

## ANALYSIS OF PARAMETERS AND IDENTIFICATION OF CHEMICAL SPECIES PRESENT IN PLASMA JETS FROM THE EXCITATION OF HELIUM AND ARGON FOR DENTAL PURPOSES

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*Ricardo Anderson da Cruz*

Universidade Federal do Rio Grande do  
Norte, Natal, Brazil

<https://www.linkedin.com/in/ricardo-anderson-cruz-091255a5/>

<http://lattes.cnpq.br/8765117400151471>

*Angelo Roncalli Oliveira Guerra*

*Maria Luiza de Medeiros Cachina*

*Ana Luiza Ohara de Queiroz*

*Nicolas Guedes Nunes*

*Leticia Amanda Fontes de Moraes*

*Ana Beatriz Villar Medeiros*

*Samara Dália Tavares Silva*

*Rafael Cavalcanti Contreras*

*Custódio Leopoldino de Brito Guerra Neto*

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**Abstract:** The study Atmospheric pressure plasma technology has become a prominent area of research due to its diverse applications in health. Areas such as pollution control, materials processing, electrochemistry and engineering as a whole have this technology as very relevant. The moment in which the chemical species in the plasma reach the treated surfaces still requires studies, so the current search aims to obtain deeper knowledge for applications in dentistry. Special attention is paid to the biological and surface effects of plasma-generated reactive species. It is the research aims to use the Optical Emission Spectroscopy (EEO) technique, a tool for plasma diagnosis, detection of reactive species and their identification, as well as the analysis of how they behave according to the parameters adopted in the process. To obtain plasma by Dielectric Barrier Discharge (DBD). To carry out this analysis, it was necessary to follow the steps search of articles to carry out the literature review, laboratory experiments to obtain data and analysis of reactive species. The results include graphs of the plasma spectrum, with its respective parameters, as well as the identification of its chemical species, with thermal images of some samples under incidence of the plasma plume, considered critical for dentistry. It is concluded that the study of reactive species is very important for several areas, especially dentistry, as the chemicals species in addition to carrying out superficial modifications, increasing the applied energy, inactive unwanted microorganisms, with sterilizing effect.

**Keywords:** Atmospheric plasma, dielectric barrier discharge, optical emission spectroscopy, reactive species, dentistry.

## INTRODUCTION

Plasma can exist in many forms and can be created in different ways. In many technological applications it can exist at low gas pressures (thermal plasma), for example, metal surface treatment, as well as atmospheric pressure (non-thermal plasma), lightning is an example of atmospheric pressure plasma (KOSTOV, KG, 2022 and Brito et al, 2022).

The terms thermal and non-thermal are not very precise. In nonthermal plasma, the cooling of uncharged ions and molecules it is more efficient than electron energy transfer, and the gas remains at a low temperature. For this reason, non-thermal plasma is also called non-equilibrium plasma, that is, the particles loaded people are always looking to balance themselves. In a thermal plasma, on the other hand, the flow of energy from electrons to heavy particles balances the flow of energy from heavy particles to the environment, only when the temperature of these particles becomes almost equal to the temperature of the electron (FRIDMAN et al., 2008 and KOSTOV, KG, 2022 and Brito et al, 2022).

The plasma ford charges in Dielectric Barrier (DBD) is a representative of non-thermal plasmas, with advantages of highly transient discharges, low temperatures and high amounts of reactive species. The typical DBD plasma can be formed by two electrodes, where at least one is coated with dielectric material, a form of resistance for the material, which is suitable for the atomization of volatile species (ZHANG et al., 2019).

When the amplitude of the electric field is sufficient to break the dielectric strength, discharge through the gas is formed, generating neutral and electrically charged species such as electrons, positive ions, negative ions, atoms and molecules (PAIVA et al., 2019 KOSTOV, KG, 2022 and Brito et al, 2022).

The DBD plasma jet has been used in recent research in the modification of thermally sensitive materials, noble metals, sterilization of bacteria and fungal decontamination (NASCIMENTO NETO et al., 2015 KOSTOV, KG, 2022 and Brito et al, 2022), in addition, there are researchers studying promising applications in the area of dentistry, applying plasma directly to eliminate one of the most significant microorganisms in the development of cavities, *Streptococcus Mutans* (GHERARDI; TONINI; COLOMBO, 2018), including modifications to the surface of dental implants, adhesion of protective materials to teeth, endodontic treatment and whitening (ŠANTAK et al., 2017).

Plasma treatment is also possible in other areas, such as dermatology, where the professional makes use of plasma jet “pens”, in filamentary areas, to promote the “regeneration” of the skin, through the production of heat, inducing thermal “damage” on the surface of the skin causing a new production of collagen, elastic fibers, modification and restructuring of the dermis (GUERRA et al., 2018 and MEYER, PF et al., 2024). Another perspective in this line of research is the use of plasma as a facilitator of skin hydrophilicity, being used as a pre-cosmetic facial procedure to hydrate the skin. This plays a significant role in improving the effects of various products available on the cosmetics market (GUERRA et al., 2018 and MEYER, PF et al., 2024).

The chemical character of DBD plasma is produced due to collisions, which form basic products such as atomic oxygen, metastable oxygen and nitrogen, with subsequent reactive collisions that produce a mixture of neutral, ionic and/or excited species (NASCIMENTO NETO, 2013 and GUERRA et al., 2018) which can act as bactericide, disinfection and healing of the skin, blood coagulation, modification of polymeric materials and thermally sensitive

materials for medical therapy, applications in dentistry, among other actions (GUERRA et al., 2018 and NISHIME et al., 2017 and MEYER, PF et al., 2024).

Therefore, DBD plasma jets present several very important advantages and conditions for applications in the biomedical area, as it is formed by low power, leaving the jet temperature close to that of the environment, and also, due to the use of the dielectric, it reduces the formation of arches (NASCIMENTO NETO, 2013 and GUERRA et al., 2018 and MEYER, PF et al., 2024).

To study this type of plasma, several detection techniques are used, such as Optical Emission Spectroscopy (EEO), which is a typical technique with high precision and without intrusiveness, recording the emission intensity (EI) of each length of plasma. wave (ZHANG et al., 2019); providing information on the kinetics of plasma generation, the active species and the relative quantity of species, as energy react with the working gas or gas mixtures during physicochemical reactions in the plasma (KOSTOV, KG, 2022 and Brito et al, 2022).

This work had as its main objective the use of the EEO technique to detect active species and their identification, as well as the analysis of the parameters for obtaining DBD plasma for applications in dentistry, aiming to obtain a deeper knowledge regarding the production kinetics of atmospheric DBD plasma, thus helping to systematize the use of this technique in dentistry.

## LITERATURE REVIEW

The physical states of matter (Figure 1) correspond to the ways in which matter can appear in nature. These states are defined according to pressure, temperature and above all, the forces acting on the molecules. Matter is made up of small particles (atoms and molecules), corresponding to everything that

has mass and that occupies a certain place in space. It can come in four states: solid, liquid, gas and plasma.

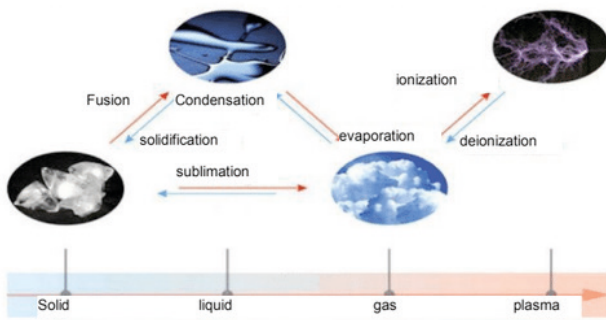


Figure 1: The four states of matter adapted from (C.; C.; J., 2013).

The term plasma was introduced by Irving Langmuir in 1928 to describe the state of matter, Meghnad Saha, an Indian physicist, estimated that more than ninety-nine percent of the universe is composed of plasma (KOSTOV, KG, 2022).

Plasma can be generated artificially through various methods for specific applications. Fluorescent lamps, neon advertising signs and plasma are typical examples that use ultraviolet light emitted by plasma.

The expression “plasma” applies to a gas that, through ionization, contains neutral and electrically charged species such as electrons, positive ions, negative ions, atoms and molecules, and can also be characterized by being a set of particles, charged and neutral, quasi-neutral, which exhibit collective behavior (KOSTOV, KG, 2022).

In recent years, cold plasmas have attracted much attention in various fields of science and technology (Figure 2). Atmospheric pressure plasma discharges are practically used due to their simplicity and low cost. Plasma jet is used for applications such as ozone production, environmental pollution control, water treatment, ignition and combustion, surface treatment, medical treatment, surgery, blood clotting, sterilization, bacteria removal, cancer

therapy, dentistry, oncology, agricultural and biological (ONO, 2016; TANAKA et al., 2017).

|  |  |
|--|--|
| <i>Plasma in medicine</i>  | <i>Plasma in agriculture</i>                           |
| Cancer treatment<br>wound healing<br>regenerative medicine<br>dental treatment | Insect elimination; sterilization;<br>improving growth |
| <i>Microfabrication</i>  | <i>Environmental science</i>                           |
| Engraving; Chemical vapor deposition   | Air pollution treatment; Water Pollution Treatment     |

Technology and plasma

Figure 2: Wide variety of applications in plasma technology (Adapted) (TANAKA et al., 2017).

## DISCHARGE IN DIELECTRIC BARRIER:

Dielectric barrier discharge has a long history. Its discovery is attributed to Werner von Siemens, who in 1857 developed a discharge tube for ozone production (GUERRA et al., 2018).

Dielectric barrier discharge (Figure 3) occurs when a high voltage is applied in the region between two metallic electrodes when at least one dielectric is inserted between them to form the plasma (NASCIMENTO NETO, 2013 and GUERRA et al., 2018).

When high voltage is supplied to the electrodes, which can be from alternating, direct or radio frequency voltage sources (BUDA et al., 2015), electrical charges accumulate on the surface of the dielectric, a process that occurs due to its polarization. At a certain point, these accumulations are enough to break the dielectric strength of the gas and a micro discharge, after this, electrons are ejected towards the anode, reducing the charge concentration and consequently the point electric field, which originated this discharge, and the microfilament extinguishes (Lira / Pluritec, 2020).

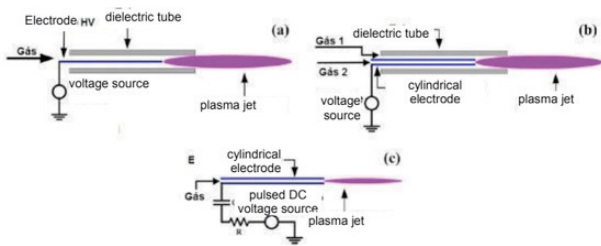


Figure 3 - Schematic drawing of DBD plasma jet devices (NASCIMENTO NETO, 2013).

The use of dielectric is essential for the operation of a DBD device, as it limits the discharge current and distributes it over the entire electrode area, preventing the formation of arcs (NASCIMENTO NETO, 2013). This discharge constitutes a safe and economical method of generating non-thermal plasma at atmospheric pressure.

There are two different operating regimes for DBD plasma: the filamentary and the diffuse regime. In most industrial applications with plasma, the discharges produced by the DBD technique are carried out in filamentary mode although, in the diffuse regime, the discharge is more homogeneous, without the formation of arcs.

### FILAMENTARY REGIME:

The DBD plasma in filament regime appears at the moment when the breakdown voltage is reached, having as its main characteristic a high current density, due to the low uniformity of the micro discharges. DBD discharge in a filamentary regime has a range of varieties of free radicals, molecules, atoms, electrons with high energy and ultraviolet radiation, which when interacting with the surface of materials can cause changes in the surface topography (roughness), as well as increased the ability of a liquid to maintain contact with a solid surface, called wettability, due to the formation of compound functional groups (ZHANG, Y. et al., 2016 and ZHANG, B. et al., 2019). However, all of this occurs

non-uniformly on the surface of the material due to the lack of energetic uniformity in the plasma produced (Lira / Pluritec, 2020).

### DIFFUSE REGIME:

After the electrical breakdown that occurred in the filament regime, if the number of micro discharges and if they are distributed over the entire surface of the dielectric, it will give rise to the DBD plasma in the diffuse regime. The transition from a filamentary discharge to a diffuse discharge mode can happen when the DBD discharge operates for a few seconds, being controlled by the frequency and voltage pulse width. There is the possibility of stabilizing DBD's in a diffuse regime, providing special conditions for the plasma generation process, such as the electrode and/or dielectric material, distance between electrodes and the gas mixture, which play an important role in plasma ignition. DBD in diffuse regime (KOSTOV, KG, 2022 and Brito et al, 2022).

The diffuse regime is more easily obtained in gases such as helium, neon and nitrogen or mixtures of inert gases with molecular oxygen, nitrogen, among others (FALAHAT et al., 2018). It is an important regime for the process at atmospheric pressure, when uniformity of the changes generated by the plasma on the materials is desired. However, if there is a concentration of impurities or instability in the operating parameters in the discharge environment, this may lead to the discharge entering a filamentary regime, which in certain applications is not suitable (NASCIMENTO NETO, 2013 and ZHANG, B. et al., 2019).

The visual difference between the two regimes is present in Figure 4, in which the diffuse regime is present in (a), in (b) where the discharge filaments are well spaced and apparent, justifying the high current density (KOSTOV, KG, 2022).

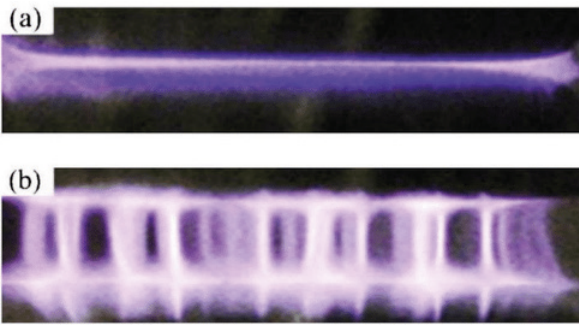


Figure 4: Images of two typical discharges: (a) diffuse DBD and (b) filamentary DBD (Liu et al., 2014).

In summary, DBD plasma jets have several advantages. Firstly, due to the low power delivered to the plasma, the jet temperature remains close to the ambient temperature. Secondly, due to the use of the dielectric, arcing is reduced. Also, it has a high density of chemically active species (for example, OH and atomic oxygen) that are oxidants and therefore very efficient for bacterial inactivation. These conditions are very important for applications in the biomedical field, including the production ozone treatment, treatment of polluting gases and toxic waste, excitation of CO lasers<sup>2</sup>, sterilization of materials, deposition of thin films and modification of surfaces (KOSTOV, KG, 2022).

## PLASMA APPLICATIONS (DBD)

### FLUORESCENT LAMPS

The DBD discharge, when operated on noble gases or mixtures of noble gases with halogens, emits intense ultraviolet radiation, UV. Therefore, this discharge is an efficient and cheap source of UV light. In fluorescent lamps, the spectrum emitted by gases is converted by the coating of the tubes that contain them into a spectrum predominantly of visible light. Thus, fluorescent lamps are quite efficient and economical, compared to incandescent lamps (KOSTOV, KG, 2022).

## SOIL TREATMENT AND AGRICULTURE

Research has demonstrated that seed treatment with DBD plasma, using working gases such as Argon (Ar) and Nitrogen (N), produces significant results in the germination rate, causing an increase of 10-20%, and also in plant development (JI et al., 2016;

Also, atmospheric DBD plasma has been presenting itself as a promising technique in the field of agriculture, as it presents a high rate of ozone formation and OH radicals, which promote soil deacidification, that is, the degradation of acidic compounds that contaminate the soil (KOSTOV, KG, 2022).

## STERILIZATION

Sterilization targets bacteria, viruses and fungi and can be achieved by heat, chemicals, irradiation and filtration. Autoclaves are widely used to sterilize medical and biological instruments, and use steam heated under high pressure (typically 121°C at 100 kPa). Ethylene oxide gas is a common chemical method of sterilization, but it is also toxic to humans and therefore residual levels of ethylene oxide must be as low as possible (TANAKA et al., 2017).

Thus, researchers developed plasma sterilization, which consists of exposing materials containing microorganisms to ionized and/or excited gas, where, it contains reactive species generated that can promote the complete elimination of microorganisms, present in a given material in a few minutes of treatment, offering advantages in relation to others already used, as they are more efficient in reducing the microbial load, in addition to developing in temperatures close to room temperature, being able to sterilize thermosensitive materials, and not using toxic gases (Sakudo A, Yagyu Y, Onodera T, 2019).

## TREATMENT OF LIVING TISSUE

The use of DBD in living tissues, such as human skin, is also possible due to the low temperature of the plasma, which allows the treatment of pathogenic agents directly on the skin, also acting as an anti-inflammatory.

Also, atmospheric plasma accelerates blood clotting, serving to act in situations that include post-surgery, post-dermatological incision and therapeutic procedures involving bleeding (NUNES; GUERRA, 2018 and MEYER, PF et al., 2024).

## DENTISTRY

Preparation or repair of tooth cavities before filling is done by removing necrotic, infected and demineralized tissue through mechanical drilling, laser techniques or ozone treatment. During mechanical drilling and laser treatment, heating occurs and most of the time it is useful.

However, the vibrations are induced and, therefore, cause pain in the patient. Furthermore, these methods are often destructive: excess healthy tissue must be removed to ensure the cavity is free of bacteria. As a result, the remaining tooth structure is weakened and prone to fractures. The accumulation of such problems, in addition to causing pain for the patient, can heat the pulp of the tooth, killing it, making it necessary to remove it. An alternative painless and non-destructive method is ozone treatment. Ozone is a powerful oxidant, which is used as a disinfectant in the food, medicine and water treatment industries (KOSTOV, KG, 2022).

Another painless, tissue-sparing approach that differs from ozone therapy is the use of cold atmospheric plasma. The principle of plasma treatment is the inactivation of bacteria in dental biofilms in a non-contact manner. The advantage of plasma treatment is that it allows irregular structures and narrow channels within the diseased tooth to

be cleaned. The treatment is superficial and non-destructive; does not cause bulk material removal. In contrast to lasers, plasmas can access small irregular cavities and fissure spaces, as they are at room temperature and do not heat the tooth pulp. The application of non-thermal plasmas in the treatment of caries is a challenging multidisciplinary research problem, which requires knowledge of both plasma physics/chemistry and dentistry. As it is non-destructive to human tissues, it can be applied in vivo. Due to its room temperature, plasma does not cause significant heating of the dental pulp and has a good capacity to kill bacteria. (LATA, S. et al., 2022).

## PLASMA PARAMETERS

In DBDs plasmas it has been demonstrated that the generation of diffuse or uniform plasmas is possible with the control of specific operational parameters, such as reactor geometry, feed gas (noble gases, diatomic or air), dielectric material and control of voltage and frequency. excitement. Table 1 shows typical plasma parameter values (KOSTOV, KG, 2022).

|                        |                           |
|------------------------|---------------------------|
| Voltage                | 5 - 100 Kv                |
| Frequency              | 50Hz - 1MHz               |
| Electron density       | $10^{14} \text{ cm}^{-3}$ |
| Electronic temperature | 1 - 10 eV                 |
| Degree of ionization   | $10^{-1}$                 |
| Pressure               | 1 bar                     |

Table 1 - Typical parameters of a Dielectric Barrier Discharge. Source: (SOUZA, 2018; WANG; ZHANG; WANG, 2012).

Thus, DBDs can have cylindrical or flat geometry and configurations that can vary their large-area discharges at a single micro discharge. Depending on the jet configuration and electrical excitation, different discharge characteristics can be obtained. They are generally supplied by high voltage (High Voltage - HV), with alternating current

(Alternating Current - AC), normally sinusoidal. With a few exceptions, they run in filamentary mode, i.e. a beam of micro discharges which more or less uniformly fill the discharge volume in a non-repetitive manner. To the micro individual discharges cells can last a few tens of nanoseconds and have a thickness of the order of 100  $\mu\text{m}$  at atmospheric pressure. The diameter of the flow tube/nozzle is often 1 to 4 mm (DI LECCE, 2014; XIONG et al., 2013 and KOSTOV, KG, 2022).

### SOURCE

The gas ionization process normally takes place in the presence of a high voltage source, which produces enough energy to overcome the dielectric strength of the medium and cause an electrical discharge capable of removing, adding or exciting electrons, causing successive collisions generating photons (light). This method follows Paschen's law (NUNES; GUERRA, 2018).

According to this law, the electrical breakdown voltage of a gas, at constant temperature, between two electrodes is a function of the product of the pressure and the distance between the electrodes, depending on the type of gas. It is described by the following mathematical equation:

$$V_{\text{break}} = \frac{Bpd}{\ln(Apd) - \ln(\gamma)} \quad (1)$$

Where, A and B are constants that depend on the gas, p is the pressure value, the distance between the electrodes and  $\gamma$  the secondary electron emission coefficient. Figure 5 shows the curves corresponding to Paschen's law in some static gases. In which, the minimum voltage necessary for a discharge to occur decreases with the increase in the pressure

product with the distance between electrodes up to a minimum value, known as the Paschen minimum (RASTEIRO, 2016).

Therefore, the potential difference has a significant effect on the formation of plasma jets. Commonly, the power frequency ranges of voltage sources are divided into low/high frequency ranges, radio frequency and microwave, with this, it can be controlled from the length, intensity to the temperature of the jets (NASCIMENTO NETO, 2013 and KOSTOV, KG, 2022).

Due to the need in the field of dentistry, to generate and apply plasma with control of the temperature rise of the samples, a high voltage and high frequency source with a specially designed purpose was developed at the Health Technological Innovation Laboratory (LAIS). for this purpose - it is a pulse duration modulator.

This modulator is an electronic circuit that switches the high voltage and high frequency generator which is used to generate the plasma. The alternating voltage signal generated by the source has a periodic format with an average energy per cycle equal to zero, that is, it is an approximately square symmetric signal, in relation to zero, with adjustable amplitude between 0 and 12 kV peak to peak (GUERRA NETO et al., 2024).

If the source power control is adjusted to 100%, the alternating signal generated by it has the "square" shape, in Figure 6, over time (the small flank oscillations are caused by the resonance, caused by the distributed inductance and capacitance in the source's high voltage circuit). If we observe this same signal, with a longer sweep time selected in Figure 7.

As it was previously mentioned, this signal needs to have its amplitude adjusted (changing the electric field inside the plasma generating pen) above the Paschen minimum, below which the passage of electric current



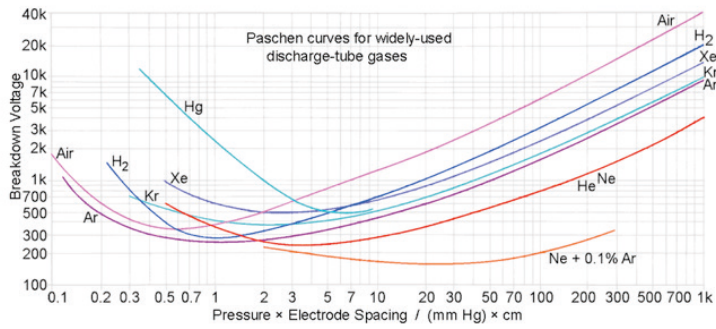


Figure 5: Paschen curves for different gases (RASTEIRO, 2016).

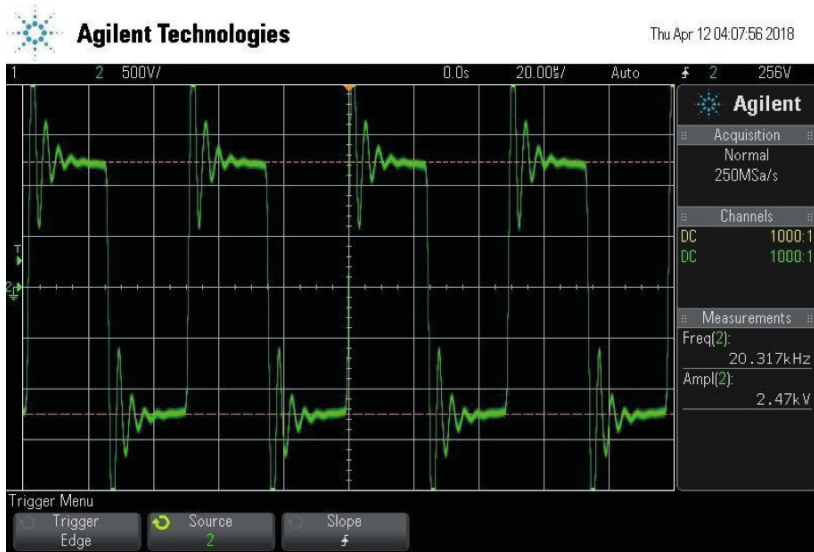


Figure 6: Waveform generated when the power control is adjusted to 100%.

Source: Prepared by the author.

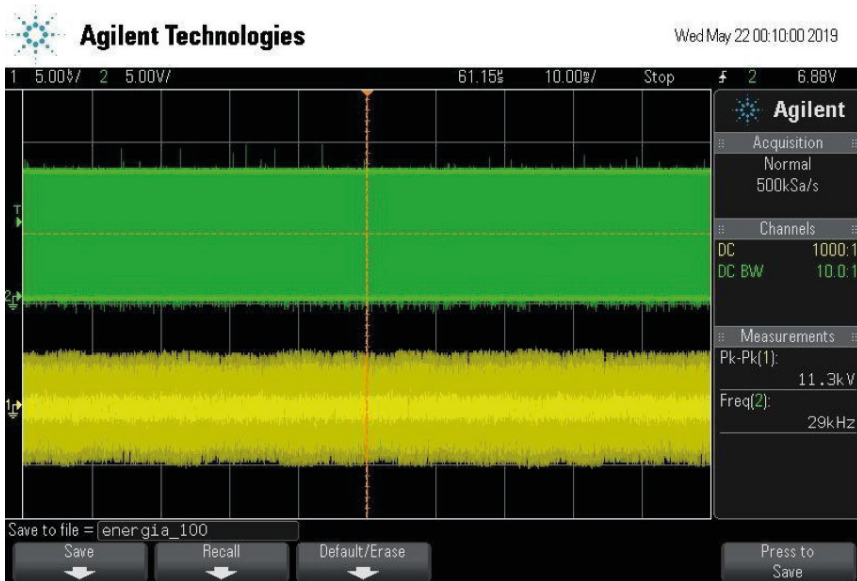


Figure 7: Signal observed on the oscilloscope with a sweep time much longer than the period of the alternating signal generated and with the power control set to 100% (in yellow the voltage and in green the current).

Source: Prepared by the author.

ceases and therefore the ionization of the gas and plasma generation.

## PLASMA REACTIVE CHEMICAL SPECIES AND THEIR FUNCTIONS

Plasma action produces reactive species such as molecules containing reactive oxygen and nitrogen, including nitric oxide (NO), superoxide (O<sub>2</sub>), hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), oxygen (O<sub>2</sub>), ozone (O<sub>3</sub>) ions of helium (He), argon (Ar) and even the hydroxyl radical (OH) which are considered the most important actors in the biological effect of plasma through interaction with living matter. It was assumed that most important biochemical processes were completely dominated by large molecules such as proteins, carbohydrates and lipids. However, it was discovered that reactive species act as part of the complex cellular communication system in aerobic biology (LU et al., 2016; NERETTI et al., 2018).

Thus, reactive species are important in the various applications of DBD plasma. Many researchers have measured the processes of density, spatial distribution, lifetime, and production and loss of reactive species in plasma using several techniques. It is worth highlighting EEO, which provides the spectrum of the plasma containing its chemical species and their respective intensities.

## OPTICAL EMISSION SPECTROSCOPY

EEO is often used to diagnose atmospheric pressure plasma as it is a highly sensitive, non-invasive and simple technique. An optical spectrum emitted by the plasma provides important information on the species present in the jet, such as the atomic and molecular species and ions formed, identifying a relative quantity represented by the luminescent intensity associated with them (ABDEL-FATTAH et al., 2017; SOUZA, 2018).

This technique is essentially based on the analysis of light emitted by different species,

such as neutral or ionized atoms, radicals or molecules from the plasma discharge medium. Furthermore, EEO is used for the characterization of excited species, the determination of the photon flux as a function of wavelength and as a diagnosis of the charged particle beam (FALAHAT et al., 2018).

One of the advantages of EEO is that the concentrations of various excited particles can be obtained from the detected absolute irradiance, if the corresponding emission lines are optically thin. According to the EEO principle, the emission intensity is proportional to the density of excited states. A spectral line can be represented as functions of intensity (I), wavelength ( $\lambda$ ) and top-level species density (N) (ROY; TALUKDER; CHOWDHURY, 2017).

## MATERIALS AND METHODS

To carry out this analysis, it was necessary to follow the steps of searching for articles to carry out the bibliographic review, experiments to obtain data and analysis of reactive species and results obtained.

Thus, for the development of this work, the following research bases were used, US National Library of Medicine National Institutes of Health (PubMed), Scientific Electronic Library Online (SciELO), SCOPUS, National Center for Biotechnology Information (NCBI), Portal Periódicos of the Coordination for the Improvement of Higher Education Personnel (CAPES) and Portal Periódicos of ``Universidade Federal do Rio Grande do Norte`` (UFRN) were researched from July to September 2019. Using the following keywords “plasma technology” or “plasma in dentistry” and “plasma emission spectra” and “optical emission spectroscopy” and “discharge by dielectric barrier” in combination with “biomedical applications”.

The following inclusion parameters were applied for the search and selection of studies:

scientific articles published in journals between 2000 and 2019, available in English/Portuguese and that met at least two keywords “plasma technology” or “plasma emission spectra”. Texts that were not available in full were excluded from the study.

Furthermore, after researching the topic in digital libraries to search for articles, we used some automatic suggestions from electronic databases that were not, initially, directly available and connected to the keyword, in order to include articles correlated to the research topic.

Subsequently, to select the publications, the summary, introduction and conclusion of each article found were read in order to verify their correspondence with the keywords. 123 articles were selected, of which 85 met the search requirements.

For the present study, plasma generating equipment was used with sufficient flexibility to meet various research demands at UFRN's LAIS using a DBD plasma jet.

With the purpose of understanding the behavior of plasma in dentistry and analyzing the temperatures of the samples and reactive chemical species present in the DBD plasma, under atmospheric pressure, varying the voltage, frequency and energy parameters, it was a high voltage and high frequency source is used with a pulse duration modulator, in which it is allowed to vary the applied voltage from 0 to 12 KV (peak to peak), frequency from 26.3 to 45 kHz and energy from 0 to 100 %. Where, it is connected to the plasma reactor which offers the possibility of obtaining plasma with any working gas.

Therefore, He and Ar were chosen as working gases, which are introduced into the rear of the reactor through a mass flow controller with a maximum limit of 15 standard liters per minute (L/min) flowing through a circuit. Figure 8 shows the schematic representation of the entire experimental

setup with the typical view of the designed plasma jet, gas container and spectrometer.

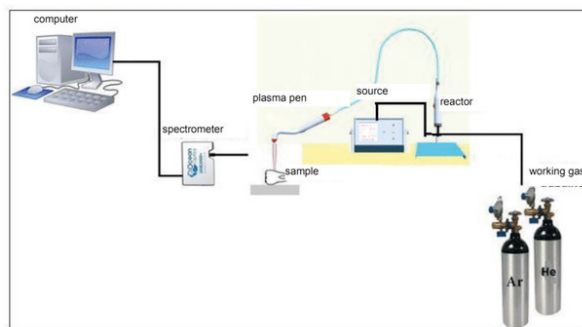


Figure 8: Schematic representation of the experiment configuration. Source:

Prepared by the author.

When the helium/argon flow is passing through the reactor, where electrodes are present, one of which is coated with dielectric material, an essential characteristic of the DBD system, an ionization wave directed to the cathode is generated, where the plasma jet is directed to exit from the pen, conventionally called a Split pen, as the plasma formation does not occur in it, but in a separate reactor, and becomes a visible plume of the plasma jet.

For the identification and labeling of atomic and molecular emitting species, measurements were carried out based on optical emission emitted by the plasma jet, in which was collected perpendicular to the jet by a lens (focal length: 20 mm), through an optical fiber, in the entrance slit of the Ocean-Optics FLAME-S-UV-VIS-ES spectrometer, which has an optical resolution of 0.1 to 10 nm (FWHM); this was achieved using a LAIS computer, equipped with the appropriate software (Ocean view), for both steering and acquisition.

All measurements were performed at the point 5 mm below the plasma jet orifice.

Spectrometer collection (Figure 9) occurs when the light from the plasma travels through a path until it is converted into computational data, in the form of a graph.

Where, the light emitted by the plasma jet enters the spectrometer through the fiber optic connector. Then, mirror 1 reflects this light to the collimator, which separates the wavelengths present in the emitted light. Afterwards, the spectrum formed is sent to mirror 2, which reflects it to the detector. The detector, in turn, converts the optical data into computational data that is read by the Spectra Suite software (SOUZA, 2013).

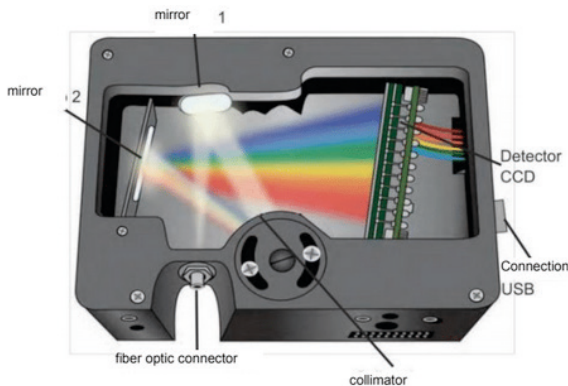


Figure 9: The optical path that the light from the plasma travels (SOUZA, 2013).

The spectral responses of the parameter variation steps involved in this research were verified in real time during the plasma generation process. To make this possible, all tests in the DBD reactor were carried out with minimal ambient light, as the electromagnetic emissions of this type of plasma in the visible range are very low, making it difficult to capture images with ambient light. That's why all The lights in the room where the processes were carried out were always turned off during data acquisition, so as not to cause errors in EEO acquisition.

In this work, thirty-five tests were carried out, selecting the most critical results for dentistry, these being with a tooth made of resin and a moistened polyurethane sponge, simulating the gum, seeking to get closer to a real situation in dentistry.

The treatments consisted of subjecting the surface of the samples to an atmosphere of plasma generated in the reactor, where the working gas was helium or argon. In which, the DBD system was subjected to different voltages, frequencies and energies for analysis of their respective reactive species and temperature control of the samples.

It is worth noting that the distance between the plasma pen and the samples was fixed at 5 mm, as well as the focal distance of the spectrometer at 20 mm, using limiters produced in appropriate software and printed on the LAIS 3D printer, avoiding saturation errors. in data acquisition.

## RESULTS AND DISCUSSIONS

Plasma production requires ideal conditions and parameters for it to be formed. In the study of this work, the DBD system was subjected to different voltages, frequencies and energies, checking the reactive species present in it and analyzing the temperatures of the samples. Below, in Table 2, the experimental values of the variations of each of the studied parameters are presented.

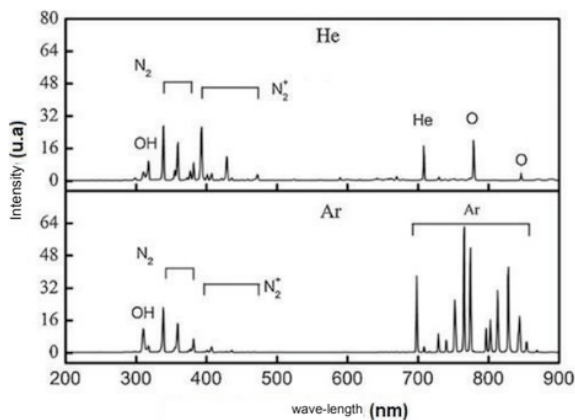
It is important to mention that, by applying the parameters, the dielectric strength of the gas is broken, consequently, a wide variety of free radicals, molecules, atoms, electrons with high energy and ultraviolet radiation are produced in the middle of the discharge of the DBD plasma jet. The ionized state in gas atoms and molecules accumulates until it reaches significant values, and plasma species begin to radiate at different wavelengths (FALAHAT et al., 2018).

When these elements interact with the surface of materials, they produce different biological effects, depending on the type of radicals produced in the jet (LEE et al., 2016). In addition to causing changes to the treated surfaces, generating roughness. And, due to the formation of functional groups composed

|     | Voltage (kV) | Frequency (kHz) | Energy (%) | Ambient humidity (%) | Flow (L/min) | Sample temperature (°C) |
|-----|--------------|-----------------|------------|----------------------|--------------|-------------------------|
| H.  | 4.9 – 12.0   | 26.3 – 41.7     | 1 - 90     | 30.0 – 50.0          | 0.5 – 2.0    | 19.3 – 36.4             |
| Air | 4.7 – 12.0   | 26.3 – 35.7     | 1 - 80     | 30.0 – 52.0          | 0.5 – 2.0    | 20.6 – 56.6             |

Table 2 – Experimental values of parameter variations with the working gases, He and Ar. Source: Prepared by the author.

of oxygen and nitrogen on the surface, a surface can be produced and sterilized, that is, free from bacteria and resistant to the emergence of them. To recognize the discharge species, the spectra obtained from the EEO technique for the plasma jet of different gases at atmospheric pressure are used, highlighting the results of research with He and Ar, which were used in the present work, also, the NIST Atomic Spectra Database Lines Form was of great importance in the recognition of some of the reactive species. The light spectrum for the DBD system obtained by expected EEO is a graph similar to that in Graph 1, where the working gas is helium and argon, respectively. (NASCIMENTO NETO, 2015 and KOSTOV, KG, 2022).



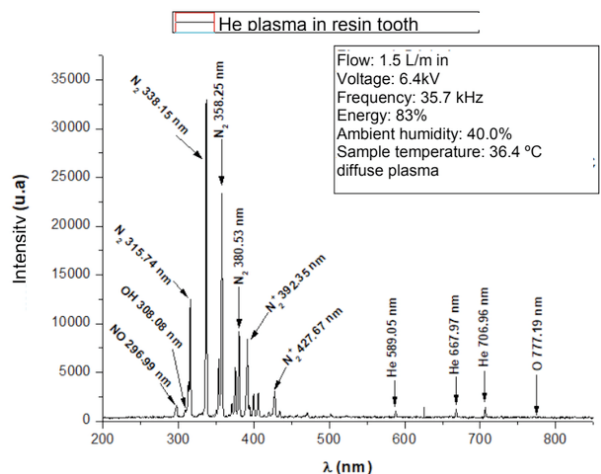
Graph 1 - Characteristic spectrum of a DBD plasma where the working gas is helium and argon, respectively, adapted from (WANG et al., 2016).

In the experiments there were used resin teeth and a moistened polyurethane sponge, simulating human gums, as they have pores that approach the fibrous tissue covered by

the mucosa of the gums. The treatments were carried out in both diffuse and filamentary regimes, but in the tooth resin, for having a greater dielectric strength, it was impossible to obtain a filamentary regime.

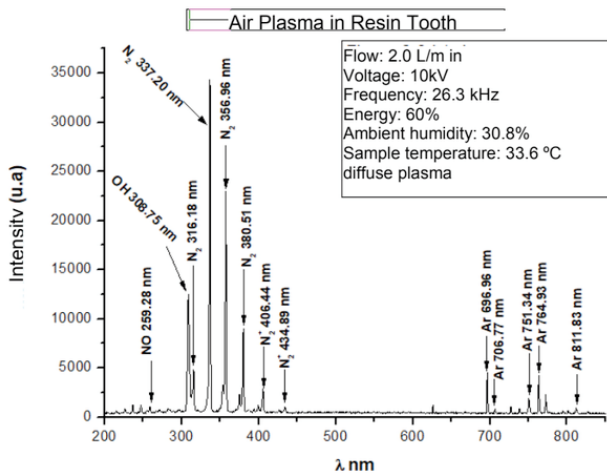
When used as the working gas of a plasma jet, noble gases (mainly He or Ar) present metastable states with relatively high excitation energy. This is why these species can play an important role in plasma chemical processes or even ionization processes.

The practical results coincided with the literature, which are presented in Graphs 2 and 3, which shows the EEO presented by a plasma jet with He and Ar, respectively, using the critical parameters for temperature control in resin tooth samples, aiming for use in dentistry. In these cases, the shapes of the investigated intensity distributions along the x-axis (wavelength ( $\lambda$  (nm))).



Graph 2 – EEO obtained from a He plasma jet with controlled parameters, applied to a resin tooth. Source: Prepared by the author.

Graph 2 shows the parameters at which the highest temperature was obtained in the resin tooth, these parameters being not recommended for application to the gums. Because the tooth has a higher dielectric strength than the polyurethane sponge, which could cause it to burn.



Graph 3 – EEO obtained from an Air plasma jet with controlled parameters, applied to a resin tooth. Source: Prepared by the author.

H.e. and Ar gases exhibit quite different discharge behavior. It is easier for He atoms in high-energy metastable states to ionize N molecules<sup>2</sup> to generate N ions<sup>2+</sup> at the He/air interface (BRUNO, F. et al., 2024). This behavior is proven in the graphs presented, where the N ions<sup>2+</sup> in He's EEO they show themselves in a wavelength range (392.35 nm to 470 nm), greater than that of Air EEO (400 nm to 434.89 nm).

In both spectroscopies, the appearance of only one OH radical was observed, which is highly reactive and can damage practically many types of macromolecules, such as lipids and amino acids in proteins (LEE et al., 2011).

This happens due to gas flow that affects OH concentration due to ambient air humidity, which affects H distribution O<sub>2</sub> and therefore the OH distribution of the plasma jet device (LU et al., 2016).

For dentistry, the emergence of OH radicals are important, as with their increased intensity, together with O radicals (present only in H.e. plasma), they increase the rate of inactivation of the bacterium *Streptococcus mutans*, which is one of the main causes of caries. Also, OH intensity can be an indicator of the tooth whitening effect (ŠANTAK et al., 2015 KOSTOV, KG, 2022).

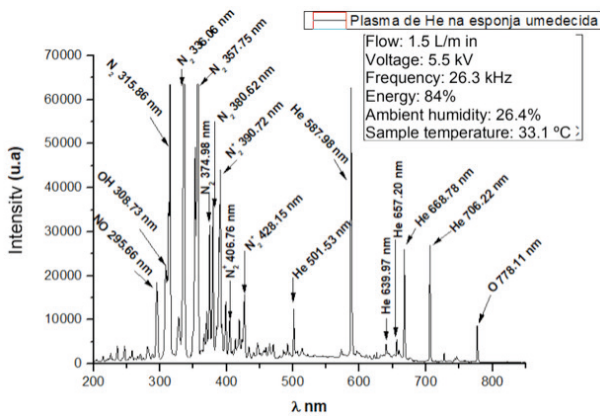
Despite presenting itself in a weak UV spectral range, it is also noted, in both EEO, the appearance of nitric oxide (NO), which together with OH radicals at atmospheric pressure, contribute significantly to biological decontamination and modifications. on the surface of biomaterials (BUDA et al., 2015). This reactive species can cause apoptosis, necrosis or, alternatively, protect cells from death, depending on the cell type, concentration of radicals, as well as the duration and specific areas of exposure (LU et al., 2016).

Nitrogen molecules in metastable states (N<sub>2</sub>) play an important role in gas discharge kinetics. These species have a long radiative lifetime (approximately 2 s) and high excitation energy (6.2 eV) and can induce various reactions in the discharge afterglow (LU et al., 2016). Currently, there are no reports on measuring absolute N density<sup>2</sup> in DBD plasma jets at atmospheric pressure in the literature. Although they act as sterilizing agents, eliminating microorganisms, improving surface properties.

Although the graphs do not show the presence of ozone (O<sub>3</sub>), it is present, being in a low UV spectral range, not being detected by the spectrometer.

In dentistry, the healing of gingival soft tissues plays an important role in aesthetic and health results after gum surgery. Gingival surgery has been widely used to improve the shape and color, as well as to prevent inflammation of teeth and dental implants (LEE et al., 2016 and MEYER, PF et al., 2024).

As with the resin tooth samples, the results of the spectra on the polyurethane sponge, simulating a gum, are quite similar. Graph 4 shows the EEO of the He plasma jet in the sample.



Graph 4 – EEO obtained from a jet of He filamentary plasma with controlled parameters, applied to a sample simulating human gums. Source: Prepared by the author.

EEO indicates that the filamentous plasma jet contains many reactive species typical (NO, N<sub>2</sub>, N<sub>2</sub><sup>+</sup>, OH) and atomic lines (He, O) observed in DBD's plasma jets fed with helium. At wavelengths from approximately 315 nm to 500 nm, the spectrum is dominated by emission from excited nitrogen species: N<sub>2</sub> and N<sub>2</sub><sup>+</sup>.

By dissociation of H<sub>2</sub> molecules what is present in the surrounding air provides evidence for the production of highly reactive OH radical in the jet. Between 501 nm and 750 nm, several helium lines are observed, but their intensity, compared to radioactive molecular nitrogen species, is relatively weak.

Some of the peaks appear in the UV region from 200 nm to 295.66 nm. These peaks indicate the existence of reactive nitrogen species (RNS). Another highly reactive species, atomic oxygen, whose line at 778.11 nm is observed, is produced after the dissociation of O<sub>2</sub> in plasma (KOSTOV, KG, 2022 and GUERRA NETO et al., 2024).

When comparing Graph 2 with Graph 4,

it is observed that the DBD discharge in a filamentary regime (Graph 4) has a range of varieties of free radicals, molecules, atoms, electrons with high energy and higher ultraviolet radiation, compared to the diffuse regime (Graph 2).

In this study, the species observed through the EEO technique are consistent with results from He and Ar DBD plasma studies (ABDEL-FATTAH et al., 2017; LI et al., 2019; LIN et al., 2019; NERETTI et al., 2018; WANG et al., 2016), which corroborates the reliability of the results.

It is observed that the application of plasma to a sample causes it to heat up from two simultaneous sources: heating caused by contact with heated gases (heat conducted from the gas to the sample) and heating caused by the passage of electric current through the sample (effect Joule). Joule heating, also known as ohmic heating and resistive heating, is the process by which the passage of an electric current through an imperfect conductor produces heat. Joule's first law, also known as the Joule-Lenz law, states that the heating power generated by an electrical conductor is proportional to the product of its resistance and the square of the circulating electric current. Both heat sources can be manipulated and controlled: the ionized gas can undergo this ionization by applying small amounts of energy and the joule effect can be reduced by applying small amounts of electrical current, that is, by passing a small number of electrical charges through the sample.

You can't reduce the applied voltage (and therefore the applied electric field) to the gas to be ionized below a certain value, the Paschen minimum, which defines the minimum voltage necessary for an electrical discharge to occur at a given pressure. a static gas. This prevents the temperature rise caused by the joule effect and heat conduction from being reduced below a certain value.

The solution found to allow electric field values above the minimum necessary for plasma formation and at the same time controlling the temperature rise was to variably “prick” this output voltage from the voltage source.

In other words, the source is turned off for a brief period of time (much longer than the period of the alternating signal generated), and it is turned on after that. This off/on period is variable, which means that the amount of energy generated is controlled in the form of “packets” of energy, which occur at high switching frequency rates. See Figure 10.

By varying the on/off time ratio we will be changing the amount of energy transferred to the gas and the sample, and consequently we will be controlling the heating of the sample. Figures 11 and 12 show thermal images captured with a FLIR C3 thermal camera.

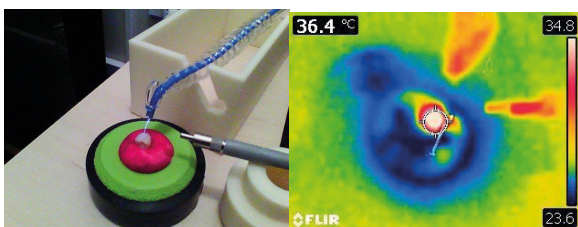


Figure 11: Thermal image of the diffuse DBD plasma, applied to the resin tooth sample with the following parameters: flow of 1.5 L/min; voltage 6.4 kV; frequency of 35.7 kHz; energy of 83% and relative humidity of 40.0%. Source:

Prepared by the author.

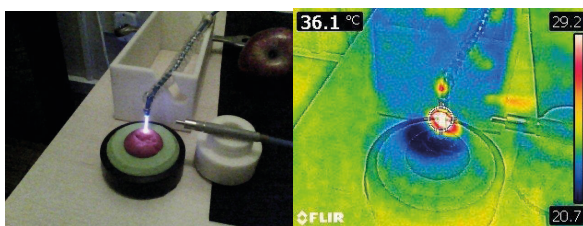


Figure 12: Thermal image of the filamentary DBD plasma, applied to the sample that simulates the human gingiva with the following parameters: flow of 1.5 L/min; voltage 5.5 kV; frequency 26.3 kHz; energy of 84% and relative humidity of 26.4%. Source: Prepared by the author.

Considering that the gingival tissue is more sensitive, it is important to note that in practice, it was found that even with values close to this limit value, heat transfer by conduction and joule effect still heated the samples (resin tooth and polyurethane sponge) above values allowed by dentistry, of approximately 42 °C, to avoid necrotizing the tissues.

## FINAL CONSIDERATIONS

Based on the above, it is pertinent to state that the ANALYSIS OF PARAMETERS AND IDENTIFICATION OF CHEMICAL SPECIES PRESENT IN DBD PLASMA JETS proved to be effective in terms of chemical identification of all species in the process.

- The use of plasma technology has demonstrated promising results in Dentistry. Its beneficial effects may be related to plasma parameters, especially the application time and the type of gas used.
- The study of reactive species is very important for several areas, especially dentistry, as the species carry out surface modifications, increasing surface energy, inactivating bacteria, cells unwanted, with a sterilizing effect and can finally facilitate the removal of cavities by breaking hydrogen bonds.
- Optical emission spectroscopy is one of the fundamental methods for investigating the composition and kinetics of plasma processes.

Due to voltage, frequency, percentage of energy, distance from the plasma jet to the target, relative humidity of the ambient air, plasma regime and interaction of radicals with atmospheric air.

- The determination of plasma parameters such as plasma temperature and relative density of excited species are keys to improving the quality of the plasma processing technique.



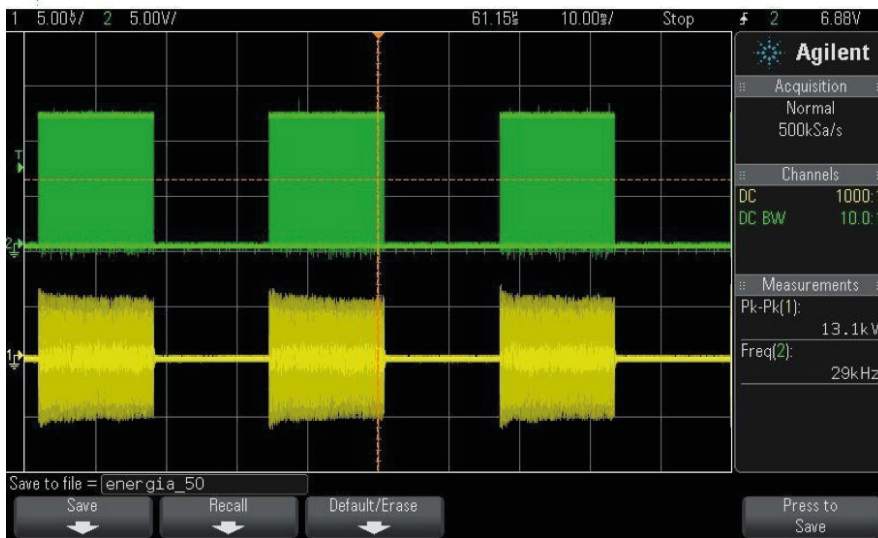


Figure 10: The alternating signal in Figure 6 is now turned on for a period (defined by the “Energy” control) and then turned off. Source: Prepared by the author.

- The parameters studied in the samples in this research, serve as a basis for treatments on samples in dentistry. The focus of this work was limited to simulations to determine parameters of gingival tissue and artificial teeth. New research will be necessary, subject to authorization from the ethics committee, to determine the real parameters applied to human teeth.
- Although the results were not obtained from the analysis on human teeth, the factors are significant, as the gingival tissue is the most critical in terms of sensitivity, while artificial acrylic teeth are those that represent less sensitivity to

filamentary discharges and variations in temperature.

- Dental plasma has no harmful effect on acrylic resin artificial teeth.
- A very important conclusion is that the maximum values of the parameters determined in this research for the gingival tissue must never be exceeded, under penalty of causing an electrocautery effect and thermal damage to the gums before the cavity is even removed.
- To exceed the aforementioned parameter values without burning the patient’s gums, it will be necessary to design a device to isolate the treated tooth.

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