne sorte imao inhibitorsko dejstvo, koje je prevaziđeno primenom PAV bogatijeg ovim vrstama. Tretman je takođe doveo do povećane aktivnosti antioksidativnih enzima (CAT, SOD, POX) i promena u profilu specijalizovanih metabolita (fenola i flavonoida), čime je potvrđeno da se kontrolom karakteristika PAV-a može ciljno uticati na dinamiku rasta i metabolizam biljnih ćelija.

Prečišćavanje vode primenom plazme

Druga oblast istraživanja odnosila se na primenu hladne plazme u tehnologijama prečišćavanja zagađene vode, sa ciljem da se ispita efikasnost plazme kao naprednog oksidacionog procesa za uklanjanje postojanih organskih zagađivača. Cilj je bio da se proceni da li se plazmom tretirana voda može ponovo koristiti, kao i da se razjasne mehanizmi razgradnje i mogućnost formiranja produkata raspada.

Prvi eksperiment obuhvatio je ispitivanje degradacije pesticida (malation, acetamiprid) rastvorene u DV, uz tretmane trajanja 5, 10 i 20 min, u uslovima pražnjenja u helijumu i argonu. Uočeno je da:

- količina vode koja ispari tokom tretmana zavisi od radnog gasa (20–30% veće isparavanje u argonu), ali ne i od prisustva pesticida u vodi; plazma ne utiče na isparavanje pesticida usled termalnog zagrevanja;
- efikasnost degradacije raste sa vremenom tretmana, pri čemu je uklanjanje matičnih molekula iznosilo 50% nakon 5 min, 85% nakon 10 min i >99% nakon 20 min;
- u toku razgradnje dolazi do formiranja produkata raspada, stoga je njihova koncentracija praćena tokom tretmana;
- testovi klijanja semena pokazali su da voda nakon dekontaminacije ne pokazuje toksične efekte, već može poboljšati klijavost, u poređenju sa zagađenom vodom.

Drugi eksperiment se odnosio na uklanjanje perfluoroalkilnih i polifluoroalkilnih supstanci (PFAS) u različitim vodenim matricama (destilovana voda, voda sa česme i sekundarni efluent), gde se pokazalo da:

- u destilovanoj vodi tretmani dovode do snažne promene pH (pad sa 7 na <3) i naglog porasta električne provodljivosti (do 325 $\mu\text{S}/\text{cm}$), dok kod realnih matrica ovi efekti nisu izraženi, što ukazuje na presudan uticaj sastava početne matrice;
- temperatura tokom tretmana nije prelazila 50°C, čime je isključeno termalna razgradnja;
- visoka efikasnost uklanjanja PFAS jedinjenja (>90%) može da se postigne za dugolančane PFAS u destilovanoj vodi, dok se u složenijim matricama dobijaju niže vrednosti (8–50%), zavisno od konkretnog rastvorenog jedinjenja i matrice;
- efikasnost degradacije zavisi od hemijske strukture jedinjenja, ali i od sastava matrice u kojoj se nalaze.

Oba eksperimenta pokazuju da hladna plazma predstavlja efikasan i ekološki prihvatljiv metod za uklanjanje različitih organskih zagađivača iz vode, sa visokim potencijalom za ponovnu upotrebu tretirane vode. Takođe je pokazano da sastav matrice i tip zagađivača određuju efikasnost procesa i dinamiku promene fizičko-hemijskih parametara tokom tretmana.

NAUČNI DOPRINOS

Naučni doprinos ove disertacije ostvaruje se u oblasti fizike jonizovanih gasova kroz detaljnu analizu strimerskog pražnjenja na atmosferskom pritisku i režima pražnjenja u kontaktu sa tečnostima. Poseban doprinos odnosi se na precizno merenje snage kod strimerskog pražnjenja u oblasti kHz frekvencija, gde su primenjene dve nezavisne metode koje omogućavaju pouzdano određivanje električnih parametara sistema. Na taj način je obezbeđen veliki skup eksperimentalnih podataka koji mogu poslužiti kao osnova za dalja teorijska i numerička modelovanja ovakvih sistema, uključujući mogućnost prikazivanja pražnjenja ekvivalentnim kolom. Takođe, sistematskim eksperimentalnim ispitivanjem pokazano je da lokalne promene električne provodljivosti tečne mete (na primer u prisustvu surfaktanata) mogu značajno da utiču na inicijaciju i stabilnost pražnjenja, iako se to ne može direktno predvideti na osnovu ukupne provodljivosti rastvora. Dobijeni rezultati predstavljaju značajan doprinos razumevanju mehanizama električnog proboja na atmosferskom pritisku i interakcije plazme sa tečnostima, čime se unapređuje fundamentalno znanje u oblasti niskotemperaturne plazme i njene primene.

Doprinos se ogleda i u razumevanju procesa stvaranja reaktivnih vrsta u sistemima neravnotežne plazme na atmosferskom pritisku i njihovog prenosa u tretiranu tečnost. Pokazano je da izbor radnog gasa, snage i režima pražnjenja direktno određuje hemijske procese u plazmi, a time definiše i kreiranje dugoživećih reaktivnih vrsta i pH tj. tako se utiče i na kinetiku reakcija u tečnoj fazi. Interakcija plazme sa tečnim metama detaljno je proučena u slučaju razlaganja modelnih jedinjenja zagađivača (malation, PFAS) i stvaranjem PAV u tretmenima čiste vode. Dobijeni rezultati doprinose dubljem razumevanju mehanizama interakcije gasne i tečne faze i uslova pod kojima se procesi mogu optimizovati u cilju degradacije zagađujućih materija i sinteze reaktivnih jedinjenja od interesa za biološke i tehnološke primene.

OGRANIČENJA ISTRAŽIVANJA I BUDUĆI RAD

Ograničenja sa aspekta osnovnih istraživanja proizilaze iz nedovoljne prostorne i vremenske rezolucije dostupnih dijagnostičkih metoda. Korišćene optičke metode su dale vremenski integrisane podatke za strimersko pražnjenje. Dalja istraživanja bi trebalo da obuhvate praćenje kinetike i morfologije pražnjenja i nastajanja reaktivnih vrsta u okviru perioda primenjenog napona, tj. u μ s domenu čime bi se rezultati električne i optičke dijagnostike preciznije povezali. Druga ograničenja korišćenih optičkih tehnika uključuju: detekcioni opseg talasnih dužina u kome je moguće merenje, rezultati proističu samo od detektovanih ekscitovanih vrsta i izostanak podataka o neutralnim vrstama. Dodatno, složenost sistema u kome se reakcije odvijaju u više faza (električno pražnjenje, gas, tečnost) ograničavaju izdvajanje doprinosa pojedinačnih tipova reakcija (na primer jonizacije, disocijacije, rekombinacije). U analizi tečne faze, za uzorke sa konkretnim zagađivačima potrebno je izvršiti detekciju različitih kretkoživećih hemijskih vrsta kako bi se u potpunosti i direktnim merenjem opisali procesi degradacije.

Sa aspekta primena, ovo istraživanje bilo je ograničeno na tretmane manjih zapremina tečnosti kako bi se proučili procesi indukovani tretmanom plazmom. Za praktičnu upotrebu neophodno je razviti sisteme većeg kapaciteta i ispitati njihovu efikasnost što je potpuno novo polje istraživanja koje nameće drugačiji pristup. U eksperimentima koji uključuju tretmane biljaka, potrebna su detaljnija istraživanja dugoročnih promena u biološkim sistemima izazvanih dejstvom plazmom aktivirane vode, dok je kod prečišćavanja neophodno razmotriti mogućnost nastanka toksičnih nusproizvoda nakon razgradnje primarnog zagađivača.

Na kraju, modelovanje sistema čvrsta elektroda–gas–tečnost i dalje je ograničeno složenošću interakcija i nedostatkom eksperimentalnih podataka za validaciju, što ukazuje na potrebu za daljim kombinovanjem naprednih dijagnostičkih tehnika sa numeričkim simulacijama.

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PRILOZI

PRILOG 1

OPIS METODA KORIŠĆENIH ZA ANALIZU KALUSA ŠARGAREPE

Izolacija ukupnih proteina

Uzorci biljnog tkiva (0,02 – 0,15 g) su homogenizovani do finog praha u tečnom azotu, nakon čega im je dodat ledeni ekstrakcioni pufer u odnosu 3:1 (v/w). Za ekstrakciju ukupnih solubilnih proteina korišćen je 100 mM fosfatni pufer (pH 6,5) koji je sadržao 2 mM EDTA (pH 8), 5 mM askorbat, 2 % (w/v) nerastvorni polivinilpolipirrolidon (PVPP) i 1 mM fenilmetilsulfonil fluorid (PMSF). Nakon ekstrakcije uzorci su centrifugirani 30 minuta na 12000 *g*, na temperaturi od 4°C. Dobijeni supernatant je čuvan na -80°C i korišćen za determinaciju ukupnih proteina i ispitivanje aktivnosti enzima. Koncentracija ukupnih proteina je određena pomoću *Qubit* fluorimetra (*Qubit 3.0 Fluorimeter*, *Life Technologies Corporation*, NY, USA) korišćenjem *Qubit* kompleta za analizu proteina (*Fisher Scientific*).

Spektrofotometrijska analiza aktivnosti enzima

Ukupna aktivnost CAT određena je praćenjem kinetike razgradnje H₂O₂ na 240 nm, po modifikovanoj metodi koju je opisao Aebi (1984). Reakciona smeša se sastojala od 340 µl reagensa 1 (0,2 % H₂O₂ u 50 mM fosfatnom puferu, pH 7,0), 650 µl reagensa 2 (50 mM fosfatni pufer, pH 7,0) i 10 µl testiranog uzorka. Aktivnost CAT se izračunava prema jednačini:

$$A_{CAT} = [(\Delta A - \Delta A_0) * V_k * 1000] / [0,0436 * V_e]$$

ΔA - promena apsorbance (min⁻¹cm⁻¹)

ΔA_0 - promena apsorbance blank rastvora (min⁻¹cm⁻¹)

V_k - zapremina reakcione smeše u kivetu (ml)

V_e - zapremina uzorka u kivetu (ml)

0,0436 - ekstinkcioni koeficijent H₂O₂ na 240 nm (mM⁻¹cm⁻¹).

Aktivnost enzima je izražena u odnosu na koncentraciju ukupnih proteina (U mg⁻¹)

Aktivnost POX detektovana je po modifikovanoj metodi koju su opisali Jiménez-Atiénzar i saradnici (2007). Reakciona smeša je sadržala 20 µl uzorka, 12,5 µl 0,5 mM rastvora H₂O₂ i 10 µl 10 mM pirogalola u 958 µl 50 mM fosfatnog pufera (pH 6,5). Oksidacijom pirogalola nastaje smeđe obojeni purpurogalin sa maksimumom apsorpcije na 430 nm. Aktivnost enzima se računa prema jednačini:

$$A_{POX} = [(\Delta A - \Delta A_0) * V_k * 1000] / [2,47 * V_e]$$

ΔA - promena apsorbance (min⁻¹cm⁻¹)

ΔA_0 - promena apsorbance blank rastvora (min⁻¹cm⁻¹)

V_k - zapremina reakcione smeše u kivetu (ml)

V_e - zapremina uzorka u kivetu (ml)

2,47 - ekstinkcioni koeficijent pirogalola na 430 nm (mM⁻¹cm⁻¹).

Aktivnost enzima je izražena u odnosu na koncentraciju ukupnih proteina (U mg⁻¹)

Ukupna aktivnost superoksid dismutaza (SOD) je određena prema modifikovanoj metodi (Beyer & Fridovich, 1987). Reakciona smeša se sastojala od 0–25 μ l proteinskog ekstrakta, 0.1 mM EDTA, 3 mM L-metionina, 5 mM NBT, 2 mM riboflavina i 100 mM fosfatnog pufera (pH 7,8). Nakon osvetljavanja reakcione smeše praćena je promena apsorbance na 540 nm (ELISA Microplate reader, LKB Vertriebs GmbH, Austria). Aktivnost ukupnih SOD je izražena kao U mg^{-1} u odnosu na koncentraciju ukupnih proteina, pri čemu jedna SOD jedinica (U) odgovara količini enzima koja je neophodna za inhibiciju 50% NBT.

Sadržaj fenola i flavonoida

Sadržaj ukupnih fenola (TPC) i flavonoida (TFC) u biljnom ekstraktu određivan je prema protokolu koji su dali Todorović et al. (2023). Za merenje TPC metanolni ekstrakti biljaka su pomešani sa 5% NaCO_3 i inkubirani 5 min, a zatim oksidovani pomoću 50% Folin—Ciocalteu reagensa (Fermentas International Inc., Burlington, ON, Canada) tokom narednih 30 min na sobnoj temperaturi, u mraku. Apsorbanca dobijene smeše merena je na 724 nm pomoću HP Agilent 8453 spektrofotometra (Agilent Technologies, Palo Alto, CA, USA). Za kvantifikaciju je korišćena standardna kriva dobijena na osnovu poznatih koncentracija galne kiseline. Rezultati su izraženi kao miligram ekvivalenta galne kiseline po gramu suve mase ekstrakta (mg GAE g^{-1} DW).

Za određivanje TFC korišćena je kolorimetrijska metoda bazirana na formiranju kompleksa aluminijum hlorida kao što su opisali Šiler et al. (2015). Ekstrakti biljaka su pomešani sa 5% KNO_2 i inkubirani na sobnoj temperaturi. Nakon dodavanja 10% AlCl_3 formira se žuto obojeni kompleks koji u baznoj sredini, nakon dodavanja 1 M NaOH, menja boju u crveno. Apsorbanca formirane kompleksne smeše merena je na 510 nm, a rezultati su dobijeni preračunavanjem na osnovu standardne krive za rutin. TFC je izražen kao miligram ekvivalenta rutina po gramu suve mase ekstrakta (mg RE g^{-1} DW).

PRILOG 2

DETEKTOVANJE I PRAĆENJE PRODUKATA RASPADA MALATIONA METODOM TEČNE HROMATOGRAFIJE I MASENE SPEKTROMETRIJE

Analiza uzoraka tretirane zagađene vode (sa pesticidom malationom) urađena je metodom tečne hromatografije uz korišćenje masene spektroskopije kao detekcionog sistema. U te svrhe korišćen je *Dionex UltiMate® 3000 LC* sistem (*Thermo Fisher Scientific, Valtham, SAD*) opremljen sa linearnim jonskim trapom *LTQ XL* (*Thermo Fisher Scientific*). Hromatografsko razdvajanje malationa i njegovih produkata raspada je postignuto u inverznoj fazi *Zorbax Eclipse®XDB-C18* kolone, $5\text{ mm} \times 4,6\text{ mm i.d.}$ i $3,5\text{ }\mu\text{m}$ veličina čestice (*Agilent Technologies, Santa Klara, SAD*). Ispred separacione kolone, postavljena je pretkolona ($12,5\text{ mm} \times 4,6\text{ mm i.d.}$ and $5\text{ }\mu\text{m}$ particle size, *Agilent Technologies*). Uzorci su bili isfiltrirani kroz $0,45\text{ }\mu\text{m}$ celulozne filtere (*LLG Labware, Mekenhajn, Nemačka*) pre analize. Mobilna faza se sastojala od vode (A), metanola (B) i 10% sirćetne kiseline (C). Gradijent se menja na sledeći način: 0 – 5 min: A 28%, B 70%, C 2% (pri protoku gasa od $0,8\text{ mL}\cdot\text{min}^{-1}$); 5 – 15 min: B 100% (pri protoku gasa od $1,0\text{ mL}\cdot\text{min}^{-1}$). Početni uslovi su ponovo uspostavljeni i zadržani 10 min. Alikvot od $10\text{ }\mu\text{L}$ uzorka je ubrizgan u LC sistem. Stabilni maseni spektri su dobijeni korišćenjem elektrosprej jonizacije (ESI) u pozitivnom režimu. Optimalni ESI parametri bili su napon izvora od 5 kV i temperatura kapilara od $300\text{ }^{\circ}\text{C}$. Korišćena aparatura prikazana je na slici **D.1**.

U razvoju MS metode, ukupni maseni spektri uzoraka su snimljeni u opsegu m/z 50–1000. Dobijeni joni su podvrgnuti daljoj MS/MS analizi u cilju odabira najosetljivijih prelaza koji se koriste za određivanje malationa i njegovih proizvoda razgradnje u režimu praćenja odabranih reakcija (SRM).

Metanol korišćen za eksperimente je imao HPLC kvalitet (*VWR Chemicals, Fontenai-sous-Bois, Francuska*), dok je sirćetna kiselina (LC-MS grade) isporučena od *Fisher Scientific-a (Valtham, SAD)*. Dejonizovana voda je dobijena propuštanjem destilovane vode kroz sistem ultra čiste vode *GenPure (TKA, Niderelbert, Nemačka)*.



Slika D.1: LC-ESI-MS/M

Biografija

Olivera Jovanović je rođena 12.08.1994. godine u Beogradu gde je završila osnovnu i srednju školu. Osnovne akademske studije na Fizičkom fakultetu Univerziteta u Beogradu – smer Primenjena i kompjuterska fizika upisala je školske 2013. godine i završila 2017. godine sa prosečnom ocenom 9,18. Master akademske studije na istom smeru završila je 2018. godine sa prosečnom ocenom 10,00 i odbranila je rad „Tretman vode zagađene pesticidima plazma mlazom na atmosferskom pritisku za primenu u poljoprivredi“ pod mentorstvom dr Nikole Škora. Bila je stipendista Ministarstva prosvete, nauke i tehnološkog razvoja Republike Srbije u periodu od 2014.-2018. godine.

Od 2018. godine je student doktorskih studija na Fizičkom fakultetu Univerziteta u Beogradu – smer Fizika jonizovanog gasa i plazme. Od 2018. godine je zaposlena u Institutu za fiziku u Beogradu u Laboratoriji za neravnotežne procese i primenu plazme, Centra za neravnotežne procese, prvo kao istraživač pripravnik (2018.), a zatim kao istraživač saradnik (2022.).

Učestvovala je na projektima Ministarstva prosvete, nauke i tehnološkog razvoja Republike Srbije - III41011 (2018. – 2020.), Fonda za nauku Republike Srbije - APPerTAin- BIOM – 7739780 (2022.-2025.) i Dokaz koncepta PoWeRCAP-EGARDEN 14916 (od 2024. – 2025.). Bila je učesnica projekta bilateralne saradnje sa Slovačkom u periodu od 2024. – 2025. i član radnih grupa u okviru evropskih COST akcija CA19110 i CA20114 u periodu od 2020. – 2025. U okviru kratkoročnih naučnih razmena, boravila je u junu 2022. godine u Gentu, u Belgiji, u Istraživačkoj grupi za primenu plazme Univerziteta u Gentu i u maju 2023. godine u Brnu, u Češkoj Republici, u Institutu za fizičku i primenjenu hemiju Tehničkog univerziteta u Brnu.

Do sada je objavila 5 radova u međunarodnim časopisima čiji je impakt faktor veći od 1 (po jedan u kategorijama M21a+, M21a i M21 i dva u kategoriji M22). Rezultate svog istraživanja predstavila je na nekoliko međunarodnih konferencija i koautor je na 53 rada u zbornicima međunarodnih konferencija.

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Име и презиме аутора Оливера Јовановић

Број индекса 8015/2018

Студијски програм Физика јонизованог гаса и плазме

Наслов рада Плазма млаз са шиљастом електродом – карактеризација и примене
у третманима течних узорака

Ментор др Никола Шкоро

Изјављујем да је штампана верзија моје докторског рада истоветна електронској верзији коју сам предала ради похрањивања у **Дигиталном репозиторијуму Универзитета у Београду**.

Дозвољавам да се објаве моји лични подаци везани за добијање академског звања доктора наука, као што су име и презиме, година и место рођења и датум одбране рада.

Ови лични подаци могу се објавити на мрежним страницама дигиталне библиотеке, у електронском каталогу и у публикацијама Универзитета у Београду.

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Овлашћујем Универзитетску библиотеку „Светозар Марковић“ да у Дигитални репозиторијум Универзитета у Београду унесе моју докторску дисертацију под насловом:

Плазма млаз са шиљастом електродом – карактеризација и примене у третманима течних узорака

која је моје ауторско дело.

Дисертацију са свим прилозима предала сам у електронском формату погодном за трајно архивирање.

Моју докторску дисертацију похрањену у Дигиталном репозиторијуму Универзитета у Београду и доступну у отвореном приступу могу да користе сви који поштују одредбе садржане у одабраном типу лиценце Креативне заједнице (Creative Commons) за коју сам се одлучила.

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