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THALITA MAYUMI CASTALDELLI NISHIME

Development and characterization of extended and flexible plasma jets

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Development and characterization of extended and flexible plasma jets

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To my parents

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“The one thing that you have that nobody else has is you. Your voice, your mind, your story, your vision. So write and draw and build and play and dance and live as only you can.”

Neil Gaiman

ABSTRACT

The use of atmospheric pressure plasmas for different purposes has increased in recent years. With the development of atmospheric pressure plasma jets, some precise treatments such as in the biomedical field or specific surface processing became more often. However, the delivery of plasma to irregular shaped objects, inside tubes or even hollow organs is limited with the use of conventional plasma jet configurations. Therefore, those limitations can be surpassed with the development of elongated or remotely generated plasma jets. In this work, two extended plasma jet configurations aiming at different application fields were further developed and characterized. Firstly, an endoscopic plasma jet (plasma endoscope) operating with a dielectric barrier discharge (DBD) configuration in millimeter dimensions that can be coupled to a typical endoscope was developed. This plasma jet can operate with helium or neon and counts with an external concentric shielding gas channel that provides the introduction of an electronegative gas curtain around the plasma plume. The shielding gas allows the preservation of the plasma jet shape when operated inside closed cavities. The construction difficulties arisen from the use of different feed and shielding gases were explored. Carbon dioxide was proven to be a good option for the curtain gas around the plasma plume avoiding the formation of parasitic discharges inside the shielding gas tube and the endoscopic housing. When operated with neon, the plasma jet was ignited with lower applied voltages and reached a wider range of transferred power. The other developed plasma jet was the remotely generated long tube plasma jet that consists of a DBD primary discharge connected to a 1 m long flexible plastic tube with a floating metal wire inside. The metal wire penetrates a few millimetres inside the discharge and allows the generation of a plasma plume at the end of the plastic tube. This configuration permits safer and better manipulation of the plasma jet. The device was characterized using two different excitation sources, an AC power supply with continuously provided signal and one operating in burst mode. The application of voltage signal in burst mode allowed a fine adjustment of discharge parameters reaching a wider range of power. In the case using “continuous” AC voltage signal, power values up to only 1.2 W were achieved. The primary discharge geometry, when using a parallel plates configuration device, exhibited minimal influence on the plasma mean power. A pin electrode configuration long plasma jet was successfully applied for inhibition of *C. albicans* in inoculated Petri dishes in which inhibition zones were observed after treatment. This plasma jet was also used for surface modification of polyethylene terephthalate (PET) samples using different tilting positions. In this case, reductions of around

60^{o} of water contact angle were observed after plasma exposure for 60s and tilting the plasma jet let to formation of bigger treated areas.

KEYWORDS: Atmospheric pressure plasma. Plasma jet. Endoscopy. Transporting plasma jet. Decontamination. Surface treatment.

RESUMO

Nos últimos anos, tem intensificado o emprego de plasmas em pressão atmosférica para diferentes aplicações. Com o desenvolvimento dos jatos de plasma em pressão atmosférica, alguns tratamentos precisos, como no campo biomédico ou em específicos processamentos de superfícies, tornaram-se mais frequentes. No entanto, a aplicação de plasma à objetos irregulares, dentro de tubos ou mesmo dentro de órgãos ocos é limitada quando se utilizam configurações convencionais de jatos de plasma. Portanto, essas limitações podem ser superadas com o desenvolvimento de jatos de plasma alongados ou gerados remotamente. Neste trabalho, duas configurações de jato de plasma longo visando diferentes campos de aplicação foram aperfeiçoadas e caracterizadas. Inicialmente foi desenvolvido um jato de plasma endoscópico (plasma endoscope) operando em configuração de descarga por barreira dielétrica (DBD) com dimensões milimétricas, versátil ao acoplamento em endoscópios típicos. Este jato de plasma pode operar com hélio ou neônio e conta com um canal externo e concêntrico de gás que permite a introdução de uma cortina de gás eletronegativo ao redor da pluma de plasma. A cortina de proteção a gás preserva a forma do jato de plasma quando operado dentro de cavidades fechadas. As dificuldades advindas do desenvolvimento deste foram investigadas quando diferentes gases foram testados como cortina de proteção dele, dentre estes, o dióxido de carbono se mostrou uma boa opção evitando a formação de descargas parasitas dentro do tubo de gás de proteção e da estrutura do endoscópio. Quando operado com neônio, o jato de plasma pôde ser iniciado com tensões mais baixas e atinge faixas mais amplas de potência transferida. Outra configuração desenvolvida foi a de jato de plasma de tubo longo gerado remotamente que consiste em uma descarga primária DBD conectada a um tubo plástico flexível de 1 m de comprimento com um fio metálico flutuante em seu interior. O eletrodo metálico penetra alguns milímetros na descarga e permite a geração de uma pluma de plasma na extremidade final do tubo. Esta configuração possibilita uma manipulação mais segura e precisa do jato de plasma. O dispositivo foi caracterizado quando utilizadas duas fontes de excitação diferentes, uma com tensão AC aplicada continuamente e outra operando em modo “burst”. O uso de sinal de tensão em modo “pulsado” (burst) permite um ajuste mais preciso dos parâmetros da descarga atingindo um intervalo mais amplo de potência transferida. No caso do emprego da fonte de tensão AC “contínua”, a faixa de potência atingida se restringe a apenas 1,2 W. A variação da geometria da descarga primária, quando um reator de placas paralelas foi utilizado, influenciou minimamente a potência média do jato de plasma. Para aplicações, um eletrodo de alta tensão em forma de haste foi utilizado na descarga primária. Com este reator, a inibição do fungo *C. albicans* inoculado em placas de Petri se fez possível, onde a formação de halos de inibição foi observada após o tratamento com plasma. Este jato também foi utilizado para a modificação de superfície de amostras de politereftalato de etileno (PET) com diferentes ângulos de aplicação. Neste caso, reduções de aproximadamente 60º de ângulo de contato com água foram obtidos após tratamentos por 60s e a inclinação do jato de plasma permitiu um aumento na área tratada.

PALAVRAS-CHAVE: Plasmas em pressão atmosférica. Jatos de plasma. Endoscopia. Jatos de plasma de transporte. Descontaminação. Tratamento de superfícies.

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LIST OF ABBREVIATIONS

AC	Alternating current
APP	Atmospheric pressure plasma
APPJ	Atmospheric Pressure Plasma Jet
DBD	Dielectric Barrier Discharge
DC	Direct current
Dist	Distance between two steps.
DNA	Deoxyribonucleic acid
EEDF	Electron energy distribution function
FOT	Fibre optical thermometer
GaAs	Gallium arsenide
HV	High voltage
ICCD	Intensified charge-coupled device
LTE	Thermodynamic equilibrium
OES	Optical Emission Spectroscopy
PET	Polyethylene terephthalate
PTFE	Polytetrafluoroethylene
M.F.C.	Mass flow controller
Q-V	Charge-voltage
RF	Radio frequency
Rn	Reynolds number
RONS	Reactive oxygen and nitrogen species
ROS	Reactive oxygen species
RNS	Reactive nitrogen species
sccm	Standard cubic centimetre
SD	Sabouraud Dextrose
slm	Standard liters per minute
UV	Ultraviolet
VUV	Vacuum ultravioleta
CFU	Colony-forming unit
WCA	Water contact angle

LIST OF SYMBOLS

He	Helium
Ne	Neon
N_2	Nitrogen
N_2^+	Nitrogen ion
NO_x	Nitrogen oxides
CO_2	Carbon dioxide
O_2	Oxygen
O_3	Ozone
OH	Hydroxide
γ	Secondary-electron-emission coefficient
V_b	Breakdown voltage
d	Distance
p	Pressure
n_e	Electron's density
T_e	Electron's temperature
T_i	Ions temperature
T_g	Gas temperature
E'	Local electric field
e	electron charge
R	Avalanche head radius
α	Number of ionizations per length of path (Townsend's first coefficient)
E_0	Applied electric field
$V_{r,c}$	Voltage across a resistor (r) or a capacitor (c)
U_c	Energy per cycle
V_i	Measured voltage in equation 3
I_i	Measured current in equation 3
M	Length of recorded data in equation 3
Δt_3	Sample interval of equation 3
T	Period of voltage oscillation
Q	Charge
P	Power
C_{cell}	Capacitance of the dielectric

C_{eff}	Effective capacitance
U_{gap}	Bandgap energy
V_{ion}	Ionization potential
Ne*	Neon metastable
He*	Helium metastable
N	Number of oscillation cycles
T_r	Repetition period
Δt	Period of high voltage oscillations
f	Frequency
D	Duty cycle

CONTENTS

1 INTRODUCTION

Non-thermal atmospheric pressure plasmas have been studied since the 19th century, when dielectric barrier discharges (DBDs) were firstly employed by Siemens for ozone generation (KOGELSCHATZ, 2003). Since then, the use of cold atmospheric plasmas such as DBD and corona discharges have been also investigated for gas purification, electrostatic precipitation and, in the last decades, for materials surface functionalization (BRUGGEMAN; IZA; BRANDENBURG, 2017, VESEL; MOZETIC, 2017). Once they do not require expensive vacuum equipment, atmospheric pressure plasmas are cost-effective alternative for some low-pressure plasma techniques, mainly concerning material processing (VESEL, MOZETIC, 2017). Additionally, in the last years, non-thermal atmospheric pressure plasmas have presented unique applications in biomedical fields such as in the so-called plasma medicine (LAROUSSI, 2018, METELMANN; VON WOEDTKE; WELTMANN, 2018).

There are many ways of generating non-equilibrium plasmas at atmospheric pressure. Among them, the use of asymmetric electrodes (corona discharge) and insulating barrier between the electrodes (DBDs) are the most common. Besides, pulsed applied voltage and high gas flow rates can also be employed (TENDERO et al., 2006). Mostly, at atmospheric pressure, the discharge gaps are in the range from few mm to some cm, which limit the size of objects that can be directly treated (LU; LAROUSSI; PUECH, 2012). To overcome this drawback, cold atmospheric plasmas can be generated in open space by combining some of the above-mentioned methods as in the case of atmospheric pressure plasma jets (APPJs). Thus, APPJs are plasma devices able of generating plasma plumes in open space when a gas flow is flushed through the reactor gap (NEHRA; KUMAR; DWIVEDI, 2008). Because of the non-confined discharge, APPJs have drawn much attention for the treatment of not only large and irregular materials, but also objects that cannot support vacuum conditions (BORGES et al., 2017).

APPJs can be used for treatment of heat-sensitive surfaces such as polymers. In this case, ions, electrons, photons and reactive species are transported to the material, interacting with the surface and functionalizing it (PENKOV et al., 2015, ONYSHCHENKO et al., 2015b). Plasma treatment can activate surfaces (KOSTOV et al., 2014), can be used for cleaning (GOTOH et al., 2016, JIN et al., 2013), etching (LUAN et al., 2017) or improving hydrophobic or hydrophilic properties (KOSTOV et al., 2013, MUSA et al., 2016, VAN DEYNSE et al., 2015, SHAW et al., 2016). Usually, polymers have low surface energy, which leads to poor adhesion. Therefore, APPJs have been successfully used for the surface activation of different polymers improving their adhesion properties (KOSTOV et al., 2014, ONYSHCHENKO; DE GEYTER;

MORENT, 2017). However, plasma jet treatments usually provide a punctual and precise modification, which is interesting for some applications, but disadvantageous for treatment of big surfaces. Thus, the inner surface modification of packages and shallow plastic objects are also possible with APPJ, but the treatment of long tubes can be challenging. Hence, new alternatives need to be investigated.

Another important and growing application of APPJs is in biomedical field such as for sterilization (BORGES et al., 2017, FRICKE et al., 2012a, LIN et al., 2016, MORITZ et al., 2017), wound healing (BREATHNACH et al., 2018, CHENG et al., 2018), blood coagulation (LEE et al., 2012), Dentistry (BORGES et al., 2018, YAMAZAKI et al., 2011) and cancer treatment (KIM et al., 2010, XU et al., 2018, YAN; SHERMAN; KEIDAR, 2017). It has been observed that APPJs can effectively eradicate microorganisms (DAESCHLEIN et al., 2010, NISHIME et al., 2016) and the generated reactive oxygen and nitrogen species (RONS) are pointed out as the major factor responsible for apoptotic cellular retrogression (WEISS et al., 2015). Some investigations concluded that RONS can cause DNA damage and lipid peroxidation (KIM et al., 2010, JOSHI et al., 2011). However, the success of APPJ treatments seems to derive from synergetic effect of several plasma agents, as electric field, UV radiation, energetic electrons and reactive species (KOSTOV et al., 2015a, YUSUPOV et al., 2017). The precise control of plasma treatment dose is crucial for the desired outcome. Low power plasma jets are able to stimulate cell proliferation and heal wounds, while higher power treatment can be used to induce apoptosis of cancer cells (KOSTOV et al., 2015a). As a result of their local effect, plasma jets can be used to precisely treat specific regions like tumors. In this case, the control of treatment area and depth while preserving the surrounding healthy cells can be achieved (RATOVITSKI et al., 2014). APPJs are widely studied for treatment of external tissues or surface treatment when a preliminary surgical procedure is necessary to provide access to the desired spot (BRULLÉ et al., 2012). Thus, one interesting and advantageous plasma jet specification is the development of elongated devices capable of delivering plasma to remote locations inside living bodies, like endoscopy, allowing minimal invasive procedures.

Endoscopes are flexible, slender and tubular instruments used for monitoring hollow organs for diagnostics or surgeries. An endoscope combines fiber optics for illumination, a charge-coupled device for imaging and, in many cases, a working channel to provide surgical instruments at the distal endoscope end. Combining this functionality with a medically active plasma jet would enable physicians to apply plasma and investigate its effects inside the body. For that, the plasma jet size needs to be minimized to few mm diameter and the discharge temperature should not overcome 40ºC (BINENBAUM et al., 2017). Additionally, the device must be long enough to reach the whole extension, or the plasma needs to be transported through a tube like in the case of transporting plasmas (OMRAN et al., 2017).

There are only few studies regarding the transport of plasma through long dielectric tubes. Robert and coauthors (2009) developed the first transporting plasma device called “plasma gun”. It consists of a DBD primary reactor connected to a gas-flushed capillary in which the primary discharge propagates through the entire tube extension generating a plasma plume at its end (ROBERT et al., 2009). The plasma gun was also tested for branching capillaries where T connectors were used and four simultaneous plasma plumes could be achieved (ROBERT et al., 2013). In addition, the discharge propagation through metallic tubes was feasible and the transfer duration was much shorter. Other groups also developed similar long plasma jets. Omran and coauthors (2017) investigated the characteristics of single and multi-channel transporting plasmas from a DBD-like plasma jet. The group studied the discharge propagation mechanisms and concluded that the transporting plasma comprises of discrete ionization waves originated from the HV electrode and propagate by deposition of positive charges at the tube wall creating a high potential region (OMRAN et al., 2017). Onyshchenko and coauthors (2015a) investigated the plasma plume extension when long thin polymeric tubes were placed 3 mm away from the plasma jet nozzle allowing their inner surface treatment. Kostov and coauthors (2015c) studied the propagation of a primary discharge through a long plastic tube with a floating wire inside. This metal wire is in contact with the DBD discharge, thus producing a strong electric field close to the tube end that helps generating a plasma plume at the tip without igniting plasma along the plastic tube. Another class of endoscopic plasma source consists of long plasma jets in which the HV electrode is localized close to the tip end. Examples of this plasma jets class are the RF APPJ arranged at the tip of an elongated and flexible capillary developed by Binenbaum and coauthors (2017) and the hollow core fiber plasma jet with bifilar helicoidally arranged electrodes created by Polak and coauthors (2012).

In the present work, two different elongated plasma jets based in pre-existing devices were investigated. The first, called plasma endoscope, is the miniaturization and improvement of the above-mentioned hollow core fiber plasma jet introduced by Polak and coauthors in 2012. This plasma jet was developed and characterized at the Leibniz Institute for Plasma Science and Technology (INP) in Greifswald, Germany. It consists of a millimeter dimension plasma jet with helicoidal electrode arrangement that can be adapted inside commercial endoscopes. The use of a shielding gas around the main noble gas stream is proved to be crucial for in-body application. The efficiency of different feed and shielding gases are investigated, where the use of carbon dioxide as shielding gas helps reducing formation of parasitic discharges within the tube. Once the development of this plasma source aims at its operation inside endoscopes, it must fulfill some requirements that are explored throughout the text. The second extended plasma jet studied here consists of a primary DBD reactor connected to a long dielectric tube with an inserted floating metal wire in its axis. It was firstly developed at the São Paulo State University (Unesp), Guaratinguetá, Brazil by Kostov and coauthors (2015c).This jet device generates a remote plasma plume that is far away (~ 1.0 m) from the HV source, in this way allowing safe manipulation of the plasma jet and facilitating plasma application to places of difficult access. In this study, different excitation systems are applied for generating the primary DBD discharge and the role of the primary reactor configuration is investigated. Additionally, applications concerning its microbial efficiency and material surface modification using tilting positions are also explored.

5 CONCLUSION

The development of remote plasma jets is a recent and important step in the plasma jets research field. It allows a better manipulation of the plasma plume improving the sample targeting by plasma species and opening new possibilities in many areas of applications such as endoscopy, odontology and material treatment. However, developing a long plasma jet or a remotely generating device brings difficulties that were explored throughout this study. The development, improvement and characterization of two elongated cold plasma jets have been carried out here first. Aiming a new and promising combination of plasma and an endoscope device, a plasma jet configuration was successfully developed. The device was tested for fulfilling the specific requirements necessary for small configuration dimension, in-body operation and safety operation conditions. Additionally, the possibility of transferring plasma through long plastic tubes by using an inner floating wire was also investigated in the present work. Obstacles concerning the construction of a remotely generated plasma jet for safe *in vivo* treatments were surpassed, until a secure plasma jet operation with low cytotoxicity was obtained.

The first plasma jet, plasma endoscope, was designed to operate inside commercial endoscopes allowing simultaneous diagnosis and treatment. Thus, this plasma jet can be applied for in-body treatments inside hollow organs or regions of difficult access.

Since the plasma endoscope is a long plasma jet device designed for endoscopic applications, it presented specific drawbacks. In this case, the high voltage wire is winded around the tube until the nozzle tip. The wire winding distance is a very important parameter and needs to be made very narrow to avoid formation of parasitic discharges inside the tube. This atmospheric pressure plasma jet can produce small plasma plumes with temperature in the range of 30ºC and maximum discharge power of 160 mW. The working temperature and the discharge mean power can be directly controlled by the input voltage. To ensure its proper operation inside closed cavities, a second shielding gas channel coaxially aligned with the main stream gas tube was arranged. An electronegative gas curtain is important to confine the discharge electrons into the main column avoiding dispersion. Carbon dioxide appeared to be a suitable alternative to air, since its breakdown voltage is higher and no parasitic discharges were observed inside the endoscope tube. The plasma jet worked evenly with helium and neon, however the neon jet ignited at lower input voltages and provided a wider range of power operation.

A comparison of emission spectra obtained with and without CO_2 shielding gas led to the conclusion that the neon jet excitation processes were less affected than helium jet when using the shielding curtain. When operated inside half-closed cavities, the plasma jet can switch between local (plasma plume) and diffuse (glow discharge) modes by controlling the shielding gas flow rate. It was proven by Winter and coauthors (2019) that the use of a HV generator operating in burst mode helps reducing the leakage current of this plasma jet. Using the new power supply, the neon plasma jet proved to be efficient in decontaminating the bacteria *P. aeruginosa*. Thus, the plasma endoscope operating with neon as feed gas and CO_2 as shielding gas connected to the new HV power supply forms a promising combination for operating inside an endoscope and shall be further investigated for future in-body application.

The second source, the long tube plasma jet, is a device capable of generating a remote helium plasma jet by transporting the discharge from a primary reactor to the tube far end. Since it transfers the discharge through a flexible plastic tube, the plasma jet can be easily and safely applied in fields like dermatology, dentistry and material treatment.

When operated with a simple AC power supply, the device produces plasma plumes with low power (below 1.0 W) but with temperatures close to the biological upper limit of 43ºC when operating in grounded conditions. The power can be adjusted by changing either the gas flow rate or the distance between tube end and target. The temperature analysis of the floating plasma plume revealed a donut-shaped temperature distribution along the plasma jet. This singular temperature profile shape can be possibly associated to the annular electron's distributions in the plasma jet. However, further investigations about the electron's density distribution and optical emission analysis with improved spatial resolution should be carried out for better understanding of this phenomenon.

Operating the long tube plasma jet in burst mode allowed the precise adjustment of parameters enabling it to work in special regions with broader range of operation. Thus, the plasma jet power can be better controlled which leads to a more precise management of treatment dose. The role of primary discharge geometry was also investigated and it has been demonstrated that only the penetration distance of the floating metal wire inside the discharge can slightly influence the discharge power.

The long plastic tube device can be adapted to other plasma jet configurations efficiently transferring the discharge to the tube tip end. It was successfully adapted to a pre-existing plasma jet improving its manipulation and allowing a safer use regarding biological applications. The long tube jet was tested against *C. albicans* in agar plates and the area of inhibition zones was used to evaluate its decontamination efficiency. The size of the inhibition zones depends on the treatment dose, which can be precisely controlled by increasing either the treatment time or the discharge power. Its maximal size is determined by the lifetime of generated species. Since the observed killing zones were much bigger than the tube diameter, it can be concluded that the long-living ROS are the most important agents for decontamination. By means of optical emission spectroscopy and the decontamination efficiency tests, it was proved that the plastic tube elongation acted as a gas channel in which the plasma is only transferred to the other extremity exhibiting similar characteristics to the primary discharge. Thus, the long tube acts as a safe extension device that can be easily bent and manipulated close to the target allowing treatment in regions with difficult access.

Since this tube extension allows fine control of the plasma plume, the treatment of surfaces with tilting positions is made possible. Thus, the effects when the plasma jet is not applied in the conventional perpendicular manner was investigated. The long tube plasma jet has been tested for surface modification of PET with different angling positions. Tilting the plasma jet increased up to 30% the treated areas and more pronounced and homogeneous modified areas were achieved when a grounded platform was used below the target polymer. Additionally, the use of laminar flow conditions also cooperated with improving homogeneity. Thus, the small diameter, the flexibility, the tube biocompatibility and the operation safety (distant from the HV electrode) broadens the field of applications of this plasma jet, making possible a secure treatment of living tissues, in-body procedures or treatment of complex-shaped targets.

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PUBLISHED PAPERS

The work presented in this Ph.D. thesis led to publication of the following papers:

[1] WINTER, J.; NISHIME, T. M. C.; BANSEMER, R.; BALANZINSKI, M.; WENDE, K.; WELTMANN, K-D. Enhanced atmospheric pressure plasma jet setup for endoscopic applications**. Journal of Physics D:** applied physics, v. 52, n. 2, 2019.

[2] WINTER, J.; NISHIME, T. M. C.; GLITSCH, S.; LÜHDER, H.; WELTMANN, K-D. On the development of a deployable cold plasma endoscope. **Contributions to Plasma Physics**, v. 58, n. 5, 2018.

[3] BORGES, A. C.; LIMA, G. M. G.; NISHIME, T. M. C.; GONTIJO, A. V. L.; KOSTOV, K. G.; KOGA-ITO, C. Y. Amplitude-modulated cold atmospheric pressure plasma jet for treatment of oral candidiasis: *in vivo* study. **PLoS ONE**, v. 13, n. 6, p. 1-19, 2018.

[4] KOSTOV, K G; BORGES, A C; KOGA-ITO, C Y; NISHIME, T. M. C.; PRYSIAZHNYI, V.; HONDA, R. Y. Inactivation of *Candida albicans* by cold atmospheric pressure plasma jet. **IEEE Transactions on Plasma Science**, v. 43, n. 3, p. 770-775, 2015.

[5] KOSTOV, K. G.; NISHIME, T. M. C.; MACHIDA, M.; BORGES, A. C.; PRYSIAZHNYI, V.; KOGA-ITO, C. Y. Study of cold atmospheric plasma jet at the end of flexible plastic tube for microbial decontamination. **Plasma Processes and Polymers**, v. 12, n. 12, 2015.