Caracterização Elétrica e Otimização de Jato de Plasma Frio Operando em Pressão Atmosférica para Aplicações Biomédicas

Relatório Científico (projeto # 3182): 01/10/2020 a 31/05/2024

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Resumo

O projeto de pesquisa visa estudar jatos de plasma frio em pressão atmosférica usando diferentes configurações geométricas e condições de trabalho. O bolsista vai desenvolver e testar um diagnóstico elétrico detalhado para fonte de tensão pulsada. Ele vai aperfeiçoar o dispositivo visando aplicações biológicas *in vivo* para gases diferentes. Através de Espectroscopia Ótica de Emissão do plasma será possível identificar as espécies excitadas e estimar as suas temperaturas (rotacional, vibracional e eletrônica). A intenção do estudo é otimizar o dispositivo para aplicações biológicas *in vivo* visando a produção de mais espécies reativas com forte efeito microbiano (tais como O, NO, NOx, OH etc.) e com menores temperaturas para evitar possíveis queimaduras. Como resultado final do projeto é esperado desenvolver e testar um dispositivo de jato de plasma portátil e de fácil operação que será adequado para aplicações médicas e odontológicas.

1 – Realizações no período

1.1 – Estudo das propriedades dos jatos de plasma DBD usando o eletrodo energizado com e sem contato com o plasma

Neste estudo foi realizada uma investigação experimental comparando as propriedades de jatos de plasma em configurações de descarga por barreira dielétrica (DBD) usando o eletrodo energizado com barreira dielétrica e sem barreira dielétrica, mantendo uma segunda barreira dielétrica sobre o eletrodo aterrado. Para tanto, foram utilizadas duas fontes de tensão diferentes para produzir os jatos de plasma, sendo que uma delas fornece alta-tensão na forma pulsada e a outra alta-tensão com forma de onda do tipo seno amortecido, que atua como uma fonte de tensão pulsada. Medições dos parâmetros do plasma foram realizadas para ambas as configurações utilizando argônio e hélio como gases de trabalho. Como resultado, se for utilizada a fonte de energia pulsada, foram encontradas diferenças significativas na potência da descarga (P_{dis}) e nas temperaturas rotacional e vibracional (T_r e T_v, respectivamente) ao se alternar entre as configurações com o eletrodo energizado com e sem barreira dielétrica, como pode ser visto na Tabela 1. Por outro lado, utilizando a fonte de tensão com forma de onda do tipo seno amortecido, apenas P_{dis} apresentou diferenças significativas na troca da configuração do eletrodo, como pode ser visto na Tabela 2. Para a fonte pulsada, observou-se que apesar do notável aumento de P_{dis} ao passar da configuração de barreira dupla para a de barreira simples, os valores obtidos para T_r e T_v também aumentaram, mas não na mesma proporção que o aumento de P_{dis}, o que sugere uma dependência não linear entre temperaturas e potência de descarga no jato de plasma. Como exemplo de aplicação de plasmas em ambas as configurações, foram realizados testes na tentativa de remover filmes de cobre depositados em substratos de alumina e, como resultado, houve remoção significativa de material apenas quando o eletrodo alimentado esteve em contato com o plasma.

Tabela 1: Resumo de resultados obtidos com fonte de tensão pulsada.

Parameter	T_r **(K)**		T_v **(K)**		P_{dis} **(W)**	
Working gas	*Ar*	*He*	*Ar*	*He*	*Ar*	*He*
Double bar.	550±36	300±24	2300±145	3100±238	0.62	0.64
Single bar.	600±33	350±21	2700±139	3500±199	2.98	3.29

Tabela 2: Resumo de resultados obtidos com alta-tensão do tipo seno amortecido.

Parameter	T_r **(K)**		T_v **(K)**		P_{dis} **(W)**	
Working gas	*Ar*	*He*	*Ar*	*He*	*Ar*	*He*
Double bar.	500±56	325±73	1900±217	3100±549	0.27	0.23
Single bar.	500±88	400±62	1900±337	3700±573	0.49	0.48

Este estudo foi importante para o projeto por ser o primeiro utilizando uma fonte de tensão de baixo custo (a que fornece alta-tensão com forma de onda do tipo seno amortecido). Fonte esta que se tornou a base do dispositivo portátil para aplicações em medicina e odontologia (maiores detalhes nas seções 1.4 e 1.5).

Os resultados obtidos neste estudo foram publicados na revista *IEEE Transactions on Plasma Science* (vide detalhes na seção 4).

1.2 – Estudo do efeito de adição de O_2 nas propriedades do jato de plasma

A adição de pequenas quantidades de O_2 no gás de trabalho utilizado para produzir os jatos de plasma (He ou Ar) influência bastante a composição de espécies reativas gerados pelo plasma, introduzindo mais espécies de oxigênio, que são conhecidas por seu forte efeito antimicrobiano [1]–[4]. Entretanto adicionando oxigênio demais ao gás de trabalho ocorre a redução da sua potência, o que eventualmente pode levar à extinção da descarga. Então, existe uma quantidade ótima de oxigênio que pode ser adicionada ao gás de trabalho, o que geralmente depende de tipo de reator e da tensão de excitação utilizada e pode ser determinada experimentalmente. Nossas medidas usando uma tensão AC com modulação de amplitude (que está sendo usada também nos tratamentos biológicos) mostraram que a melhor condição para tratamentos é adicionar entre 1 e 2% de oxigênio na ao gás de trabalho. Um exemplo da variação de parâmetros dos jatos de plasma em função da proporção de oxigênio molecular adicionada ao gás de trabalho (hélio) é mostrado na figura 1, onde podemos notar que tanto os valores de potência (1a) quanto os de corrente eficaz (1b) diminuem com a adição de O_2 , que as temperaturas rotacional e vibracional (1c e 1d) permanecem aproximadamente constantes e que a intensidade das emissões de oxigênio atômico (1e), medidas para a linha com comprimento de onda igual a 777 nm, tem valor máximo com 2% de O_2 adicional. Com isso são produzidos no plasma uma maior quantidade de espécies reativas de oxigênio com uma potência considerável e sem prejudicar a descarga, sendo assim possível diminuir o tempo de aplicação de plasma durante os tratamentos biológicos.

Além da produção de uma maior quantidade de espécies reativas de oxigênio no plasma com a adição de O_2 ao gás de trabalho, foi analisado um aspecto ainda não explorado na literatura, que é o comportamento das formas de onda da descarga formada a partir de tensão AC com modulação de amplitude em função da proporção de O_2 adicional. A partir de tal análise, foi observado um fenômeno ainda não reportado na literatura: a redução na duração da descarga e atraso na ignição do plasma.

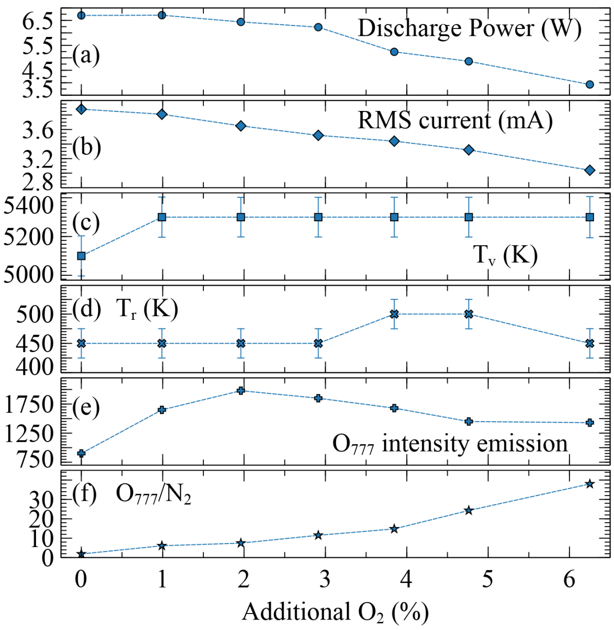

Figura 1: Curvas de parâmetros do jato de plasma em função da proporção de oxigênio molecular adicionada ao gás de trabalho hélio: (a) potência, (b) corrente elétrica eficaz, (c) temperatura vibracional, (d) temperatura rotacional, (e) intensidade total de linha emissão de oxigênio atômico com comprimento de onda em 777 nm e (f) intensidade relativa de emissões de oxigênio atômico comparadas à emissões de nitrogênio molecular.
Fonte: próprio autor.

Os resultados obtidos neste estudo foram publicados na revista *Applied Sciences* (vide detalhes na seção 4).

1.3 – Desenvolvimento de configuração alternativa para gerar jatos de plasma frios na extremidade de tubos longos e flexíveis

A geração de jatos de plasma frios na extremidade de tubos longos e flexíveis já vinha sendo feita no Lab. de Plasmas da FEG/UNESP há bastante tempo e sendo utilizada no Lab. Genoma há alguns anos [5]. A tecnologia utilizada para gerar jatos de plasma em tais condições é baseada em uma técnica chamada de transferência de jato de plasma [5], que consiste em colocar um fio condutor no interior do tubo longo e conectar o conjunto a um reator que produz plasma por descarga de barreira dielétrica (DBD), de modo que o fio condutor atue como um eletrodo flutuante. Assim, quando se forma um plasma no interior do reator (descarga primária, na figura 2), o condutor no interior do tubo de plástico é carregado criando um forte campo elétrico na outra extremidade do fio, o que é suficiente para iniciar uma descarga secundária, que é o próprio jato de plasma remoto.

Durante os trabalhos de pós-doutoramento propusemos uma configuração alternativa. Trata-se de um novo conceito para geração de jatos de plasma na extremidade de um tubo longo utilizando a técnica de transferência de jatos de plasma. A nova configuração consiste em um arranjo coaxial de dois tubos poliméricos com uma malha metálica entre eles. O material do tubo externo e interno são Nylon e PTFE, respectivamente.

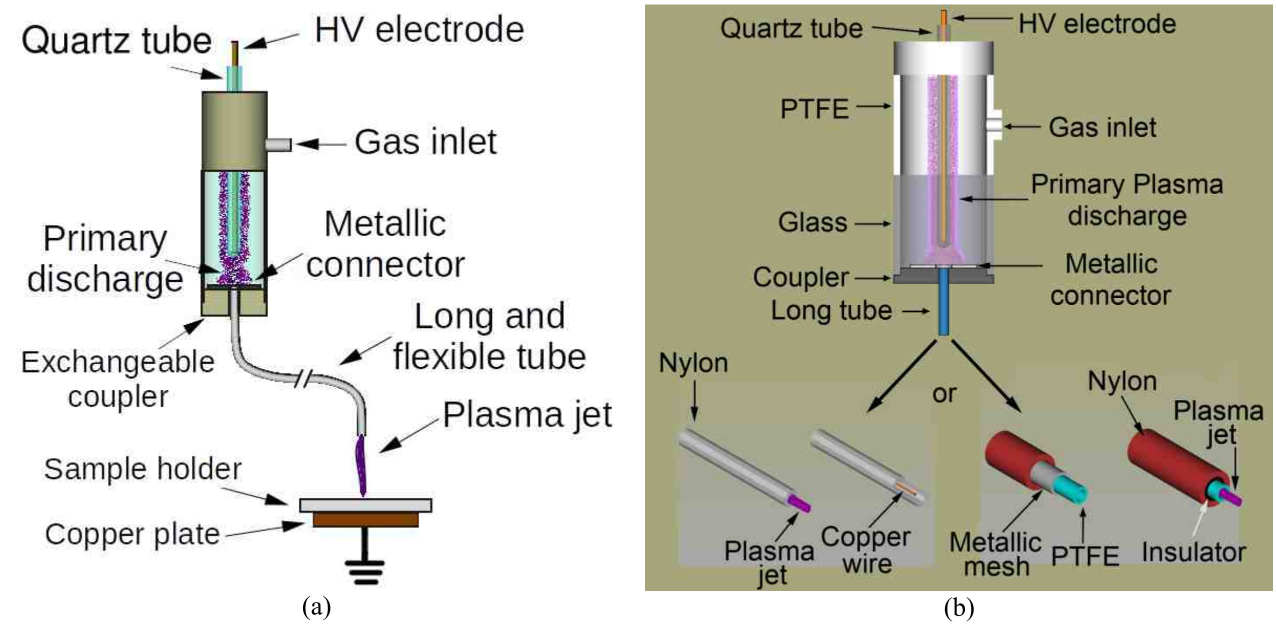

Figura 2: Visão geral do sistema para gerar jatos de plasma na extremidade de um tubo longo a partir da descarga primária de um reator DBD. (b) Detalhes das configurações com fio e malha. Fonte: próprio autor.

Utilizando a configuração de jato longo com malha foi possível baixar as temperaturas tanto do plasma com hélio quanto com argônio. Sendo que este último era um dos objetivos do projeto de pós-doutorado atual. Além da redução nas temperaturas dos jatos de plasma, na configuração com a malha também foram medidas correntes elétricas (i_{RMS}) menores do que na configuração com fio.

Além disso, foram realizados estudos comparando os resultados da aplicação em polímeros usando os jatos de tubo longo nas configurações que usam fio e malha. Um artigo com parte dos resultados, intitulado "*Different configurations for transferred atmospheric pressure plasma jets and their applications to polymer treatment*", foi publicado na revista *Contributions to Plasma Physics* (vide detalhes na seção 4).

Os estudos com esta nova configuração forneceram dados para um segundo artigo, intitulado " *Different Radial Modification Profiles Observed on APPJ-treated Polypropylene Surfaces According to the Distance Between Plasma Outlet and Target*", o qual foi publicado na revista *Polymers* (detalhes na seção 4).

1.4 – Estudo de um jato de plasma de pequeno porte, que é simples de operação, poderá ser facilmente transportado e designado para futuras aplicações em ambulatório

A figura 3 mostra fotos do dispositivo compacto e portátil, na versão de tubo longo, que vem sendo desenvolvido desde o início do projeto. Na figura 3a, temos a visão geral do dispositivo, onde destacamos: 1- fonte de tensão portátil e compacta, 2- reator conectado à fonte de tensão e 3- tubo longo e flexível de onde se extrai o jato de plasma. Já a figura 3b mostra o jato de plasma sendo aplicado a uma cultura de micro-organismos.

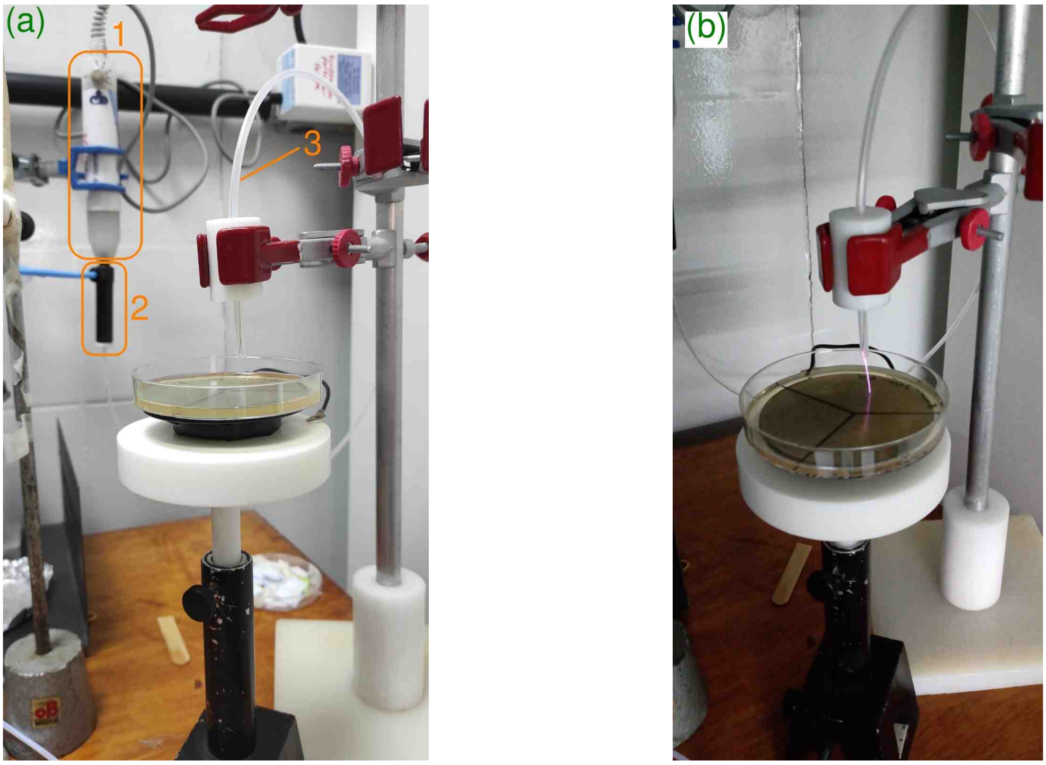

Figura 3: Fotos do dispositivo compacto e portátil para gerar jatos de plasma, na configuração de tubo longo e flexível: (a) visão geral, (b) teste *in vitro* em cultura de micro-organismos. Fonte: próprio autor.

Após os testes realizados com micro-organismos *in vitro*, foram realizados ensaios para avaliar a citotoxicidade celular do plasma produzido com este equipamento e, em seguida, os testes *i n vivo*. De um modo geral, os testes de citotoxicidade celular se mostraram satisfatórios. Sendo que no caso típico de aplicação que vinha sendo adotado como padrão nos tratamentos com plasma no Laboratório Genoma em experimentos anteriores (distância de 1,5 cm, tempo de exposição de 5 minutos, utilizando gás hélio a 2,0 l/min), mas com potência de descarga de apenas ~300 mW, a viabilidade celular aferida no teste de citotoxicidade foi superior a 70%. Em relação aos estudos *in vivo*, resultados preliminares indicam que a redução na área da lesão da mucosa, avaliada 14 dias após os ensaios, é significativamente maior nos indivíduos expostos ao plasma quando comparados com o grupo de controle.

Os detalhes sobre os ensaios *in vivo* são apresentados na tese de doutorado da Dra. Aline Sampaio, cuja versão final está para ser depositada no repositório institucional da Unesp. Estes estudos estão sendo mencionados aqui por serem importantes para a parcela da equipe de pesquisadores que é responsável pelo desenvolvimento de dispositivos de plasma. Pois, deste modo, podemos cruzar as informações de desempenho da aplicação (resultados *in vitro*/*vivo* e citotoxicidade) com as propriedades do jato de plasma para adequar os parâmetros de operação de modo a obter um dispositivo eficiente e seguro.

Além da colaboração da Dra. Aline Sampaio, os testes com o dispositivo portátil para gerar jatos de plasma para inativação de micro-organismos e ensaios *in vivo* realizados no Lab. Genoma contaram com a colaboração da pós-doutoranda Noala Vicensoto Moreira Milhan.

Parte dos resultados dos testes *in vitro* e de citotoxicidade, assim como os resultados de caracterização da fonte portátil de plasma (detalhes na seção 1.5, a seguir), entraram num artigo publicado na revista *IEEE Transactions on Radiation and Plasma Medical Sciences* (detalhes na seção 4).

1.5 – Colaboração com o INP-Greifswald/Alemanha

Como parte dos experimentos previstos no meu estágio de pesquisa no exterior, no INP-Greifswald/Alemanha, foram realizadas caracterizações do jato de plasma produzido com a fonte portátil desenvolvida na FEG. O principal objetivo dos experimentos era verificar se o plasma produzido pelo nosso dispositivo está de acordo com normas de segurança necessárias para que este possa ser utilizado como um dispositivo para aplicações médicas, ou seja, se está dentro das especificações estabelecidas pela norma DIN 91315, que é uma norma alemã, baseada em parte na DIN EN 60601-1, que se relaciona com a IEC 60601-1 e trata dos limites de segurança de fontes de plasma para aplicações médicas [6]–[8].

A norma DIN 91315 (a ser referida como DINSpec daqui em diante) consiste em caracterizar a fonte de plasma de modo a:

- verificar a radiação UV efetiva emitida pelo plasma → definirá o tempo máximo de exposição ao plasma
- medir a temperatura do gás (T_{gas}) e do alvo (T_{targ}, uma placa de cobre) → não é recomendável que o valor máximo de T_{gas} exceda 40 °C e o valor máximo de T_{targ} deve ser inferior a 40 °C
- verificar os valores de corrente elétrica que chega ao paciente (PLC para abreviação do termo em inglês *patient leakage current*) → não deve exceder 100 μA-AC
- aferir a geração dos gases O_3 e NO_x pelo plasma → definirá o tempo máximo de exposição ao plasma
- verificar de eficácia antimicrobiana, toxicidade celular, etc. (etapa realizada no Lab. Genoma, no Brasil)

Em todas as medições realizadas para caracterizar o plasma de acordo com a norma DINSpec foi utilizado um fluxo de gás de 2.0 l/min, valor este que foi adotado como padrão nos experimentos realizados no Lab. Genoma, pois esta foi a vazão que proporcionou melhores resultados nos ensaios biológicos realizados ao longo dos últimos anos. As medições dos parâmetros necessários para caracterizar a fonte de plasma foram feitas para dois gases de trabalho: argônio (Ar) e hélio (He). Foram utilizadas duas condições de potência, uma nomeada como baixa potência, que corresponde à tensão de pico de 9.0 kV, o menor valor utilizado nos experimentos no Brasil, e outra nomeada como alta potência, cuja tensão de pico é de 13,3 kV, o maior valor nos experimentos no Lab. Genoma.

Os resultados dos trabalhos de caracterização da fonte portátil de plasma, realizados no INP-Greifswald/Alemanha, podem ser resumidos da seguinte forma:

i. A radiação UV emitida pelo plasma gerado com o dispositivo portátil está em níveis baixos, mesmo na condição de potência mais alta → o jato de plasma pode ser aplicado em tecidos humanos por longos períodos
ii. Os valores de temperatura do gás são baixos (T_{gas} < 40 °C) → não há riscos de danos a estruturas celulares associadas a temperatura do gás
iii. Os valores da corrente que chega ao paciente (PLC) estão, de um modo geral, dentro do limite estabelecido pela norma DINSpec. Porém, em algumas distâncias de operação (16 mm < d <= 260 mm) os valores de PLC estiveram ligeiramente acima do limite de segurança (100 µA) para o dispositivo é operando em alta potência → isto deverá ser contornado com ajustes nos valores ou na forma de onda da tensão aplicada para gerar plasma
iv. As emissões de gases nocivos (O_3 e NO_2) durante a operação do jato de plasma ficaram abaixo dos valores de referência quando o He é o gás de trabalho e para 14 mm de distância entre a saída do plasma e o alvo → isso ainda pode ser melhorado com o ajuste de tensão proposto em iii
v. As emissões de O_3 e NO_2 estão acima dos valores de referência em todas as outras condições → em um primeiro momento, isso apenas limitará o tempo de tratamento. Porém, este efeito pode ser mitigado se adicionarmos um sistema de sucção de gás ao dispositivo. Deste modo, o excesso de gases nocivos produzido seria removido da região onde o plasma está sendo aplicado

Com base nos resultados obtidos podemos concluir que estamos no caminho certo para obter um protótipo de fonte de plasma para aplicações odontológicas e médicas.

Os resultados obtidos com a caracterização da fonte portátil de plasma no INP-Greifswald/Alemanha, junto a alguns dados obtidos no GREMI-Orléans (vide próxima subseção) e no Lab. Genoma, deram origem a um manuscrito submetido à revista *IEEE Transactions on Radiation and Plasma Medical Sciences* (vide detalhes na seção 5). Antes disso, um trabalho com parte dos dados obtidos no INP-Greifswald já haviam sido publicados na revista *Physica Scripta* (detalhes na seção 4). Um estudo aprofundado sobre as descobertas publicadas na revista *Physica*

Scripta está sendo realizado no INP-Greifswald pelo estudante de doutorado Bruno Honorat, sob supervisão do Dr. Torsten Gerling e com a colaboração do nosso grupo de pesquisas. Um manuscrito está em preparação e deverá ser submetido para consideração para publicação em breve (detalhes na seção 5).

1.6 – Colaboração com o grupo de pesquisa GREMI (*Groupe de Recherches sur l'Energétique des Milieux Ionisés*), da Universidade de Orléans – França

Em anos anteriores, os grupos de pesquisa do GREMI/França e da FEG/Brasil iniciaram uma colaboração científica para realizar algumas investigações sobre plasmas de pressão atmosférica (APPs) produzidos por diferentes dispositivos. Essa colaboração resultou em uma visita técnica ao GREMI, durante o período de vigência do estágio de pesquisa no exterior (entre 17/10/2022 e 28/10/2022). Tal visita técnica, apesar de curta foi extremamente produtiva, pois foram coletados dados experimentais suficientes para serem analisados após o retorno ao Brasil. Foram obtidos dados em experimentos com duas fontes de plasma diferentes: (1) o dispositivo portátil que está sendo desenvolvido na FEG dentro do projeto temático (processo nº 2019/05856-7) e (2) um dispositivo na forma de funil para desinfecção de superfícies, cujos estudos iniciais se deram também na FEG. Boa parte dos dados medidos para o dispositivo 2 durante a visita técnica ao GREMI entraram em um artigo científico publicado em outubro de 2023, na revista *Plasma Chemistry Plasma Processing* e alguns dos dados obtidos para o dispositivo 1 entraram no artigo submetido para a revista *IEEE Transactions on Radiation and Plasma Medical Sciences* (vide detalhes na seção 4).

1.7 – Aplicação de jatos de plasma para a inativação de biofilmes criados no interior de tubos T traqueais

Além da aplicação em odontologia, o projeto temático FAPESP 2019/05856-7 incluiu em seus objetivos um novo desafio envolvendo o uso de jatos de plasma em pressão atmosférica (APPJs) para tratamento de implantes traqueais. Esta foi uma necessidade urgente que surgiu devido à pandemia de COVID-19, que resultou na intubação de muitos pacientes hospitalizados, muitos deles necessitando de um implante traqueal. Este tipo de implante está sujeito à colonização por fungos e bactérias, o que pode eventualmente levá-lo à falha. Este problema pode ser remediado pelo tratamento com plasma da parede interna do tubo T, evitando a formação de biofilme e aumentando a vida útil do implante.

Ensaios para inativação de tais biofilmes vêm sendo conduzidos pelo estudante de doutorado Diego Morais Da Silva no Lab. Genoma, sob a orientação da Profa. Dra. Cristiane Yumi Koga-Ito. Atualmente, os testes têm sido efetuados com a fonte de plasma tradicional. Os resultados obtidos até o momento indicam que o jato de plasma tem ação efetiva sob biofilmes multi-espécies. Minha colaboração nos experimentos está relacionada à caracterização dos jatos de plasma e ao design dos experimentos. Um artigo com parte dos resultados obtidos nos experimentos foi elaborado e submetido para publicação pelo Sr. Diego.

Os resultados deste estudo foram publicados na revista *Microorganisms* (vide detalhes na seção 4).

1.8 – Otimização da produção de espécies reativas em jatos de plasma utilizando He como gás de trabalho

Experimentos preliminares realizados no Lab. de Plasmas da FEG/UNESP, mostraram que a produção de espécies reativas em jatos de plasma varia com a distância **d** entre a saída do plasma e o alvo. Uma foto do jato de plasma e resultados da produção de espécies reativas e parâmetros elétricos são mostrados na figura 4. Apesar desta informação já estar disponível na literatura, medições comparando as emissões de espécies reativa na saída do plasma (região 1, na figura 4) com as emissões medidas na superfície do alvo (região 2) ainda não haviam sido realizadas. Além disso, os trabalhos publicados não reportam o comportamento dos demais parâmetros de plasma em função de **d**.

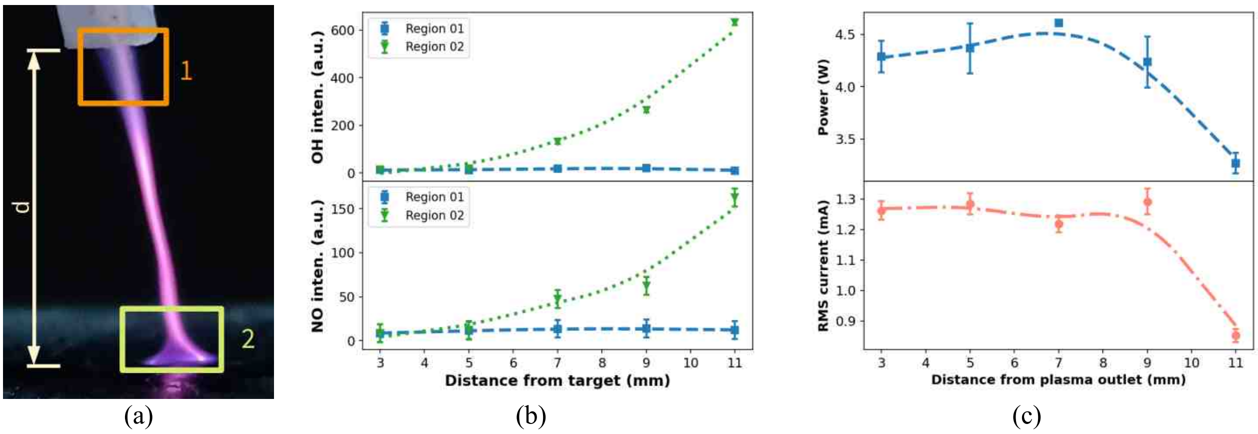

Figura 4: (a) Foto do jato de plasma destacando regiões onde foram feitas medições de espectroscopia (1 e 2). (b) Produção de espécies reativas OH e NO em função de **d** nas regiões 1 e 2. (c) Potência e corrente RMS em função de **d** Fonte: próprio autor.

Os resultados preliminares destes experimentos foram apresentados no congresso CBrAVIC 2021.

1.9 – Estudo da mistura argônio/hidrogênio nas propriedades dos jatos de plasma

Uma das propostas deste projeto de pós-doutorado era produzir jatos de plasma utilizando o gás argônio, porém com temperatura de gás da ordem da temperatura ambiente. Este objetivo foi alcançado com a utilização do dispositivo portátil reportado na seção 1.5, conforme pode ser visto na figura 5. No entanto, apesar do sucesso em produzir jatos de plasma à temperatura ambiente com gás argônio, os valores de corrente elétrica da descarga obtida com este gás são muito elevados quando comparados aos valores obtidos com o gás hélio. Em especial, a corrente elétrica que chega ao paciente (PLC para abreviação do termo em inglês *patient leakage current*) é aproximadamente três vezes maior com argônio do que com hélio, como pode ser visto na figura 6.

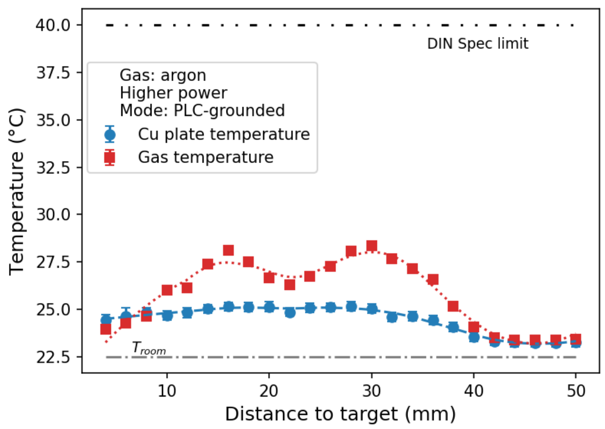

Figura 5: Curvas de temperatura do gás e do alvo (uma placa de cobre) em função da distância ao alvo para jato de plasma produzido com o dispositivo portátil e operando com gás argônio. A temperatura limite para aplicação em humanos é de 40 °C. A temperatura ambiente era ~22,5 °C. Fonte: próprio autor.

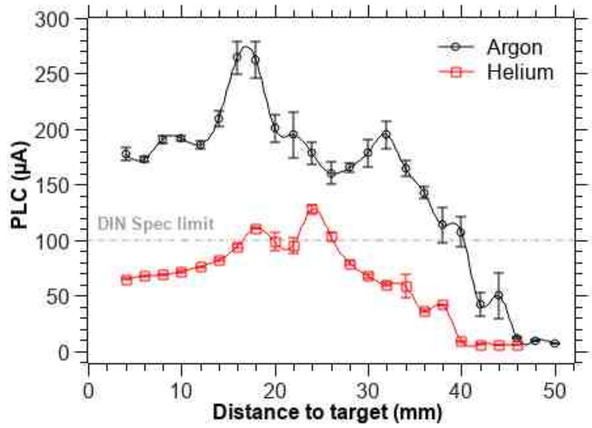

Figura 6: Valores de PLC para argônio (Ar) e hélio (He) em função da distância ao alvo obtidas com o dispositivo portátil. Fonte: próprio autor.

Em um trabalho publicado por Wu *et al* [9], os autores reportaram uma transição no regime de descarga de um jato de plasma utilizando argônio, de filamentar para difuso, quando havia um percentual acima de 3% de hidrogênio adicionado ao argônio. Descargas difusas normalmente são associadas a menores valores de corrente elétrica em jatos de plasma. Com base nestas informações, estudos com a mistura argônio/hidrogênio, na proporção 96,5%(Ar)/3,5%(H_2) foram iniciados. Em resultados preliminares, foi observado que, para o dispositivo portátil com a configuração de fio, não houve mudança no regime de descarga. Ou seja, a descarga se manteve filamentar tanto utilizando argônio puro quanto a mistura Ar/H_2.

Entretanto, ao utilizar o dispositivo portátil na configuração de malha, foi verificada uma tendência de transição para descarga difusa. Com base nestes resultados, mais estudos serão realizados de modo a entender quais são as condições necessárias para que a descarga seja sempre difusa com a mistura Ar/H_2.

1.10 – Coorientação de estudantes de mestrado e doutorado

Desde o início das atividades de pós-doutorado na FEG venho coorientando os estudantes de doutorado Kleber A. Petroski e Ananias A. Barbosa. No ano de 2023, iniciei a coorientação da estudante de doutorado Ana C. P. L. Almeida, a qual concluiu o mestrado em novembro de 2022 sob minha coorientação.

Documentos comprobatórios de coorientação de alunos estão nos anexos 1a e 1b.

1.11 – Estágio em docência – pós-doutorado

No período de 22/05/2023 até 02/02/2024 participei do programa de estágio docente da UNESP, lecionando a disciplina Física Experimental I, oferecida pelo Departamento de Física da Faculdade de Engenharia e Ciências de Guaratinguetá.

O documento comprobatório de atividades de formação/aprimoramento em docência está no anexo 2.

Referências

[1] Y. S. Seo, A.-A. H. Mohamed, K. C. Woo, H. W. Lee, J. K. Lee, and K. T. Kim, "Comparative Studies of Atmospheric Pressure Plasma Characteristics Between He and Ar Working Gases for Sterilization," *IEEE Trans. Plasma Sci.*, vol. 38, no. 10, pp. 2954–2962, Oct. 2010, doi: 10.1109/TPS.2010.2058870.

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[9] S. Wu, X. Lu, D. Zou, and Y. Pan, "Effects of H2 on Ar plasma jet: From filamentary to diffuse discharge mode," *J. Appl. Phys.*, vol. 114, no. 4, p. 043301, Jul. 2013, doi: 10.1063/1.4816318.

2 – Plano de Gestão de Dados

Os dados produzidos neste projeto (que é um subprojeto do projeto temático) estão sendo geridos da mesma forma que os dados do projeto principal (temático), ou seja, as planilhas e imagens obtidas, com seus metadados, estão sendo salvos no Repositório da UNESP (https://repositorio.unesp.br/) pela equipe da Profa. Dra. Cristiane Yumi Koga Ito.

3 – Participação em eventos científicos nacionais e internacionais

3.1 – Eventos internacionais

No período a que se refere este relatório apresentei o seguinte trabalho em evento internacional:

– Fellype do Nascimento, Kleber Petroski, Ananias Barbosa, Ana Almeida e Konstantin Kostov, "*Changes on Discharge Parameters and Production of Reactive Species in Argon and Helium Plasma Jets with Oxygen Addition*" – trabalho apresentado em forma oral/vídeo no *8th International Conference on Plasma Medicine* – ICPM8, realizado de forma *online* entre 03 e 06 de agosto de 2021.

E fui coautor nos seguintes trabalhos:

– Konstantin G. Kostov, Fellype Nascimento, Thalita M. C. Nishime, Gabriela M. G. Lima, Marcia H. Tanaka, and Cristiane Y. Koga-Ito, "*Development of Cold Atmospheric Pressure Plasma Jets for Biomedical e Dental Applications*" – trabalho apresentado pelo Prof. Dr. Konstantin Georgiev Kostov em forma oral/vídeo no *8th International Conference on Plasma Medicine* – ICPM8, realizado de forma *online* entre 03 e 06 de agosto de 2021.

– Konstantin G. Kostov, Fellype Nascimento, Kleber A. Petroski, Ana-Carla P. L. Almeida, Thalita M. C. Nishime, Gabriela M. G. Lima, Noala V. M. Milhan, Lady D. P. Leite, Maria A. C. O. Campos, Cristiane Y. Koga-Ito, "*Development of remote atmospheric plasma jets for biomedical applications*" – trabalho apresentado pelo Prof. Dr. Konstantin Georgiev Kostov, na forma de **palestra convidada** no *9th International Conference on Plasma Medicine* – ICPM9, realizado de forma presencial entre 27 de junho e 01 de julho de 2022, em Utrecht/Holanda.

– F. do NASCIMENTO, A. STANCAMPIANO, K. TREBULOVA, K.G. KOSTOV, S. DOZIAS, J.M. POUVESLE e E. ROBERT, "*Plasma electrode DBD for low power, large surface applications*" – trabalho apresentado pelo Dr. Eric Robert, na forma de **palestra convidada**, na *International Conference on Phenomena in Ionized Gases* – ICPIG XXXV, realizado de forma presencial entre 09 e 14 de julho de 2023, em Egmond aan Zee/Holanda.

3.2 – Eventos nacionais

No período a que se refere este relatório, apresentei os seguintes trabalhos em eventos nacionais:

– F. Nascimento, K. Petroski, A. Alves e K. G. Kostov, "*Effects of oxygen addition on atmospheric pressure plasma jet parameters*" – trabalho apresentado em vídeo no XLI CBrAVIC 2020 (*XLI Brazilian Congress of Vaccum Applications in Industry and Science*), realizado de forma *online* de 09 a 11 de dezembro de 2020.

– F. Nascimento e K. G. Kostov, "*Variation of plasma jet parameters according to the distance to target*" – trabalho apresentado em forma de pôster no XLII CBrAVIC 2021 (*XLII Brazilian Congress of Vaccum Applications in Industry and Science*), realizado de forma *online* de 01 a 03 de dezembro de 2021.

– Fellype do Nascimento, Diego Morais da Silva, Aline da Graça Sampaio, Mariana Raquel da Cruz Vegian, Noala Vicensoto Moreira Milhan, Torsten Gerling, Paulo Francisco Guerreiro Cardoso, Cristiane Yumi Koga Ito, Konstantin Georgiev Kostov, "*Influence of the working gas on the temperature of atmospheric pressure plasma jets*" – Trabalho apresentado na forma de apresentação oral no XXI B-MRS Meeting, em Maceió/AL, de 01 a 05 de outubro de 2023.

– Fellype do Nascimento, Kleber A. Petroski, Thalita M. C. Nishime, Konstantin G. Kostov, "*Discharge power dissipated in devices with double plasma ignition*" – Trabalho apresentado na forma de pôster no XLIV CBrAVIC, em Campos do Jordão/SP de 25 a 29 de novembro de 2023.

Fui também coautor dos seguintes trabalhos:

– Konstantin Georgiev Kostov, Fellype do Nascimento, Torsten Gerling, Aline da Graça Sampaio, Mariana Raquel da Cruz Vegian, Diego Morais da Silva e Cristiane Yumi Koga Ito, "*A low-cost atmospheric pressure plasma jet for applications in oral medicine*" – Trabalho apresentado pelo Prof. Dr. Konstantin Georgiev Kostov, na forma de **palestra convidada**, no XXI B-MRS Meeting, em Maceió-AL, de 01 a 05 de outubro de 2023.

– Diego Morais da Silva, Noala Vicensoto Moreira Milhan, Fellype do Nascimento, Daniel Legendre, Paulo Francisco Guerreiro Cardoso, Fabio Gava Aoki, Konstantin Georgiev Kostov e Cristiane Yumi Koga Ito, "*In vitro evaluation of the antibacterial activity and cytotoxicity of a helium cold plasma jet on the treatment of tracheal prostheses*" – Trabalho apresentado pela Profa. Dra. Cristiane Yumi Koga

Ito, na forma de apresentação oral, no XXI B-MRS Meeting, em Maceió-AL, de 01 a 05 de outubro de 2023.

– Aline da Graça Sampaio, Noala Vicensoto Moreira Milhan, Fellype do Nascimento, Konstantin Georgiev Kostov e Cristiane Yumi Koga Ito, "*Effect of home-made non-thermal plasma jet on oral mucosal wound healing in rats undergoing chemotherapy treatment: a screening setting*" – Trabalho apresentado por Aline da Graça Sampaio, na forma de pôster, no XXI B-MRS Meeting, em Maceió-AL, de 01 a 05 de outubro de 2023.

– Aline da Graça Sampaio, Noala Vicensoto Moreira Milhan, Fellype do Nascimento, Konstantin Georgiev Kostov e Cristiane Yumi Koga Ito, "*The influence of exposure time of a non-thermal plasma jet prototype applied to biomedical pathogens for clinical use*" – Trabalho apresentado por Aline da Graça Sampaio, na forma de pôster, no XXI B-MRS Meeting, em Maceió-AL, de 01 a 05 de outubro de 2023.

– Ananias Alves Barbosa, Ana Carla De Paula Leite Almeida, Fellype do Nascimento, Konstantin Georgeiv Kostov, "*Understanding the interaction of plasma jets with the human body for medical applications*" – Trabalho apresentado na forma de pôster no XLIV CbrAVIC (novembro de 2023) por Ananias Alves Barbosa.

– Ana Carla de Paula Leite Almeida, Ananias Alves Barbosa, Fellype do Nascimento, Konstantin Georgiev Kostov, "*Rotational temperatures of N_2 molecules produced by cold plasma jets inside montgomery tubes*" – Trabalho apresentado na forma de pôster no XLIV CbrAVIC (novembro de 2023) por Ana Carla de Paula Leite Almeida.

– Fellype do Nascimento, Bruno Silva Leal, Konstantin Kostov, Study of Different Configurations of Transferred Plasma Jet Operated at Atmospheric Pressure – Trabalho apresentado por K. Kostov, na forma de apresentação oral, no Encontro de Outono da Sociedade Brasileira de Física (abril de 2022)

– BARBOSA, A. A.; ALMEIDA, A. C. P. L.; NASCIMENTO, F.; KOSTOV, K. G., Treatment of inner walls of polydimethysiloxane (PDMS) cylindrical objects using atmospheric pressure plasma jet – Trabalho apresentado por A. A. Barbosa, em forma de pôster, no Encontro de Outono da Sociedade Brasileira de Física (abril de 2022)

– ALMEIDA, A. C. P. L.; BARBOSA, A. A.; NASCIMENTO, F.; KOSTOV, K. G., Study of rotational temperatures and optical characterization for different configurations in DBD plasma jets inside a tube using the massiveoes software – Trabalho apresentado por A. C. P. L. Almeida, em forma de pôster, no Encontro de Outono da Sociedade Brasileira de Física (abril de 2022)

– Kleber Alexandre Petroski, Fellype do Nascimento, Konstantin Georgiev Kostov, Influence of the Tube Length and Leakage Current on a Transferred Cold Atmospheric Pressure Plasma Jet – Trabalho apresentado por K. A. Petroski, em forma de pôster, no Encontro de Outono da Sociedade Brasileira de Física (abril de 2022)

– Kleber Petroski, Fellype do Nascimento, Konstantin G. Kostov, “The target distance influence on the power of a transferred cold atmospheric pressure plasma jet” – Trabalho apresentado por K. Petroski, em forma de pôster, no XLIII CbrAVIC (agosto de 2022)

– ALMEIDA, A. C. P. L.; BARBOSA, A.A.; NASCIMENTO, F.; KOSTOV, K. G., Optical characterization of APPJs produced inside a tube from massiveoes automation using different settings – Trabalho apresentado por A. C. P. L. Almeida, em forma de pôster, no XLIII CbrAVIC (agosto de 2022)

– BARBOSA, A.A.; ALMEIDA, A. C. P. L.; NASCIMENTO, F.; KOSTOV, K. G. . Improved wetability of polydimethsiloxane (PDMS) sample through plasma jet at atmospheric pressure – Trabalho apresentado por A. A. Barbosa, em forma de pôster, no XLIII CbrAVIC (agosto de 2022)

– KOGA-ITO, CRISTIANE YUMI, MILHAN; NOALA VICENSOTO MOREIRA; SAMPAIO, ALINE DA GRAÇA; DO NASCIMENTO, FELLYPE; CHIAPPIM JUNIOR, WILLIAM; MIRANDA, FELIPE SOUZA; PESSOA, RODRIGO; KOSTOV, KONSTANTIN GEORGIEV; Cold atmospheric plasma applications in Biomedicine and Dentistry – Trabalho apresentado por C. Y. KOGA-ITO na forma de palestra convidada no XX B-MRS Meeting (setembro de 2022), Foz do Iguaçu/PR

– SILVA, D. M.; NASCIMENTO, F.; LEGENDRE, D; AOKI, F. G.; CARDOSO, P.; KOSTOV, K. G.; KOGA ITO, CRISTIANE YUMI, Study of medical-grade silicone surfaces’ behavior after cold atmospheric plasma exposure – Trabalho apresentado por D. M. Silva, em forma de pôsterno XX B-MRS Meeting (setembro de 2022), Foz do Iguaçu/PR

4 – Lista das publicações no período a que se refere o Relatório Científico

4.1 – Artigos em revistas científicas indexadas (comprovantes nos anexos A1–A9):

[A1] F. do Nascimento, K. G. Kostov, M. Machida, and A. Flacker, “*Properties of DBD Plasma Jets Using Powered Electrode With and Without Contact With the Plasma*,” *IEEE Transactions on Plasma Science*, vol. 49, no. 4, pp. 1293–1301, Apr. 2021, doi: 10.1109/TPS.2021.3067159 – link para repositório institucional: https://repositorio.unesp.br/items/fc01ff05-93bb-447d-acd3-5f8ea5ddd145)

[A2] F. Nascimento, K. Petroski, and K. Kostov, “*Effects of O_2 Addition on the Discharge Parameters and Production of Reactive Species of a Transferred Atmospheric Pressure Plasma Jet*” *Applied Sciences*, vol. 11, no. 14, Art. no. 14, Jul. 2021, doi: 10.3390/app11146311 – link para repositório institucional: https://repositorio.unesp.br/items/1c203463-e10d-4ca9-9c5a-994428664773)

[A3] Fellype do Nascimento, Bruno Silva Leal, Antje Quade, Konstantin Georgiev Kostov, "*Different Radial Modification Profiles Observed on APPJ-treated Polypropylene Surfaces According to the Distance Between Plasma Outlet and Target*", Polymers, vol. 14, p. 4524, Oct, 2022, doi: 10.3390/polym14214524 – link para repositório institucional: https://repositorio.unesp.br/items/28b7a32d-f53d-4c69-8956-76364545a71c)

[A4] Fellype do Nascimento, Antje Quade, Mara Adriana Canesqui, Konstantin Georgiev Kostov, "*Different configurations of transferred atmospheric pressure plasma jet and their application to polymer treatment*", *Contributions to Plasma Physics*, Vol. 63, Num. 1, p. e202200055, Jan. 2023, doi: 10.1002/ctpp.202200055 – link para repositório institucional: https://repositorio.unesp.br/items/c34d60bd-d5c8-4a52-be5b-a8b60914716d)

[A5] Fellype do Nascimento, Torsten Gerling and Konstantin Georgiev Kostov, "*On the gas heating effect of helium atmospheric pressure plasma jet*", *Phys. Scr.*, vol. 98, no. 5, p. 055013, Apr. 2023, doi: 10.1088/1402-4896/accb17 – link para repositório institucional: https://repositorio.unesp.br/items/5f3062de-bab3-40c9-9fea-c07ffb08a3e5)

[A6] C. Y. Koga-Ito, K. G. Kostov, F. S. Miranda, N. V.M. Milhan, N. F. Azevedo Neto, F. Nascimento and R. S. Pessoa, "*Cold Atmospheric Plasma as a Therapeutic Tool in Medicine and Dentistry*", *Plasma Chem. Plasma Process*, Aug. 2023, doi: 10.1007/s11090-023-10380-5 (Publicação em processo de arquivamento em repositório institucional)

[A7] Fellype do Nascimento, Augusto Stancampiano, Kristína Trebulová, Sébastien Dozias, Jan Hrudka, František Krčma, Jean-Michel Pouvesle, Konstantin Georgiev Kostov and Eric Robert, "*Plasma Electrode Dielectric Barrier Discharge: Development, Characterization and Preliminary Assessment for Large Surface Decontamination*", *Plasma Chem. Plasma Process*, Oct. 2023, doi: 10.1007/s11090-023-10409-9 (Publicação em processo de arquivamento em repositório institucional)

[A8] Fellype do Nascimento, Aline da Graça Sampaio, Noala Vicensoto Moreira Milhan, Aline Vidal Lacerda Gontijo, Philipp Mattern, Torsten Gerling, Eric Robert, Cristiane Yumi Koga-Ito, Konstantin Georgiev Kostov, "*A low cost, flexible atmospheric pressure plasma jet device with good antimicrobial efficiency*" – *IEEE Transactions on Radiation and Plasma Medical Sciences*, 13 March 2024, Vol. 8, Issue 3, pp. 307-322, doi: 10.1109/TRPMS.2023.3342709 (Publicação em processo de arquivamento em repositório institucional)

[A9] Diego Morais da Silva, Fellype do Nascimento, Noala Vicensoto Milhan, Maria Alcionéia Carvalho de Oliveira, Paulo Francisco Guerreiro Cardoso, Daniel Legendre, Fabio Gava Aoki, Konstantin Georgiev Kostov, Cristiane Yumi Koga-Ito, "*Cold Atmospheric Helium Plasma in the Post-COVID-19 Era: A Promising Tool for the Disinfection of Silicone Endotracheal Prostheses*," *Microorganisms*, vol. 12, no. 1, Art. no. 1, Jan. 2024, doi: 10.3390/microorganisms12010130 – acesso aberto em: https://www.mdpi.com/2076-2607/12/1/130 (Publicação em processo de arquivamento em repositório institucional)

5 – Trabalhos em preparação

[A10] Fellype do Nascimento, Kleber Alexandre Petroski, Thalita Mayumi Castaldelli Nishime and K onstantin Georgiev Kostov, "*Measurements of power dissipated in devices with double plasma discharge ignition*" – em preparação.

[A11] Bruno Honnorat, Fellype do Nascimento, Konstantin Georgiev Kostov, Torsten Gerling, "*Explanation of temperature gradients induced by a helium jet flushing into ambient air under consideration of the Dufour Effect – a simulation and experimental approach*" – em preparação – a s er submetido para revista *International Journal of Heat and Mass Transfer*.

Fellype do Nascimento (bolsista) Konstantin Georgiev Kostov (supervisor)

Guaratinguetá, 19 de junho de 2024

As páginas 20 a 23 foram removidas deste documento por se tratarem de documentos administrativos.

Anexo A1: Artigo publicado na revista *IEEE Trans. Plasma Sci.* (Abril de 2021) – 1ª página

Properties of DBD Plasma Jets Using Powered Electrode With and Without Contact With the Plasma

Fellype do Nascimento, Konstantin Georgiev Kostov, Munemasa Machida, and Alexander Flacker

***Abstract*— An experimental investigation comparing the properties of plasma jets in dielectric barrier discharge (DBD) configurations using a powered electrode with and without a dielectric barrier, while keeping a second dielectric barrier over the grounded electrode, is reported in this work. For this purpose, two different power sources were used to produce the plasma jets, with one of them producing a pulsed high-voltage (HV) output and the other one producing a damped sine-wave HV output, which acts as a pulse-like power source. Measurements of plasma parameters were performed for both configurations using argon and helium as working gases. As a result, if the pulsed power source is used, significant differences were found in discharge power (P_{dis}) and rotational and vibrational temperatures (T_r and T_v, respectively) when switching from one configuration to the other. On the other hand, using the pulse-like HV, only the P_{dis} parameter presented significant differences when switching the electrode's configuration. For the pulsed source, it has been observed that despite the remarkable increase in P_{dis} when changing from the double barrier configuration to the single barrier one, the values obtained for T_r and T_v also increased, but not in the same proportion as the increase in P_{dis}, which suggests a nonlinear dependence between temperatures and discharge power in the plasma jet. As an example for application of plasmas in both configurations, tests in an attempt to remove copper films deposited on alumina substrates were performed and, as a result, there was significant material removal only when the powered electrode was in contact with the plasma. As a general conclusion, if higher power is really required for applications that do not involve *in vivo* targets, it is better to use this configuration.**

***Index Terms*— Atmospheric pressure plasma jets (APPJ), dielectric barrier discharge (DBD), DBD plasma, plasma jets, plasma properties.**

I. Introduction

PLASMA plumes produced under atmospheric pressure and in open environment generally referred to as atmospheric pressure plasma jets (APPJs) have been extensively studied in recent years, and a large number of applications for these devices have been developed due to their versatility, easy operation, and low cost of implementation compared to low-pressure plasmas [1]–[4]. The APPJs applications can be in industry, biology, and medicine, with the last field receiving great attention, especially due to successful treatments of cancerous tissues and, more recently, for combating viruses, including the Sars-Cov-2, and the success in biological treatments using APPJs is strongly related to the reactive oxygen and nitrogen species produced in such gas discharges [5]–[17]. Dielectric barrier discharge (DBD) is a kind of electrodes arrangement commonly employed to produce APPJs. Its main characteristic is the presence of at least one insulating layer between metallic plates [1], [3]. Among the various DBD configurations, the cylindrical geometry is the most commonly employed because it naturally takes advantage of using the gas flow to produce a plasma jet [2]. Even though many studies on different APPJ arrangements have been carried out, yet there is no a particular configuration that can be considered ideal, even for specific applications [3], [4]. Therefore, research goes on and different geometry configurations and device variations have been explored. It has been a considerable challenge to control plasma jet properties as well as to establish a relationship between different plasma parameters.

Among different electrode arrangements in cylindrical APPJ reactors, those setups that present powered electrodes at the center of the reactor have been commonly employed. They can have such an electrode covered with a dielectric barrier or not, that is, with the powered electrode in contact with the working gas or not. Each configuration will produce a plasma jet with different temperatures, power, and/or density of reactive species that are created when the plasma interacts with the surrounding ambient air or with a surface [4], [18]. The first mentioned configuration (encapsulated powered electrode) presents higher electrical safety when compared to the other (a powered electrode without a dielectric barrier) and, for this reason, it is preferred for medical applications of plasma jets, especially when the treatments are *in vivo*.

Studies regarding the interaction between APPJs and different substrates have shown that the conductivity or dielectric properties of the substrate, as well as the distance from device output to the target surface, influence not only the treatment results but can also modify the characteristics of the plasma jet itself [19]–[24]. Works concerning differences in plasma jet properties when impinging on grounded or floating targets have been reported [25], [26]. To generate APPJs, most devices use sinusoidal voltages, ranging in frequencies from kilohertz to megahertz. Previous works of our group revealed that using conducting or dielectric targets (or sample holders) has an important effect not only on plasma jet properties but also

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Color versions of one or more figures in this article are available at https://doi.org/10.1109/TPS.2021.3067159.

Digital Object Identifier 10.1109/TPS.2021.3067159

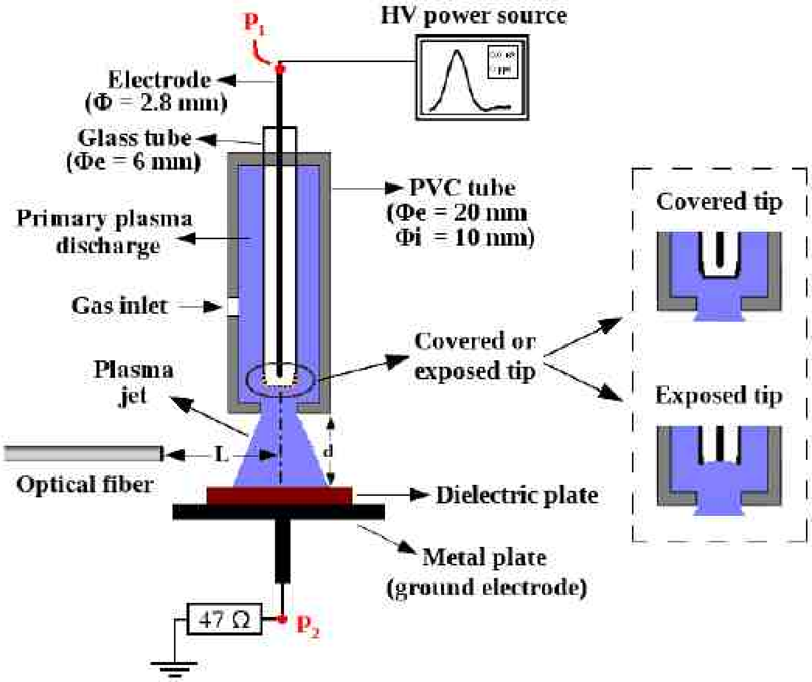

Fig. 1. Scheme used to produce the plasma jets in both configurations.

on plasma treatment outcome [19], [27]. For instance, it was verified that better adhesion of polydimethylsiloxane (PDMS) samples was achieved after plasma treatment using a conductive sample holder [27].

In this work, we studied the behavior of three main parameters of an APPJ: rotational and vibrational temperatures (T_r and T_v, respectively) and mean discharge power (P_{dis}) in a DBD device in two distinct configurations. The first one employed is more often reported in the literature, in which a plasma jet is produced between two dielectric barriers, one enveloping the powered electrode and the other covering the grounded one. In the second configuration, the dielectric on the powered electrode was partly removed, while the one covering the grounded electrode was kept. This last configuration was not extensively explored yet, mainly due to safety issues appearing in medical applications of APPJs. Some works using microwave, sinusoidal, and pulsed power sources have been reported using it but with no mention of measurements of plasma power, vibrational or rotational temperatures, or comparison for different working gases [28]–[36]. Since some applications may not require the safe operating conditions provided by the dielectric barrier over the powered electrode, these can take advantage of the higher plasma power and other jet properties that can be achieved when the dielectric barrier is removed.

II. Materials and Methods

A schematic layout of the experimental setup is shown in Fig. 1. In order to produce the plasma jets using the pulsed power source, high-voltage (HV) pulses of 20-kV amplitude and ~250-ns width (at half peak voltage value) with positive polarity followed by a negative polarity pulse with −12-kV amplitude and ~170-ns width, with 60-Hz repetition rate, were applied to a pin electrode covered with a glass tube with one of its ends closed (covered tip) or open (exposed tip), for the double- (DB) and the single-barrier (SB) configurations, respectively. Details about the pulsed power source can be found in [37]. A biaxially oriented polyethylene terephthalate (BoPET) foil, 500 μm thick and 10 cm sided, was placed on the top of a grounded electrode and acted as a common dielectric barrier (the dielectric plate in Fig. 1) in both cases. The distance **d** between the end of the dielectric enclosure (a polyvinyl chloride tube) and the dielectric plate was kept constant and equal to 10 mm. Argon (Ar) and helium (He), both with 99.99% purity, were used as working gases at the same flow rate of 3.0 l/min. The damped sine-wave power source consists of an HV supply whose output presents a damped sine waveform with an oscillation frequency of ~150 kHz and 60-Hz repetition rate. This power source acts as a pulse-like one due to the small duration of the voltage waveform when compared to its repetition rate. The maximum positive voltage peak provided by this power source is ~15 kV. The other parameters used in the experiment with the damped sine-wave power source are the same as those mentioned for the pulsed one.

The light emitted by the plasma was collected using an optical fiber with 600 μm core diameter and numerical aperture of 0.22, providing an acceptance angle of 25.4°. The optical fiber was placed at the center of the plasma column, in both horizontal and vertical directions. The distance **L** between the end tip of the optical fiber and the center of plasma device was kept unchanged in all experiments and equal to 10 mm. The spectroscopic measurements were performed using a portable multichannel spectrometer from OceanOptics (model HR4000), with a spectral resolution [full-width at half-maximum (FWHM)] equal to (0.545 ± 0.007) nm, measured at 632.8 nm.

Anexo A2: Artigo publicado na revista *Applied Sciences* (Julho de 2021) – 1ª página

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MDPI

Article

Effects of O_2 Addition on the Discharge Parameters and Production of Reactive Species of a Transferred Atmospheric Pressure Plasma Jet

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Abstract: The therapeutic effects of atmospheric pressure plasma jets (APPJs) have been associated with the presence of reactive species, mainly the reactive oxygen and nitrogen ones, generated in this kind of plasmas. Due to that, many studies attempting to enhance the production of reactive species in APPJs have been performed. The employment of gas admixtures, usually mixing a noble gas with oxygen (O_2) or water vapor, is one of the most common methods to achieve such goal. This work presents a study of how the addition of small amounts of O_2 affects the electrical parameters and the production of reactive species in a transferred APPJ produced at the tip of a long and flexible plastic tube. The study was carried out employing helium (He) as the working gas and applying a high voltage (HV) in the form of amplitude-modulated sine waveform (burst mode). With this configuration it was possible to verify that the O_2 addition reduces the discharge power and effective current, as a result of late ignition and shorter discharge duration. It was also found that the addition of O_2 to a certain content in the gas admixture makes the light emission from oxygen atoms increase, indicating an increment in oxygen related reactive species in the plasma jet. However, at the same time the light emitted from hydroxyl (OH) and nitric oxide (NO) exhibits the opposite behavior, i.e., decrease, indicating a reduction of such species in the APPJ. For these reasons, the addition of O_2 to the working gas seems to be useful for increasing the effectiveness of the plasma treatment only when the target modification effect is directly dependent on the content of atomic oxygen.

Keywords: DBD plasma; plasma jets; plasma properties; reactive species; RONS

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1. Introduction

In recent years atmospheric pressure plasma jets (APPJs) produced in open environments have received a lot of attention, not only because of their versatility and ease of use, but also due to the encouraging results achieved in the most diverse applications [1–5]. Special attention has been given to medical and biological applications of APPJs, whose beneficial effects have been attributed to the reactive oxygen and nitrogen species (RONS) produced by the plasma jets [6–8]. The actions of the RONS are not only limited to biological materials, since they also contribute to the hydrophilization and surface activation processes of many materials treated with plasma [9,10].

Many studies indicated that the addition of small amounts of oxygen (O_2) to the gas employed to produce plasma, usually argon (Ar) or helium (He), can increase the amount of atomic oxygen (O) produced in the APPJ [11–17]. To verify this increase in the population of O most authors have measured the light emission from excited O atoms coming from the 777 nm triplet or, in some cases, from the 844 nm triplet [13]. In some works, it was found that there is an optimal O_2 percentage that maximizes the intensity of the O emission lines. There are also works in which the authors reported that some applications of the plasma jets, with both inorganic and biological materials as a target, showed better results when there was a small O_2 addition to the plasma. However, only a few works analyzed the production of other reactive species, such as OH and NO, together with the O production. In particular, Li et al [15] studied the production efficiency of RONS as a function of the O_2 content in the He-O_2 admixture, in the range from 0–2% of the total gas flow rate, and applying different voltage waveforms to generate APPJs. In that work, Li et al have found that the production efficiency of both OH and NO decreases with O_2 addition but the opposite occurs for the O_3 and NO_2 molecules. In the case of the production efficiency of O atoms, a peak value was observed for 0.5% of O_2 content. Those results were almost the same for any HV waveform applied to generate the APPJ.

Regarding the effects of additional O_2 on APPJ parameters, the main changes reported in the literature are decrease in jet length, reduction on discharge power and current, and variations in temperatures (rotational and vibrational) [18–24]. Some works also identified an increment in the applied voltage as a function of the O_2 content, even with the power source configured to apply a fixed voltage to the electrodes, a fact that suggests a change in the load impedance followed by an impedance matching [25,26]. Lazzaroni et al [27] performed an investigation on the stability of APPJs as a function of the O_2 content in the working gas which, based on a global model for APPJs produced with He-O_2 admixtures, has shown that there is no discharge equilibrium if the oxygen fraction exceeds 1% in a configuration that employs a power source with frequency of 13.56 MHz and a gap of 1 mm between the electrodes. The discharge dynamics in dielectric barrier discharge (DBD) plasmas with O_2 in the gas admixture was studied only using N_2-O_2 in a work aimed to investigate the transition from diffuse to filamentary regime [28].

Most studies found in the literature evaluate the production of RONS for only one fixed flow rate for the working gas, varying solely the amount of O_2 added to it. The present paper presents results using two different flow rates for the working gas together with the variation in the percentage of O_2 added to the plasma. The analysis of the RONS production as a function of the amount of O_2 present in the gas admixture is also performed for different distances from plasma outlet (in this case the end of a long and flexible plastic tube) to the target. This work also aims to evaluate the possible changes in the electrical parameters of plasma jets, mainly the discharge power and the effective current when small amounts of O_2 are added to the working gas. In addition, the unique combination of a plasma jet produced at the end tip of a long and flexible plastic tube with a high voltage (HV) presenting a sinusoidal-burst waveform can provide insights about the mechanisms involved in the interaction between the additional O_2 and the working gas in the generation of plasma jets.

2. Materials and Methods

Figure 1a shows the experimental setup used in this work, with detailed views of the light collection scheme shown in Figure 1b. Figure 1c presents an example of the waveform applied to the high voltage (HV) electrode without plasma ignition. The plasma jet device presented in Figure 1a consists mainly of a DBD type reactor, composed by a metal pin electrode placed inside a closed-end quartz tube, which in turn is placed inside a dielectric chamber. The working gas is fed into the chamber and flushed to the ambient air through a 1-meter long and flexible plastic tube connected to the reactor output. The plastic tube material is nylon-6, with outer and inner diameters equal to 4.0 mm and 2.0 mm, respectively. A conducting wire with 0.25 mm in diameter is put inside the plastic tube (see the floating electrode in Figure 1a). This floating electrode penetrates a few millimeters inside the reactor in a way that it does not touch the quartz tube. The other wire tip ends 2 mm before the plastic tube exit. When the primary discharge inside the DBD reactor is on it polarizes the floating electrode and a small plasma jet is ignited at the end of the plastic tube. This remote plasma jet was directed towards a copper plate, placed at a distance **d** from the plasma outlet, which is connected to an electric circuit whose impedance follows an international standard (IEC 60601-1). It is a simplified electrical circuit model aimed to mimic the electrical properties of the human body [29]. To obtain controllable gas admixtures of He and O_2 as a working gas, both gases have their flow rates

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ORIGINAL ARTICLE

Contributions to Plasma Physics

Different configurations of transferred atmospheric pressure plasma jet and their application to polymer treatment

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Abstract

The employment of atmospheric pressure plasma jets (APPJs) in a large sort of applications is limited by the adversities related to the size of the treated area and the difficulty to reach the target. The use of devices that employ long tubes in their structure has contributed significantly to overcome these challenges. In this work, two different plasma systems employing the jet transfer technique are compared. The main difference between the two devices is how the long plastic tube was assembled. The first one uses a copper wire placed inside a long plastic tube. The other device has a metallic mesh installed in a concentric arrangement between two coaxial plastic tubes. As a result, the two APPJ systems exhibit different properties, with the wire assembly being more powerful, also presenting higher values for the electrical current and rotational temperature when compared to the mesh mounting. X-ray photoelectron spectroscopy (XPS) demonstrates that both configurations were capable of inserting O-containing functional groups on the polypropylene (PP) surface. However, the transferred plasma jet with wire assembly was able to add more functional groups on the PP surface. The results from XPS analysis were corroborated with water contact angle measurements (WCA), being that lower WCA values were obtained when the PP surface presented higher amounts of O-containing groups. Furthermore, the results suggest that the APPJ with wire configuration is more appropriate for material treatments, while the transferred jet with mesh arrangement tends to present lower electrical current values, being more suitable for biological applications.

KEYWORDS

DBD plasma, plasma jets, plasma properties, plasma treatment, transferred plasma

1 | INTRODUCTION

In recent years atmospheric pressure plasma jets (APPJs) produced in open environment have received a lot of attention, not only because of their versatility and ease of use, but also due to the encouraging results achieved in the most diverse applications.[1–4] Special attention has been given to medical and biological applications of APPJs, whose beneficial effects have been attributed to the reactive oxygen and nitrogen species (RONS) produced by the plasma jets.[5–7] The actions of the RONS are not only limited to biological materials, since they also contribute to the hydrophilization and surface activation processes of a large number of materials treated with APPJs.[8,9]

Most APPJ devices produce plasma jets with small cross-section area, ranging from a several square millimetres to few square centimetres (usually less than 2 cm^2). In most cases this is a limitation, since the targets to be treated with APPJs usually have larger dimensions. In other cases, like in dentistry and medicine, it is an advantage because it provides a localized treatment. However, in all situations, it is necessary to provide a way for the plasma to reach the target to be treated. Whether it is bringing the target closer to the device that produces the plasma, or finding a way to bring the plasma to it. For the last purpose, some devices that produce plasma jets at the end tip of long and flexible tubes have been developed.[6,10–25] Being that some of them use the plasma jet transfer technique, which is basically a secondary plasma jet formed away from a primary discharge that is generated inside a reactor or from a primary plasma jet.[6,15,17,24,26,27] One of the advantages of use the jet transfer technique in APPJ devices with long tubes is that the plasma jet is produced far from the high voltage source which improves the electrical safety in its operation and handling. Furthermore, the use of the jet transfer technique facilitates the employment of an APPJ to treat large target materials by scanning their surfaces.

One of the problems related to the generation of plasma jets using long polymeric tubes is the possibility of plasma production in the tube length instead of obtaining a plasma jet only at the end tip of the tube. This is a problem because the plasma tends to interact with the tube material causing a degradation. Some studies found that this can be prevented by using a second tube surrounding the first, with a space to flow a different gas between the two tubes.[21,28] A study by Onyshchenko et al. aimed to investigate penetration depth of plasma inside flexible tubes suggests that this problem can be minimized by employing long tubes with an appropriated internal diameter.[29] In that work it was observed that the plasma penetration depth in tubes depends on their internal diameters, being that there is a certain tube diameter that maximizes the penetration depth.

The devices that employ long and flexible tubes to produce plasma jets for materials treatment have shown results that are as good as those that do not use long tubes. However, the most interesting results are related to the possibility of performing treatments in cavities, specially in combination with endoscopes.[21,22,28] Nonetheless, before to apply a new configuration or technique to produce APPJ on biological materials or on in vivo targets, it is convenient, and also a common practice, to begin studies with inorganic materials like polymers.

In this work we present a modified design concept using the jet transfer to generate APPJs at the ends of long and flexible tubes. It consists of a metallic mesh placed between two long plastic tubes in a concentric arrangement. The main objective of using this design is to reduce the electrical current that flows through the plasma jet thus making it more suitable for in vivo applications. This configuration was compared with another that employs a metal wire inside a long tube. Both devices were characterized with electrical and optical diagnostics and comparisons of the plasma parameters obtained on each case are provided. A study comparing the use of the two different configurations for polymer treatment was also performed. The results revealed differences in the surface treatments when each configuration is used.

2 | EXPERIMENTAL SETUP

Figure 1a shows the experimental setup used in this work, with a detailed view of the two long tube configurations illustrated in Figure 1b. The plasma jet device presented in Figure 1 is based on a dielectric barrier discharge (DBD) type reactor, composed by a metal pin electrode encapsulated in a closed-end quartz tube, which in turn is placed inside a dielectric chamber. Completing the device, there is a 1-m long and flexible plastic tube connected to the DBD reactor. In this work, the long tube employed to produce APPJ was assembled in two different ways. In the first one a 0.5 mm thin copper wire is put inside a plastic tube, whose material is Nylon 6, with outer diameter (OD) equals to 4 mm and inner diameter (ID) equals to 2 mm. In the second configuration the long tube is composed by a metallic mesh, with 90% closed area extended over a polytetrafluoroethylene (PTFE) tube with OD = 3 mm and ID = 2 mm, being that both of which are placed inside a Nylon 6 tube with OD = 6 mm and ID = 4 mm.

Both the wire and the mesh are fixed to a metallic connector that goes inside the reactor and acts as a floating electrode (see Figure 1). Besides that, each set formed by the long tube and metallic connector is attached to its own exchangeable coupler, which is made of polyoxymethylene (POM). Furthermore, both copper wire and metallic mesh ends 2 mm before the plasma outlet, and in the last configuration there is an electrical insulation at the end of the mesh in order to avoid discharges coming directly from it.

Anexo A4: Artigo publicado na revista *Polymers* (Outubro de 2022) – 1ª página

polymers

MDPI

Article

Different Radial Modification Profiles Observed on APPJ-Treated Polypropylene Surfaces according to the Distance between Plasma Outlet and Target

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Abstract: The plasma jet transfer technique relies on a conductive wire at floating potential, which, upon entering in contact with a primary discharge, is capable of igniting a small plasma plume at the distal end of a long flexible plastic tube. In this work, two different long tube configurations were employed for the surface modification of polypropylene (PP) samples using argon as the working gas. One of the jet configurations has a thin copper (Cu) wire, which was installed inside the long tube. In the other configuration, the floating electrode is a metallic mesh placed between two plastic tubes in a coaxial arrangement. In the first case, the tip of the Cu wire is in direct contact with the working gas at the plasma outlet, whereas, in the second, the inner plastic tube provides an additional dielectric barrier that prevents the conductor from being in contact with the gas. Water contact angle (WCA) measurements on treated PP samples revealed that different surface modification radial profiles are formed when the distance (d) between the plasma outlet and target is changed. Moreover, it was found that the highest WCA reduction does not always occur at the point where the plasma impinges the surface of the material, especially when the d value is small. Through X-ray photoelectron spectroscopy (XPS) analysis, it was confirmed that the WCA values are directly linked to the oxygen-functional groups formed on the PP surfaces after the plasma treatment. An analysis of the WCA measurements along the surface, as well as their temporal evolution, together with the XPS data, suggest that, when the treatment is performed at small d values, the plasma jet removes some functional groups at the point where the plasma hits the surface, thus leading to peculiar WCA profiles.

Keywords: DBD plasma; plasma jets; transferred plasma; plasma treatment; polymer treatment

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1. Introduction

The surface modification of materials through the use of cold atmospheric pressure plasmas (CAPPs) has been a subject of intense research and development for both academic and technological purposes. This enhanced interest is caused by the fact that CAPPs are able to perform both chemical and physical action on the treated surfaces by creating/removing functional groups and changing the surface roughness [1–4]. For a large quantity of materials, surface modifications are required or desired to facilitate the occurrence of other processes, such as adhesion, or to enhance another surface property [5–10].

The device configurations that can be used to produce CAPPs for material treatment are quite diverse [5,11–14]. A few examples of systems that can operate in open environments are dielectric barrier discharge (DBD), discharge inside a funnel and atmospheric pressure plasma jets (APPJs) [12,15–17]. Planar DBD devices can be assembled in ways appropriate to treat materials with large area surfaces [18]. On the other hand, APPJs are able to provide localized surface treatment due to the small plasma plume dimensions of the typical plasma jets [19–21]. APPJs produced at the end of long and flexible tubes are able to treat complex surfaces as well as the ones inside cavities [22,23]. In addition, since the device is flexible, it can be mounted onto a scanning tool and adapted to treat large area surfaces.

APPJs can be produced by direct plasma extraction from the discharge in a dielectric enclosure or through the jet transfer technique (as a secondary plasma jet generated from a primary one) [13,22,24,25]. The advantage in the first case is that the voltage values required to ignite the discharge are, in general, lower than in the second. However, the jet transfer technique has as an advantage in that the high-voltage electrode is far from the plasma application spot. When the jet transfer technique is employed, the voltage that ignites the remote plasma jet is the one on the floating metallic conductor, which is lower than the applied voltage [24,25]. When APPJs are produced using the jet transfer technique, most of the species in the primary discharge are mainly the ones from the working gas atoms (in both excited and ionized states). However, some molecules from the air eventually enter the reactor chamber and sum up to others that originate along the pipes connecting the main gas bottle to the plasma device, becoming chemically active due to the ignition of the primary discharge. Nonetheless, the largest amount of active species is produced together with the secondary discharge, that is, the plasma jet, due to the interaction with the surrounding air.

When comparing the surface modification induced by punctual plasma sources, such as the plasma jets, to DBD systems that are able to treat large areas at once, the latter can achieve a better treatment homogeneity than the first. However the typical discharge gap in DBD systems is of the order of a few millimeters, thus limiting their application to thin flat samples. On the other hand, besides their small modification range, APPJs have a higher energy density per unit area; therefore, they perform a much faster treatment, and the surface modifications tend to be more intense [26]. Thus, the challenge of employing APPJs for the treatment of large objects and/or irregular objects is carrying this out in a homogeneous manner. Some works reported success in achieving uniformity in surface treatment by employing multiple plasma jets operating in parallel [27,28]. However, the individual plasma jets tend to interact with each other, making the operation of a plasma jets array a quite complex task.

A commonly used way to evaluate the interaction between plasmas and materials is to check if there were changes in the surface wettability, which is usually assessed by water contact angle (WCA) measurements, comparing the WCA values before and after plasma exposure [29]. Most of the works on polymer surface treatment by APPJs report similar modification profiles, obtained through WCA measurements, that resemble an inverted Gaussian profile [5,30,31]. A work by Naramisa et al. presents a slightly different WCA profile, with a deeper WCA reduction close to the spot where the plasma impinges the target [32]. A work by Shao et al. reported that the WCA reduction as a function of the distance d between the plasma outlet and the target surface does not present a monotonic behavior as a function of d when polymethyl–methacrylate samples are exposed to plasma treatment [33].

In this work, we report on the surface modification of polypropylene (PP) using APPJs. The treatments were performed using APPJs produced at the tip of long and flexible plastic tubes. Two different device configurations were employed in this study. Both configurations exploit the jet transfer technique, but their design concepts differ significantly. WCA measurements revealed that plasma treatment enhances the surface wettability and that the WCA radial profile along the surfaces changes significantly according to the distance between the plasma outlet and the target. X-ray photoelectron spectroscopy (XPS) analysis confirmed that the WCA profiles are related to the insertion of oxygen-functional groups on the polymer surface.

2. Materials and Methods

Figure 1a shows a schematic of the experimental setup used in this work. A detailed view of the two long tube configurations is depicted in Figure 1b. The main plasma source

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PAPER

On the gas heating effect of helium atmospheric pressure plasma jet

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Keywords: low temperature plasma, plasma medicine, plasma jet

Abstract

Plasma medicine is an emerging research field which has been driven by the development of plasma sources suitable to generate low temperature plasmas. In many cases, such devices can operate without a gas flow, producing a plasma discharge from the ambient air. However, the most common case is the use of a working gas at a given flow rate to produce a plasma jet. Helium (He) is one of the gases commonly used as the carrier gas to generate cold atmospheric pressure plasma jets (CAPPJs) due mainly to the easiness to ignite a gas discharge with it. However, in this work it was observed that most of the heating of a He CAPPJ can come just from the expansion of the He gas. This was found through measurements of gas temperature (T_{gas}), using fiber optic temperature (FOT) sensors, and thermal output, using both FOT and infrared imaging with the He flow impinging on a copper (Cu) plate. Such findings were achieved through comparisons of T_{gas} and the temperature on the Cu (T_{Cu}) plate in the conditions with and without discharge ignition, as well as comparing T_{gas} in the free gas/jet mode with and without discharge ignition. It was verified that the T_{gas} values increased as the distance from the gas outlet was enlarged, especially at low He flow rates, even without discharge ignition. Despite the temperature increase with distance, it is possible to produce plasma jets with temperatures lower than 40 °C at low He flow rates.

1. Introduction

Cold atmospheric pressure plasma jets (CAPPJs) have been successfully employed for treatment of a large sort of materials and in plasma medicine over the last two decades [1–6]. When applied on non-biological substrates the CAPPJs have both chemical and physical action on the treated surfaces by creating/removing functional groups and changing the surface roughness [7–10]. When interacting with biological tissues the plasma jets still have physical action, but the main interaction mechanism have been attributed to chemical reactions, like oxidative cellular stress, due to the generation of reactive oxygen and nitrogen species (RONS) within the plasma [4, 11, 12]. Some works also reported that pulsed electric fields generated within the CAPPJs have significant effects on biological targets, acting as a physical agent in synergy with other plasma properties and the RONS [13, 14]. Both physical and chemical actions of CAPPJs interacting with materials have significant dependence on the working gas [15, 16]. Air, argon (Ar) and helium (He) are the gases that are employed most often to generate CAPPJs, sometimes in admixtures with nitrogen (N_2), oxygen (O_2), water vapor or a different gas combination [11, 17]. The use of He as the carrier gas is one of the easiest ways to ignite a plasma discharge at atmospheric pressure using relatively low high voltage (HV) values. Only plasma jets generated with neon (Ne) usually have a lower ignition voltage than that of He for the same plasma device and operating conditions [18]. The choice of the working gas depends not only on the easiness in the plasma ignition, but also on factors like the gas temperature (T_{gas}) in the discharge phase, discharge power and current, production of reactive species and operation costs. In one of the first works comparing the use of He and Ne to produce plasma jets, it was found that the plasma propagation velocity can be higher when using Ne [19]. In addition, when considering endoscopic applications gas diffusion in tissues is also a critical factor to be considered, since He can diffuse in tissues, eventually in blood vessels or into the lungs differently from Ne, for example [20].

Anexo A5: Artigo publicado na revista *Phys. Scr* (Abril de 2023) – 2ª página

When a plasma jet is applied in the plasma medicine context, one of the most important parameters to be considered is the T_{gas}. Being that, for application in human tissues the T_{gas} value should not exceed 40 °C in order to avoid cellular damage due to warming [21, 22]. Most works in the literature report that He is one of the working gases that produces atmospheric pressure plasma jets (APPJs) with T_{gas} values close to the room temperature, that is, 300 K or 27 °C. Nonetheless, this is not always the case, since the T_{gas} value in a plasma jet depends on several parameters like the discharge power and current, conductivity of the material on which the plasma impinges, gas flow rate (Q) and, eventually, on the room temperature [23, 24]. In addition, it is possible to cool down the working gas before using it to generate APPJs, which also can affect the gas temperature during the discharge phase. The employment of high Q values is one of the common ways to reduce the gas temperature [25, 26]. A shielding gas can also be used for such purpose [26].

Another important parameter to be considered when applying plasma jets for treatment of materials or tissues is the distance (d) between the plasma outlet and target. The d value influences many discharge parameters like temperature, discharge power, electrical current, electric field, as well as the production of reactive species, ultra violet (UV) light emission, etc [27]. Being that, all these parameters can contribute with the so-called plasma dose [28]. In fact, measurements of some plasma parameters as a function of d are required by the German DIN (German Institute of Standardization)-specification 91315. That specification can be seen as a complement of the IEC 60601-1 standard, as part of the criteria to certify plasma sources that are aimed for medical applications [29–31].

Gas temperature measurements in CAPPJs can be assessed through different techniques and equipment. The main difficult to measure this parameter is to obtain precise and accurate values without disturb or change other plasma jet properties. Optical emission spectroscopy (OES) is a non-disturbing technique that is used for T_{gas} measurements. However, the maximum precision in this case is of the order of 10 K (10 °C), which is not good enough in some cases [32]. In some cases, infrared cameras (IR cams) can also be used as a tool to estimate T_{gas} without disturbing the plasma. However, it is not so easy to capture the effluent image if T_{gas} is close to the room temperature (T_{room}). The employment of thermocouples for T_{gas} measurements are also reported on the literature [33–35]. Nonetheless, this kind of device usually has metallic components in the sensor probe. This can be a problem because plasma jets interact strongly with metals and, therefore, the results may not be accurate in this case. In addition, a thermocouple can also act as a floating electrode when in contact with the plasma jet, eventually influencing the discharge parameters [36, 37]. This can be avoided if the probe is covered with a dielectric material, as in [35]. However, in this case the response in time of the temperature readings became slower. Fiber optic temperature (FOT) sensors also have been used to measure T_{gas} in plasma jets [27, 38–41]. Such kind of device does not have metallic compounds in the sensor region. Its tip is usually composed by a semiconductor and coated with a thin dielectric material. Thus, this will not present significant electrical interaction with the plasma. Nonetheless, such sensor can disturb the gas flow depending on the relative dimensions of both plasma jet and FOT. Besides that, FOT sensors can usually be assembled or chosen with appropriate dimensions and provide T_{gas} measurements with an accuracy of the order of 0.2 °C when calibrated at room temperature.

It is known that helium and hydrogen (H_2) are the only gases that present negative values for the Joule-Thomson coefficient at room temperature, which makes a gas to heat up under adiabatic expansion at constant enthalpy [42–44]. It is also known that the He gas is subjected to buoyancy when it is flushed into the ambient air, mainly changing the propagation direction of the gas flow. This fact can affect some plasma jet parameters and the interaction of the electric field with the gas stream [45–48]. The buoyancy effect in He APPs tends to be less significant as the gas flow rate is increased [46]. Nevertheless, the effects related to these phenomena on the temperature of APPJs have not been fully taken into account in applications that employ He as the working gas, especially the medical and biomedical ones.

In this work, using FOT sensors to perform gas temperature and both FOT and infrared (IR) imaging for surface temperature and thermal output measurements. Through these measurements it was possible to identify that the main heating source of an APPJ that uses He as the working gas can be the He gas itself. It was found that the He gas temperature increases considerably as the distance from the gas outlet is incremented, with or without discharge ignition. This was observed in both free gas/jet mode and with a Cu plate in the He flow way. In the last case thermal energy was transferred to the target even without plasma discharge, that is, the Cu plate was heated up through simple exposure to the He flow. It was also found that the increment in T_{gas} values as a function of d depends significantly on the gas flow rate.

2. Materials and methods

Gas temperature (T_{gas}) measurements were performed using helium (He, 99.999% purity) with ignition of a plasma discharge and also without that, that is, only the neutral gas temperature was measured in the last case. In

Anexo A6: Artigo publicado na revista *Plasma Chem. Plasma Process* (Agosto/2023) – 1ª página

Plasma Chemistry and Plasma Processing
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REVIEW ARTICLE

Cold Atmospheric Plasma as a Therapeutic Tool in Medicine and Dentistry

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Abstract
This comprehensive review explores decade-long research conducted on the applications of Cold Atmospheric Plasma (CAP) and Plasma Activated Liquids (PAL), including Plasma Activated Water (PAW), in the fields of Medicine and Dentistry. CAPs, operating at atmospheric pressure, offer unique advantages over conventional medical devices, generating Reactive Oxygen Species (ROS) and Reactive Nitrogen Species (RNS) that interact with substances, enabling diverse applications. The review examined CAP and PAL efficacy against infectious diseases using in vitro, ex vivo, in vivo, and direct application methods. Significant strides were observed in wound healing, cancer treatment, and dental care. However, ensuring patient safety through rigorous plasma source standards remains crucial. The study underscores CAPs and PALs' potential to transform medical and dental therapies, urging further research and development in these groundbreaking technologies. These findings highlight the transformative impact of CAPs and PALs, offering promising avenues for innovative medical and dental treatments.

Keywords Cold atmospheric plasma · Plasma-activated liquids · Plasma-activated water · Plasma medicine · Plasma dentistry

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Introduction

In the fields of medicine and dentistry, a diverse range of therapeutic tools has been harnessed to address various health conditions and challenges. From traditional pharmaceutical interventions to cutting-edge surgical techniques, the quest for effective and innovative therapies continues to drive research and development in these crucial domains.

Among the recent advancements, Cold Atmospheric Plasmas (CAPs) have emerged as a promising technology with the potential to revolutionize various applications in academia and industry. What sets CAPs apart from conventional medical devices is their unique ability to operate at atmospheric pressure, distinguish them from other technologies requiring different pressure levels for therapeutic interventions.

The distinguishing feature of operating at atmospheric pressure empowers CAPs to generate a diverse array of active agents, each holding immense potential for applications across a multitude of medical and dental scenarios. Figure 1 showcases CAP generation with diverse sources, such as RONS, UV radiation, and electric fields, promising applications in direct and indirect treatments. Among the noteworthy active agents produced by CAPs are Reactive Oxygen Species (ROS) and Reactive Nitrogen Species (RNS), collectively referred to as RONS. These reactive species have garnered significant attention due to their remarkable ability to interact with various substances and induce chemical reactions, thereby playing a pivotal role in numerous fields. In the biomedical sector, RONS have demonstrated valuable antimicrobial properties, making them particularly advantageous for sterilization and disinfection processes. Their potency against harmful microorganisms has shown promise in effectively combating infections and safeguarding patients in clinical settings [1, 2]. Moreover, CAPs have the capacity to generate ultraviolet (UV) photons, characterized by their high energy levels. These UV photons hold tremendous significance in a myriad of applications. For instance, they are instrumental in sterilization processes where their ability to initiate photochemical reactions proves invaluable. By targeting the DNA of microorganisms, UV photons can lead to their inactivation, ensuring a hygienic environment and enhancing patient safety [3]. Furthermore, CAPs possess the unique capability to generate charged particles, offering vast potential in numerous applications. In healthcare, these charged particles find utility in ionizing radiation treatments, while in technology, they contribute to the creation of microelectronic devices, among other uses. The cancer therapy is one area that stands to benefit significantly from CAPs, as the charged particles produced by these plasmas can cause DNA damage in cancer cells, ultimately leading to their death [4].

Over the past decade, CAPs have drawn considerable interest in the scientific community due to their distinctive properties and diverse potential applications in the fields of engineering, materials science, environment, medicine, etc. In the field of medicine, CAP have made a significant impact, providing innovative therapeutic applications such as wound healing, cancer treatment, and medical equipment sterilization [5]. Furthermore, the interaction of CAPs with living tissues has unveiled a multitude of simultaneous biological effects, encompassing antimicrobial, anti-inflammatory, tissue repair, and antineoplastic properties [2, 6]. In dentistry, CAPs have also demonstrated their transformative potential, contributing notably to dental implant sterilization, tooth whitening, and the treatment of various oral diseases [7], further underlining their promise in revolutionizing dental treatments, and enhancing patient outcomes.

Anexo A7: Artigo publicado na revista *Plasma Chem. Plasma Process* (Outubro/2023) – 1ª página

Plasma Chemistry and Plasma Processing
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ORIGINAL PAPER

Plasma Electrode Dielectric Barrier Discharge: Development, Characterization and Preliminary Assessment for Large Surface Decontamination

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Abstract

The paper gives the detailed information about a newly developed plasma system applicable for conductive target non-thermal plasma indirect treatment. High voltage microsecond duration pulses delivered in the kHz range are used to ignite a discharge in a glass funnel vessel flushed with argon and equipped with a needle electrode. An air dielectric barrier discharge (DBD) can subsequently be generated if a grounded grid is set a few millimeters apart from the thin glass plate constituting the funnel base, in the funnel-DBD setup. Thus, this air DBD operates with its powered electrode consisting in the transient argon streamer discharge spreading inside the funnel and over the glass plate. This "plasma electrode DBD" is characterized using time-resolved ICCD imaging together with voltage and current probes. This work reports for the first time the funnel-DBD proof of concept operation and its potentialities for large surface decontamination. Argon and air plasma temporal and spatial development is documented and analyzed while electrical characterization using Lissajous plots provide key information on the power and capacitances of the funnel-DBD setup. It is reported that the funnel-DBD operates as a large surface and low power discharge. As with any air-DBD plasma, the modulation of the power density delivered across the air-DBD, processed with changing the pulse repetition rate, results in the control of the ozone concentration. Beyond the plasma electrode-DBD development and characterization, the main motivation of this work is the treatment of conductive samples with the perspective of large surface decontamination. Preliminary demonstrations of the bacterial and yeast inhibition are thus reported for in vitro cultivations through indirect treatment with the funnel-DBD delivering reactive nitrogen and oxygen species.

Keywords Dielectric barrier discharge · Surface decontamination · ICCD imaging · Streamer discharge · Plasma electrode

Extended author information available on the last page of the article

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Introduction

Nowadays, surface modification and decontamination by atmospheric pressure plasmas are well established technologies. By tailoring the surface properties, it becomes possible to improve adhesion between polymers and other materials, enable printing or coating processes, enhance surface energy, introduce functional groups, reduce friction or wear, increase resistance to chemicals or environmental degradation, enable specific biological interactions, inactivate germs and induce tissue healing among other possibilities. These modifications pave the way for advancements in industries such as automotive, aerospace, packaging, electronics, and healthcare, as well as stimulate the continuous development of novel atmospheric pressure plasma sources.

Atmospheric pressure plasma jets (APPJs) are low-cost devices that emerged as an alternative to the conventional dielectric barrier discharge (DBD) systems that are well suited for processing plane samples, but inadequate for treating complex 3D structures. Typically, in an APPJ electric discharge is ignited in a noble gas that flows through a dielectric channel and the resulting plasma is ejected into the surrounding environment (usually ambient air), forming a plasma plume [1]. The plasma plume is the discharge region, driven by ionization waves, expanding at the outlet of the capillary channel. As the plasma stream propagates into the surrounding atmosphere [2], it interacts with the ambient molecules creating reactive oxygen and nitrogen species (RONS) that upon reaching a target can induce surface modification [3, 4] and decontamination [5] effects. The plasma jets are characterized by diverse geometries and electrode configurations and can be driven by pulsed DC, AC, RF and microwave sources using different operating parameters, which makes them very versatile [1, 4]. In addition, the APPJs normally generate a several cm-long plasma plume [6] that can be easily adapted to treat irregular 3D objects and internal surfaces of narrow tubes or cavities [5]. Moreover, the gas temperature can be kept quite low (under 40 °C in some cases), which makes the plasma jets suitable for medical applications and treatment of thermosensitive materials [4]. All these beneficial properties of the plasma jets led to intense research in the last two decades resulting in the development of many different APPJ systems [1, 5].

However, the plasma jet application in material surface modification and decontamination presents its challenges. For instance, for many technological applications achieving uniform treatment across extensive areas, maintaining treatment conditions, and ensuring process scalability, are among the issues that need to be addressed. An inherited drawback of most common APPJs is the fact that the plasma plume diameter is determined by the inner diameter of the dielectric tube or capillary [1, 6, 7], (normally few mm), which combined with the short lifetime of the reactive species results in a limited treatment zone [8]. In fact, the size of the modified area of an atmospheric plasma jet is a dynamic parameter that can be controlled and adjusted to a certain extent through various means, including jet exit nozzle design, gas flow rate, discharge operating parameters, and distance to the target [9]. Understanding and optimizing these factors is crucial for achieving the desired outcome in plasma jet applications. Yet, the substrate area altered by a single plasma jet is typically limited to a few cm^2 [9, 10]. Moreover, within the plasma-treated surface the degree of material modification/decontamination is not uniform, being more intense at the center (where the plasma jet hits the target) and less pronounced at the edges [11]. As shown in [12] by using proper shielding gas the plasma-modified area can be further extended by ~ 20%, but this gain is at the expense of introducing new components and increasing the system complexity.

Anexo A8: Artigo aceito para publicação na revista *IEEE Transactions on Radiation and Plasma Medical Sciences* (Dezembro/2023) – 1ª página

A low cost, flexible atmospheric pressure plasma jet device with good antimicrobial efficiency

Fellype do Nascimento, Aline da Graça Sampaio, Noala Vicensoto Moreira Milhan, Aline Vidal Lacerda Gontijo, Philipp Mattern, Torsten Gerling, Eric Robert, Cristiane Yumi Koga-Ito, Konstantin Georgiev Kostov

Abstract—**Plasma sources suitable to generate low temperature plasmas has been fundamental for the advances in plasma medicine. In this research field, plasma sources must comply with stringent conditions for clinical applications. The main requirement to be met is the patient and operator's safety and the ethical requirement of effectivity, which encompasses the electrical regulations, potential device toxicity and effectiveness in relation to the desired treatment. All these issues are addressed by the German pre-standard DIN SPEC 91315:2014–06 (DINSpec), which deals with the safety limits, risk assessment and biological efficacy of plasma sources aimed for medical applications. In this work, a low cost, user-friendly and flexible atmospheric pressure plasma jet (APPJ) device was characterized following the DINSpec guidelines. The device, which is still under development, proved to be safe for medical applications. It is capable of producing an APPJ with low patient leakage current and UV emission, gas temperature lower than 40 °C, production of harmful gases within the safety limits and low cytotoxicity. The most differentiating feature is that the device presented good antimicrobial efficacy even operating at frequency of the order of just a few hundred Hz, a value below that of most devices reported in the literature.**

Index Terms—**low temperature plasma; plasma jet; plasma medicine; DIN SPEC 91315;**

I. Introduction

DURING the last two decades plasma sources aimed to produce atmospheric pressure plasmas (APPs) and their applications have received a lot of attention. Since then, APPs have been used for the treatment of materials, for medical and biomedical applications [1], [2], [3], [4], [5], [6]. Medical and biomedical applications, including dentistry, that employ cold atmospheric pressure plasmas (CAPPs) have advanced significantly and are becoming therapeutic alternatives or part of combination treatment for some conventional treatments [7], [8], [9], [10], [11], [12], [13], [14]. This is happening due to the recent advances in the development of plasma sources that produce cold plasmas with gas temperature close to that of the environment, combined with the generation of a large amount of reactive species among other relevant properties [9], [15], [16], [17], [18].

The treatment of materials or tissues using APPs can be done in basically two ways. One of them is with the plasma impinging/touching the target, which will be called direct treatment, and the other way is with the plasma without any contact with the target (indirect treatment). In a direct treatment the action of the plasma on the material is a combination of physical and chemical processes [19], [20], [21] and is subjected to the type of treated material [22], [23]. The physical action occurs mainly due to the electric current, electric field, the gas temperature and the gas flow velocity [24], [25]. The chemical action is mainly due to the interaction of the reactive species produced by the plasma with the substrate [26], [27], [28]. In indirect treatments only the reactive species are supposed to interact with the target. Of course, in situations in which the plasma source produces a plasma jet, the gas flow can also produce some physical effect on the target due to the gas temperature, even if the plasma does not touch the target [29], [30], [31], [32]. In addition, ultraviolet (UV) radiation can be produced by the plasma source in both direct and indirect treatments [33], [34]. Plasma activated liquids (PAL) also performs indirect plasma treatments on the substrates in contact with the liquids [35], [36], [37], [38]. The PAL action on the substrate is exclusively due to the presence of reactive species in the liquid.

In medical and biomedical applications, some processes are favored when using a direct plasma treatment due to the higher degree of interaction between the plasma and the tissue [34], [39], [40], [41]. However, direct plasma treatments may present some risks to the target tissue, since in this operating mode the plasma has physical properties that can cause damage if the plasma device is not correctly set up, and such damage can be serious for human tissues if above a critical safety level [42], [43], [44]. Electrical current and gas temperature are the main discharge properties to be considered when performing a direct treatment on human tissues, being that the first must not exceed 100 µA-rms in AC mode or 10 µA in DC operation and the second must not exceed 40 °C [45], [46]. Regarding electrical safety, the situation with the accumulation of short duration pulses, as delivered by many APP sources, is so far not so well-documented and regulated. In addition to the safety related to the discharge properties, UV radiation and toxic gasses produced within the plasmas lead to limitations of exposure time to plasma treatment [43], [46].

This work did not involve human subjects or animals in its research.

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Anexo A8: Artigo aceito para publicação na revista *IEEE Transactions on Radiation and Plasma Medical Sciences* (Dezembro/2023) – 2ª página

[47]. The UV radiation directly affects human tissues and the maximum daily exposure (D_{max}) to UV radiation is limited to 3 mJ/cm^2 [48]. The toxic gasses that can be produced by an APP, like ozone (O_3), nitrite (NO_2) and nitrate (NO_3), do not cause damage to the target tissue, but can affect human lungs if their concentration in air is above the safe levels. When working in an environment for 8 hours, the maximum recommended concentration of O_3 in air is 0.055 ppm and accumulation in a treatment room needs to be considered [49], [50]. For NO_2 in the same working conditions the maximum recommended value is 0.019 ppm [51].

To comply with these limitations, any plasma source for medical applications should be characterized to ensure that the above mentioned quantities are within the safety levels. In this way, the DIN SPEC 91315:2014–06 (to be referred as DINSpec from now on) was established, which is a German pre-standard based in part on DIN EN 60601-1, which relates to IEC 60601-1. The DINSpec deals with the safety limits of plasma sources for medical applications and suggests a protocol for risk assessment of plasma sources and establishes basic parameters for checking the biological efficacy of a device [46], [47], [52]. The DINSpec protocol can be applied to any plasma source. However, some parameters like the patient leakage current (PLC) and gas temperature (T_{gas}) are more important when using devices that produce plasma jets since, in this situation, such parameters can increase with the distance from target, instead of only decreasing as with devices that do not employ a gas flow or are aimed to be used for indirect treatment [34], [44], [53].

Although low frequency plasma sources are common for material treatment, in general, the ones aimed to be used in medicine and dentistry operate at frequencies higher than the ones in the electrical line (50/60 Hz) [27], [54], [55]. Some of them use radio frequency (RF) power supplies to generate the plasmas. Others employ power supplies with frequencies ranging from 200 Hz to a few tens of kHz [56], [57], [58]. In the opposite direction, Xaubet *et al* reported the operation of the Magiplas device whose voltage waveform is a 50 Hz sinusoidal burst modulated at 2 Hz, which can be seen as a pulsed signal with a frequency of 2 Hz [59]. Even using a low frequency plasma source, a good antimicrobial efficacy was achieved with such equipment.

The operating frequency of a power supply used to ignite an APP has direct influence on the discharge parameters and behavior [54], [60], [61]. One of the effects is that the discharge ignition tends to be easier at higher frequencies, which allows a reduction in the voltage amplitude required for the ignition. Another one is related to the power dissipated on a discharge (P_{dis}), which is directly proportional to the voltage frequency [62]. This last fact has a direct influence on the gas temperature of an APP, with higher P_{dis} usually leading to higher T_{gas} values. An advantage of plasma sources that operate at higher frequencies is that the effective plasma duration is higher than in those devices operating at lower frequencies. In addition, the production of reactive species can also be enhanced by operation at higher frequencies [63], [64]. Those facts can lead to higher efficiency in the plasma treatment with shorter exposure time. However, the plasma source must be very well designed in this case in order to avoid the excessive heating related to the high frequency operation.

In this work, the results of characterization and antimicrobial efficiency of a low cost and low frequency APPJ device aimed for applications in dentistry and medicine are presented. The low cost of the device is due to the fact that the costs involved in manufacturing the plasma source, which are very low when compared to devices that are already commercially available, such as kINPen® MED, plasma care®, and PlasmaDerm® Flex [58]. The device characterization aimed to check if the device complies with safety requirements for plasma sources to be applied as a medical tool. Such characterization was accessed by carrying out a set of measurements suggested by the DINSpec. The biological assays with different sets of bacteria and fungi, as proposed by the DINSpec, were also carried out in order to ensure that the plasma jet produced with the device has antimicrobial properties. Experiments were also performed in order to ensure that the APPJ has no cytotoxic effects.

II. Materials and Methods

A. Characterization of the plasma sources for medical applications

The characterization of the plasma source used in this work was carried out performing a set of measurements suggested by the DINSpec, which consists mainly of:

- ensure the rms values of electrical current reaching the patient (PLC as abbreviation for the so-called patient leakage current) → must not exceed 100 µA-AC
- determine the effective UV radiation emitted by the plasma → will define the maximum exposure time to plasma treatment
- measure the temperature of the gas (T_{gas}) and the target (T_{targ}, a copper plate) → it is not recommended that the maximum value of T_{gas} exceeds 40 °C and the maximum value of T_{targ} must be less than 40 °C
- the generation of ozone (O_3), nitrogen oxide (NO) and nitrite (NO_2) gases produced by the plasma → define the maximum exposure time to plasma treatment and minimum distance from the plasma outlet
- check for antimicrobial efficacy, cellular toxicity, etc.

B. Portable plasma device and characterization processes

Figure 1 a) shows an overview of the plasma source and the main scheme for obtaining the waveforms of applied voltage (V), discharge current (i) and PLC when producing a plasma jet impinging on a copper (Cu) target. Figure 1 b) presents a detailed view of the dielectric barrier discharge (DBD) reactor which integrates the plasma source. The development of the plasma source used in this work started in our research groups with the discovery of the plasma jet transfer technique by Kostov *et al* in 2015 [65]. In the same year, a study demonstrated that the plasma generated at the tip of a long tube using the newly developed technique had the potential to inactivate microorganisms [66]. After that, many studies were conducted using the device developed by our teams [67].

Anexo A9: Artigo publicado na revista *Microorganisms* (Janeiro de 2024) – 1ª página

microorganisms

MDPI

Article

Cold Atmospheric Helium Plasma in the Post-COVID-19 Era: A Promising Tool for the Disinfection of Silicone Endotracheal Prostheses

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Abstract: Despite the excellent properties of silicone endotracheal prostheses, their main limitation is the formation of a polymicrobial biofilm on their surfaces. It can cause local inflammation, interfering with the local healing process and leading to further complications in the clinical scenario. The present study evaluated the inhibitory effect of cold atmospheric plasma (CAP) on multispecies biofilms grown on the silicone protheses' surfaces. In addition to silicone characterization before and after CAP exposure, CAP cytotoxicity on immortalized human bronchial epithelium cell line (BEAS-2B) was evaluated. The aging time test reported that CAP could temporarily change the silicone surface wetting characteristics from hydrophilic (80.5°) to highly hydrophilic (<5°). ATR-FTIR showed no significant alterations in the silicone surficial chemical composition after CAP exposure for 5 min. A significant log reduction in viable cells in monospecies biofilms (log CFU/mL) of *C. albicans*, *S. aureus*, and *P. aeruginosa* (0.636, 0.738, and 1.445, respectively) was detected after CAP exposure. Multispecies biofilms exposed to CAP showed significant viability reduction for *C. albicans* and *S. aureus* (1.385 and 0.831, respectively). The protocol was not cytotoxic to BEAS-2B. CAP can be a simple and effective method to delay multispecies biofilm formation inside the endotracheal prosthesis.

Keywords: multispecies biofilm; non-thermal plasma; endotracheal tubes; silicone prosthesis; COVID-19

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1. Introduction

Tracheal stenosis is a clinical condition characterized by a reduction in the central airway diameter through a congenital or acquired pathological process. The COVID-19 pandemic produced a high number of patients submitted to intubation and prolonged mechanical ventilation worldwide, resulting in higher percentages of laryngotracheal stenosis [1–4]. Commonly, patients with tracheal stenosis after COVID-19 cannot undergo tracheal resection since maturation of the stenosis, as well as a reduction in inflammation, needs to be achieved before the procedure [5]. Additionally, the recurrence of tracheal stenosis is a risk during the first three months after surgery [6]. The prevalence of post-intubation tracheal stenosis in retrospective studies ranges from 6% to 20% and that of post-tracheostomy from 0.6% to 21% [7].

Patients with benign tracheal stenosis or tracheal tumors who are not eligible, either temporarily or permanently, for definitive treatment via tracheal surgical resection are considered for airway stenting to maintain airway patency. Silicone airway stents are widely used for their safety, patient tolerance, easy handling, and lower cost than that of self-expanding stents. The silicone T-tube was introduced in 1965 [8]. It requires a tracheostomy for anchoring the horizontal limb of the prosthesis that is occluded by a removable silicone cap. The straight-studded silicone stent was introduced in 1990 [9] and does not require a tracheostomy. Both are used as endoluminal support for the trachea, enabling breathing and phonation through the natural airway, thus improving quality of life [10].

Medical-grade silicone (MGS) is the primary material in endotracheal prostheses. MGS has attractive properties such as chemical inertness, water and temperature resistance, biocompatibility, and flexibility [11]. The constant contact between the inhaled air passing through the airway prosthesis and the tracheal underlying mucosa promotes the accumulation of biofilms composed of bacteria and fungi. The interaction between the polymicrobial biofilm and the surrounding mucosa can potentially aggravate the severity of the stenosis [10,12,13]. *Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Candida albicans* are the main microorganisms in the polymicrobial biofilm formed inside the patient's silicone prosthesis implant [14]. Biofilm formation usually takes approximately seven days, and severe complications such as pneumonia or sepsis can occur [15]. On the other hand, the MGS degradation promoted by the polymicrobial biofilm is progressive, requiring the prosthesis to be changed every 6 to 12 months after implantation. This can negatively impact the patient's quality of life and induce higher costs [16,17].

Previous studies focused on modifying the MGS surface to promote antimicrobial activity. Silver ions and nanoparticles were used as an alternative approach and were effective against *Escherichia coli*, *P. aeruginosa*, and *Staphylococcus aureus*. The limitations of these methods are the potential cytotoxicity and mucus accumulation on the silver-coated surface, which can reduce its antimicrobial properties [18].

Cold atmospheric plasma (CAP) stands for a low-temperature (<40 °C) gas discharge plasma with extensive applications in the medical and biomedical fields [19–22]. The literature has already reported microbial inactivation due to the reactive oxygen and nitrogen reactive species (RONS) and UV radiation produced by CAP. The antimicrobial effect was observed on several species of bacteria and fungi, including *S. aureus*, *P. aeruginosa*, and *C. albicans* [23–25]. CAP can be delivered through long, flexible plastic tubes [26,27], enabling clinical use. Moreover, previous studies reported that CAP has low toxicity to mammalian cells [28,29].

This pre-clinical in vitro study focused on the effect of the Helium-CAP over monospecies and multispecies biofilms grown on the MGS surface in a laboratory setting, which mimics what is clinically observed inside the T-tube. The CAP treatment's effects on biofilms' viability were assessed, along with the possible surface changes on the MGS caused by CAP. Lastly, the cytotoxicity of the protocol was tested using a BEAS-2B immortalized human bronchial epithelium cell line. The motivation of this study is to develop a protocol for CAP treatment that can be applied directly to the external limb of the T-tube during outpatient visits, aiming to control the biofilm proliferation in the lumen of the silicone prosthesis. Such features can prolong the prosthesis' durability, reduce the interaction between the biofilm and the adjacent mucosa, and enhance the local healing process.

2. Materials and Methods

The MGS samples were prepared in disks measuring 8 mm in diameter and 2 mm in height. The specimens had the same chemical composition as T-tube implants. They were used to perform the surface characterization of silicone before and after the plasma treatment and the microbiological assays. Before treatment, all samples were washed in an ultrasonic bath by immersion for 10 min in water and 10 min in isopropyl alcohol. After that, another step of water washing was performed for 10 min. Finally, the samples were