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Recommendations and guidelines for the description of cold atmospheric plasma devices in studies of their application in food processing

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ABSTRACT

In this article, we propose an approach to provide a schematic and exhaustive description of a Cold Atmospheric Plasma (CAP) process; particular focus is placed on defining and differentiating the operating conditions of a plasma discharge and the fundamental characteristics of the resulting processes.

After identifying the main techniques to characterise a CAP treatment, two fundamental analyses to support their study are described in detail: electrical analysis, presenting different methods together with the equipment required to properly perform the measurements, and chemical analysis of the gas phase, discussing optical absorption spectroscopy (OAS) as a quantitative technique that poses relatively low challenges in terms of equipment availability and elaboration of acquired data.

Finally, a practical example of a detailed description of a plasma device, its electrical characterization and its gas phase characterization by means of OAS will is provided.

1. Introduction

Cold atmospheric plasmas (CAPs), ionized gases with limited temperature (below $100 \,^{\circ}$ C), are being increasingly explored as non-thermal technology suitable for a wide range of processes along the food supply chain, with possible roles extending from the farming/production to the processing phases (Chen et al., 2021; Gao et al., 2022; Guo et al., 2021a; Li et al., 2022; Lin et al., 2022; Puač et al., 2018; Šimek and Homola, 2021).

Focusing on food processing, CAP treatments can be classified in two main categories:

- bio-decontamination of pathogens to reduce spoilage and increase shelf-life;
- enhancement of nutritional properties and food functionalization.

In both cases, researchers aim to exploit the peculiar combination of active principles made available by CAPs: charged species, reactive species, UV radiation, temperature and electric fields. All these elements are formed when CAPs are produced, typically by means of electromagnetic excitation: charged species origin from the gas ionization, reactive species from the complex plasma chemistry initiated by electron collisions with neutrals, UV radiation from the relaxation of excited atoms and molecules, temperature from elastic collisions and relaxation processes, and electric fields, excluding those applied to ignite the plasma itself, from local accumulation of charged species.

While the field of CAP food processing was initially established by plasma physicists, chemists and engineers, it now involves researchers with very diverse scientific backgrounds: from food technologists to doctors, from agronomists to materials experts. Such specialists have brought a very positive impact on the field by introducing *i*) a better understanding of the food processing industry, *ii*) more refined techniques to assess the efficacy of CAP processes and *iii*) a focus also on the eventual side-effects of CAP treatments, such as the modification of organoleptic properties and the presence of toxic residuals and/or harmful byproducts. On the other side, the diversification of the scientific base researching the field of CAP food processing poses a communication challenge, since a common terminology and sensibility to mandatory information/data should be adopted to guarantee the replicability of performed experiments and a systematic and synergistic

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Fig. 1. Schematics of CAP devices for food processing: a) VDBDs, b) SDBDs, c) PJs, d) CDs, e) MWs.

development of the field. This issue was recently covered in the 'Technical roadmap - key food applications and standardized procedures', a document prepared by the European community working on CAP food processing in the context of the European project CA19110 Plagri (Plasma applications for smart and sustainable agriculture) (https:// plagri.eu/, n.d.). More than one hundred researchers contributed to the document and identified the standardization of procedures as an appropriate strategy to systematically approach the requirements posed by food applications on CAP processing, enabling objective comparisons of results obtained by different research groups and taking advantage of the efforts of the entire community of scientists working in this field. Three classes of standards were thus suggested:

- 1. standards to evaluate the characteristics of the employed CAP;
- 2. standards to evaluate the treatment efficacy;
- 3. standards to evaluate the safety and quality of plasma treated products.

This paper intends to elaborate on the first item of the list, presenting a prevailing point of view of researchers with a plasma physics/chemistry/engineering background.

Defining standards for evaluation of CAP employed in food technologies (washing, drying, extraction, decontamination etc.) should start with the community using unified terminology and a way of classification of CAPs. In the literature this is generally the case, but in some cases one can still find a description for a proposed new CAP system (or a way to classify them) that in the end can be classified as a member of one general group of CAPs. However, these are not sufficient for comparison of different CAPs because the same geometry of CAP can yield different modes of plasma (glow, filamentary etc.) depending on the frequency and power delivered to the plasma, feeding gas etc. To create necessary standards, community should define elementary plasma characterization procedures and protocols that are not expensive, do not perturb the plasma, do not require complex data evaluations and have the potential to be used as a data source for automatic control of treatment processes. In this paper, first, a classification of plasma devices and plasma set-ups will be provided, focusing on terminologies and important, albeit sometimes overlooked, equipment components. Second, some relevant parameters of widespread use for describing plasma discharges will be introduced, along with characterization techniques suitable for their measurement; here the importance of a rigorous electrical characterization of the plasma devices and of developing an understanding of the plasma gas phase chemistry will be stressed. The electrical characterization of plasma devices will then be covered in detail, presenting different methods together with the equipment required to properly perform the measurements. Afterwards, optical absorption spectroscopy (OAS), being a quantitative technique to measure long living reactive species that poses relatively low challenges in terms of equipment availability and elaboration of acquired data, will be discussed as an introductory technique to assess plasma gas phase chemistry. The density measurements of reactive species are related to chemistry involved in the food product treatment (disinfection origin, potential harmful byproducts, ...) and could be used for the control of the total food treatment process. Power and electrical measurements are very relevant also in the industrial perspective, since they allow to draw considerations on the efficiency of the process and its economic footprint. In addition, information from electrical characterization (such as power density) can also be used to predict the nature of the plasma discharge produced and thus the type of chemistry that governs the process. Finally, a practical example of a detailed description of a plasma device, its electrical characterization and its gas phase characterization by means of OAS will be provided for a surface dielectric barrier discharge (SDBD).

2. Classification of plasma devices

The efficiency of a CAP treatment for food processing strongly depends on the selected CAP device and its related operating parameters (e.g. discharge power and gas composition) (Domonkos et al., 2021a). For this reason, in recent years, numerous CAP devices have been investigated in the food sector (Laroque et al., 2022a). Despite a standard classification has not yet been proposed, CAP devices for food processing are often ascribable to these categories: dielectric barrier discharges (DBDs), plasma jets (PJs), corona discharges (CDs), and microwave-driven discharges (MWs). Each of these devices exhibits its own characteristics, which will be discussed in the following paragraphs.

Among the mentioned CAP devices, DBDs represent the most used nowadays. They consist of two conductive electrodes (a powered electrode and a ground electrode) and, as the name suggests, at least one dielectric barrier (Pipa and Brandenburg, 2019; Rao et al., 2023).The dielectric barrier is generally made of an insulating material (such as a polymer, glass, quartz, or ceramics) and prevents the transition of the discharge to an electrical arc which would otherwise occur between two uncovered metal electrodes (Chizoba Ekezie et al., 2017a; Domonkos et al., 2021b; Peeters and Butterworth, 2019). According to the assembly of the electrodes and the barrier, it is possible to distinguish between volumetric DBDs (VDBDs, Fig. 1a) and surface DBDs (SDBDs, Fig. 1b) (Misra and Roopesh, 2019). In VDBDs, the electrodes covered by dielectric material are separated in such a way that the plasma discharge is generated in the volume between the electrodes (Niemira, 2012; Shaw et al., 2015). The distance between the electrodes is usually called "gap" and ranges from 0.1 mm to several centimeters (Laroque et al., 2022b). These dimensions create inevitable limitations in the size of the sample to be treated, which is positioned in the gap. Furthermore, the presence of the sample between the electrodes must be necessarily considered for

an appropriate analysis of the overall electrical circuit. For SDBDs, both the electrodes are in direct contact with the dielectric barrier and the discharge is generated on the surface of the dielectric. Frequently, SDBDs involve one metallic mesh electrode adherent to the dielectric surface and a metallic sheet on the opposing side of the dielectric as counter electrode (Cullen et al., 2018). Whatever the configuration, DBDs are typically driven by AC or pulsed DC signals at frequencies in the kHz range (Bruggeman et al., 2017; Domonkos et al., 2021b). The major advantage of DBDs is their unprecedent flexibility with respect to electrode geometries, working gases (nearly every combination of gases can be used), and operating parameters (Chizoba Ekezie et al., 2017b; Wagner et al., 2003). Furthermore, DBDs are especially interesting for industrial applications because they operate with low gas flows, allow large area treatments, and are characterized by low maintenance cost (Rao et al., 2023; Surowsky et al., 2015). On the other hand, DBDs require relatively high voltages (10 kV or more) to be ignited, thus making the adoption of proper precautions or isolations essential (Ehlbeck et al., 2011).

PJs constitute another class of CAP devices suitable for food processing. In PJs, a flow of gas (typically a noble gas such as helium or argon) pushes the plasma discharge, formed in the electrode region, outside. In this way, the plasma discharge is projected in the open environment with a "jet-like" appearance (Domonkos et al., 2021b; Laroque et al., 2022b). As shown by Lu et al., many plasma jet configurations have been studied over the years (Lu et al., 2012). PJs often consist of two electrodes (e.g. a powered ring electrode and a ground ring electrode), but single-electrode configurations (e.g. a powered needle electrode and a virtual ground electrode) are encountered as well (Surowsky et al., 2015). As an example, Fig. 1c reports a schematic of a PJ, where the gas flows in the dielectric tube surrounded by two ring electrodes. Since in this case the dielectric tube plays the same role than in DBDs, such configuration is typically referred as "DBD-jet". For this configuration and for the others not described here in detail, the powered electrode can be connected to a wide range of power supplies (e.g. kHz AC, pulsed DC, and RF) and the discharge ignition is generally reached at lower voltages than in the case of DBDs (Laroque et al., 2022b; Lu et al., 2012; Misra and Roopesh, 2019). The main strength of PJs is their ability to produce plasma plumes with lengths of several centimeters and diameters of few millimeters. Indeed, this aspect enables localized treatments both at the surface of 2D and 3D substrates and in narrow cavities (Booth et al., 2022; Surowsky et al., 2015). As a consequence, PJs clearly are not the most convenient option for large area treatments. However, alternative solutions can be adopted in this case, such as an arrangement of several PJs or the movement of a single PJ along a defined pattern (Kong et al., 2009).

CDs in atmospheric air have been studied experimentally and theoretically over many decades, finding application in a wide variety of industrial sectors, including the food-related one (Timoshkin et al., 2012). CDs are generated near high voltage electrodes characterized by sharp edges or protrusions (e.g. needle or thin wire electrodes) under the action of strong electric fields (Chang et al., 1991; Misra and Roopesh, 2019). Once generated, they appear as faint filamentary discharges radiating outward from the powered electrode. Indeed, CDs are very weak discharges, characterized by very low electron and ion densities (Chang et al., 1991; Domonkos et al., 2021b). A typical geometry for CDs is the point-to-plate geometry shown in Fig. 1d, which includes a pointed electrode and a flat electrode. This configuration can be operated in DC or pulsed mode and the pointed electrode can have a negative or a positive potential (Surowsky et al., 2015). As a final remark, CDs are usually inexpensive and relatively easy to operate, but their applicability is limited by the treatment non-uniformities and the small treated areas (Chizoba Ekezie et al., 2017b; Rao et al., 2023; Surowsky et al., 2015).

Finally, MWs are generated through a magnetron or by a solid-state power supply that emits electromagnetic waves at 2.45 GHz (microwaves) which are guided to an ignition chamber by a waveguide (Surowsky et al., 2015). The electrons present in the gas in the ignition chamber absorb energy from the microwave electric field and are consequently accelerated, thus resulting in the formation of a plasma discharge (Laroque et al., 2022b). The main advantages of MWs with respect to the other presented classes are the following: i) absence of electrodes (as can be seen from the schematic in Fig. 1e), ii) increased electron density in the ionized gas, iii) high efficiency in generating reactive species, iv) possibility to ignite the discharge in air environment (Laroque et al., 2022b; Rao et al., 2023). Nonetheless, MWs are still little used because of their price and complexity of operation (Surowsky et al., 2015).

At this stage, a clarification about MWs should be provided. MWs at atmospheric pressure are usually classified as "hot" plasmas, with characteristic temperatures of some thousand kelvin (Fridman, 2008; Surowsky et al., 2015). Therefore, in this context, MWs can be included in the list of CAP devices mainly considering their afterglow. Indeed, the afterglow (remote plasma) is the region, spatially located downstream of the primary discharge (plasma glow), where the gas temperature is substantially lower and useful reactive species, such as excited and longlived species, are still present (ions, electrons and short-lived reactive species are lost due to recombination) (Moreau et al., 2000).

For sake of completeness, it is good to mention in this context also two other categories of CAP devices which are sometimes associated to food processing.

Gliding arc discharges (GAs) are known as hot plasma sources, however, under specific conditions, can be classified as cold plasma sources. They are typically created between two or more diverging metallic electrodes operating at different voltages. When the process gas is pumped into the discharge gap between the electrodes, an arc is formed in the narrowest gap. The arc is subsequently blown away by the inlet gas into the diverging area: the discharge increases its volume and length in the flow direction, progressively decreasing its temperature (Chizoba Ekezie et al., 2017b; Domonkos et al., 2021b; Niemira, 2012).

Radio frequency discharges (RFs) are achieved when a gas is placed within an oscillating electromagnetic field inductively or capacitively coupled with the powered electrode. They usually work at frequencies in the range of MHz and at atmospheric pressure, depending on the geometry and deposited power into the discharge, they have wide range of temperatures (from low to very high gas temperatures). For this reason, the considerations expressed above for MWs can be applied also to RFs (Chizoba Ekezie et al., 2017b; Niemira, 2012; Varilla et al., 2020).

All the CAP devices intended for use in food processing that have been presented in the previous section can be operated in a direct mode or in an indirect (also called remote) mode. In the direct mode, plasma is in direct contact with the substrate to be treated. This results in a close interaction between the two, which is based on irradiation (VUV, UV), charged molecules, radicals, and reactive particles (Schlüter et al., 2013). Indeed, in the direct mode all the components of the plasma discharge can be involved in the treatment, even reactive species which have short lifetimes in the range of some milliseconds (Surowsky et al., 2015). An example of CAP devices which are typically used in direct mode consists of VDBDs. In this case, food can be placed in the gap between the electrodes thus allowing to benefit of the consequent attachment of the plasma discharge to the food surface. Nonetheless, under specific discharge conditions, the close interaction between plasma and food may lead to non-uniform treatment or excessive localized heating. For this reason, the design of direct treatments can be sometimes challenging (Niemira, 2012). In some cases, to fully preserve the quality of the food, it is preferable to indirectly expose the food to the plasma by placing it adjacent rather than inside the volumetric discharge (Georgescu et al., 2017; Shaw et al., 2015).

In the indirect mode, the substrate to be treated is located at a point physically separated from the plasma generation zone. Therefore, only the "plasma exhaust" (previously also defined as "afterglow") comes into contact with the sample. With respect to the direct mode, this strongly simplifies the operation and increases the flexibility in terms of shapes and sizes of the samples to be treated (the confinement between

Table 1

Typical operating conditions of a plasma process.

Category	Operating condition	
Electrical	Voltage	
	Frequency	
	Current	
	Power	
	Duty cycle	
Treatment	Mode	
	Duration	
	Distance plasma-sample	
Process gas	Composition	
6	Flow rate	
Treated material	Туре	
	Weight or volume	

the electrodes is no longer present) (Niemira, 2012). On the other hand, since both charged species and short-lived reactive species are mainly confined to the surroundings of the dielectric and do not reach the substrate, the treatment cannot benefit from the positive effects induced by these species (Niemira, 2012; Shaw et al., 2015; Surowsky et al., 2015). As reported by numerous works, successful indirect treatments of food can be achieved using SDBDs, which, by definition, are characterized by the generation of plasma on the surface of the dielectric (Foligni et al., 2022a; Han et al., 2020; Yong et al., 2015). Indirect treatments are possible even with PJs if proper electrode arrangements and distances between the jet nozzle and the food are selected (Shaw et al., 2015). Finally, it is appropriate to mention in this section the key role of MWs in the production of plasma-processed air (PPA), an air enriched with reactive oxygen and nitrogen species that has attracted a lot of interest in recent years as method for microbial disinfection of fruits and vegetables (Bußler et al., 2020; Schnabel et al., 2018; Yang et al., 2023).

In addition to direct and indirect modes, the described CAP devices can be used also in a third mode, which involves the mediation of liquids. We prefer to keep this mode in a separate section because i) the paper will be focused mainly on CAP devices operated in direct and indirect modes, ii) the topic would require a thorough discussion of plasma induced liquid chemistry, a subject which has already been covered in numerous works (Bruggeman et al., 2016; Jablonowski and von Woedtke, 2015; Von Woedtke et al., 2022) and which is far beyond the purpose of this paper. Therefore, just some basic information about this mode will be provided.

'Activating' water or other liquids through the exposure to plasma discharges represents a field of strong interest for the food industry (Cullen et al., 2018). In recent years, particular attention has been dedicated to the interaction of plasma active species with aqueous samples to produce the so-called plasma-activated water (PAW), a promising method to replace conventional sanitizing agents for food decontamination (Perinban et al., 2019). Despite research on the mechanisms which make PAW an antimicrobial agent for foods is still ongoing, many studies indicated that the main active substances of PAW are reactive oxygen species and reactive nitrogen species (Cullen et al., 2018; Guo et al., 2021b). These species are formed directly in the liquid or at the liquid-gas interface, depending on the selected approach: plasma generated in contact with water or plasma generated over the water surface (Han et al., 2023; Thirumdas et al., 2018). To support these approaches, numerous CAP devices can be employed, including some of those previously described (e.g. CDs, DBDs, PJs, and GAs) (Bruggeman et al., 2016; Chen et al., 2018; Laurita et al., 2015; Laurita et al., 2021; Magureanu et al., 2008).

3. Operating conditions and plasma parameters

Providing an accurate description of operating condition and plasma characteristics is of paramount importance to ensure a correct understanding of the presented results and the replicability of the described

experiments.

A first distinction must be made between operating conditions and plasma parameters: the former are the conditions imposed on the system in order to obtain a certain plasma discharge; these conditions describe in detail what happens upstream of a plasma process and are fundamental to being able to replicate an experiment. On the other hand, "plasma parameters" are the measurable quantities describing the discharge itself, whose analysis is fundamental for the understanding of observed phenomena.

Focusing on operating conditions, it should be noted that these are very heterogeneous and encompass all aspects of the preparation of an experiment; referring to Table 1, we can classify them into four main areas: electrical conditions, treatment modalities, process gases and samples to be processed. Some of these quantities are easier to measure and are not altered by the plasma process; while others, such as the electrical characteristics of the discharge, must be monitored throughout the entire treatment in order to gain a complete picture of the process being studied. It is worth mentioning that Table 1 refers to the electrical conditions for the case of AC or pulsed excitation, the most frequent in plasma processing of food; in some cases, such as RF and MW driven plasma devices, the electrical operating condition is instead defined by the applied power. Given the centrality and complexity of electrical measurements, the entire *Chapter 4* is dedicated to these analyses to determine the power dissipated in the discharge.

On the other side, 'plasma parameters' is used here a broad category encompassing every characteristic useful to describe the physical and chemical properties of a plasma discharge, such as chemical composition, electron density and average temperature, vibrational and rotational temperatures, ion concentration and UV radiation. Over the years, several techniques have been developed and optimized to assess the characteristics of plasmas (Tanarro et al., 2011). Since plasma is a gas that emits and absorbs light radiation, the most commonly used investigation techniques are spectroscopies. Electromagnetic waves have a very wide range of wavelengths, but in practice analyses are carried out in the ultraviolet, visible or near infrared range. Below 200 nm, in fact, radiation is unable to propagate at atmospheric pressure without being absorbed; similarly, above 1 µm it is not possible to obtain information on the plasma due to thermal background noise (Hieftje, 1992). Radiation in the UV and visible spectral ranges originates from atomic and molecular transitions.

In general, plasma spectroscopy can be divided into two approaches: emission-based techniques, such as optical emission spectroscopy (OES), which exploit the light emitted by the plasma to derive certain characteristics and are therefore referred to as "passive" techniques, and absorption techniques which exploit the ability of molecules and atoms to absorb photons at a certain frequency; the latter can be further divided between techniques that rely on the analysis of the light absorbing capacity of the plasma, as in the case of optical absorption spectroscopy (OAS), and others where what is measured is a light emission following the absorption of a photon with proper wavelength, such as in laser induced fluorescence (LIF).

The analysis of the emission spectrum of a plasma discharge, obtained for example through OES, can provide a direct observation of the chemical composition of the discharge (atoms, molecules, ions and radicals), enabling the identification of the presence of specific excited species (such as OH and O from water vapour presence in the discharge or particles released from a surface by sputtering) (Tanarro et al., 2011). Other characteristics of the plasma can be derived analysing more precisely the shape of the lines in the spectrum: the temperature of the gas can be determined through the analysis of the Doppler broadening, the electron density can be determined through the study of Stark broadening, and finally, the strength of the magnetic fields can be measured from Zeeman broadening; details of line broadening mechanisms and their applications to plasma diagnostics can be found in (Fantz, 2006; Hieftje, 1992; Lochte, 1995). Finally, electron temperature can be determined analysing the absolute line radiation with a technique that requires an absolutely calibrated spectroscopic system (Fantz, 2006). As a final note, when performing OES it is worth remembering that, being an emission technique, it cannot provide direct information on the ground state population of the species and that it provides line-of sight averaged parameters.

Moving to absorption techniques, the first developed is OAS and involves the use of a source of photons that are passed through a gas. By comparing the intensity of the sampling radiation, at a specific wavelength, before and after passing though the gas, it is possible to calculate the quantity of specific chemical species using the Lambert-Beer law (Fantz, 2006). This technique will be explained in detail in *Chapter 5*. One of the main limitations of the OAS technique is that the measurement is limited by the absorption cross-section of the species of interest: if that is relatively small compared to the absorption cross-section of conflicting species, meaning species absorbing at the same wavelength of the species of interest, present in the gas to be analysed, it is impossible to measure its concentration. This limitation can be overcome by means of techniques such as Cavity Ring-Down Spectroscopy (CRDS), Laser Induced Fluorescence (LIF) and Two-photon Absorption Laser Induced Fluorescence (TALIF).

CRDS is a technique where the light absorption is intensified by artificially increasing the optical path of the sampling radiation across the gas; this is obtained using a cavity with highly reflective mirrors and focusing a laser beam in such cavity. Due to the large number of reflections the photons are confined inside the cavity, the optical path inside the cavity increases up to a few kilometres, greatly increasing the accuracy of the measurement (Stancu et al., 2010).

LIF, one of the most widely used techniques in the plasma spectroscopy field, employs a highly energetic laser to excite the species present within the gas; by carefully choosing the frequency of exciting photons, it is possible to analyse the emission resulting from the relaxation of the species of interest (Fantz and Wimmer, 2011). TALIF on the other hand exploits the successive absorption of two photons at different frequencies by the same species, subsequently observing the emission during the relaxation of the excited species (Stancu et al., 2010); by using two lasers at different frequencies, it is therefore possible to further extend the range of applicability of spectroscopy techniques.

Another spectroscopic analysis technique used in the plasma world, but not based on absorption or emission, is Raman spectroscopy. When a photon collides with an atom or molecule, there is also the possibility of it being scattered; if the collision is elastic, this is referred to as Rayleigh scattering, while inelastic collisions are defined as Raman scattering. The possibility of inelastic collisions occurring is very low (1 per 10⁷ photons) and totally dependent on the type of chemical species present in the gas; this peculiar property of molecules enables us to trace the concentration of certain chemical species (Gao et al., 2019; Mulvaney and Keating, 2000a; Mulvaney and Keating, 2000b; Petrov et al., 2018; Petry et al., 2003).

The last analysis technique reported in this overview is mass spectroscopy (MS) that can sample at atmospheric pressure and can be used to classify ions based on different mass-to-charge ratios (Große-Kreul et al., 2015; Maletić et al., 2012). It can give information of both molecular and atomic reactive oxygen and nitrogen species relevant for biomedical applications, agriculture or food technologies. While in other fields of application this technique must rely on an energy source, such as an electron beam, to produce the ions, in the field of plasma technology can be employed to measure the ions naturally occurring in the plasma (Bruggeman et al., 2010; Oh et al., 2015).

4. Electrical characterization of plasma devices

Electrical characterization allows to acquire profound knowledge about the power dissipated in the discharge, thus representing an essential tool for the diagnostics, the comparability, and the up scaling of atmospheric pressure plasma sources (Pipa and Brandenburg, 2019). According to literature, there are two main methods to measure the



Fig. 2. Schematic representation of the typical electrical characterization setup related to the standard method.

dissipated discharge power: the standard method and the Lissajous method (Ashpis et al., 2017; Peeters and Butterworth, 2019; Pipa and Brandenburg, 2019). A comprehensive description of these methods and of their associated electrical characterization setups will be provided in the following.

4.1. The standard method (V-I measurement)

Fig. 2 reports the schematic representation of the typical electrical characterization setup related to the standard method.

To provide transversal character to the explanation, the "black box" in Fig. 2 indicates a non-specific atmospheric pressure plasma source including two generic conductive electrodes, one connected to the power supply, and one connected to the ground. This arrangement commonly allows to apply to the neutral gas comprised between the electrodes an electric field sufficiently strong to ignite and sustain the plasma discharge (Raizer, 1991).

Atmospheric pressure plasma sources can be driven by numerous power supplies, which are often classified according to the frequency (f) of the applied voltage: direct current power supplies (either operated continuously or pulsed); alternating current power supplies, with 100 kHz \leq f \leq 100 MHz; microwave power supplies, with f > 100 MHz (Napartovich, 2001). The choice of the proper power supply is strictly dependent on the plasma source and to the target application. As an example, dielectric barrier discharges (DBDs) are characterized by a working principle which necessarily requires an operation under AC or pulsed signals (Brandenburg, 2017). Similarly, in the context of the production of plasma activated water for decontamination it has been demonstrated that the type of power supply strongly affects the formation of oxygen and nitrogen reactive species (Gott et al., 2023).

Other key elements of the electrical characterization setup presented above are the electronic probes located along the electrical circuit to acquire the voltage and the current signals. The voltage is preferably measured using a calibrated high voltage probe (typically with a 1000:1 attenuation), connected as close as possible to the high voltage electrode on the high voltage line (Peeters and Butterworth, 2019). The exceptions to this are electrical discharges with power supply in the range of several MHz and higher where such a probe introduces significant changes into electrical circuit due to its capacitance. On the other hand, the current is usually measured on the grounded line by using an inductive Rogowski coil or the combination of a known shunt resistor and a low voltage probe (Ohm's law) (Ashpis et al., 2017; Celestin et al., 2008; Desmet et al., 2009). The decision to measure the current on the grounded line arises from two main motivations. First, it helps in interpreting the performances of the plasma source since the current flowing through this line is effectively representative of the behavior in the plasma discharge. Secondly, it allows to prevent crowding of elements the high voltage line which can lead to unwanted discharging, thus increasing the safety during the experiments. The only exception to these considerations is represented by plasma sources which have a poorly defined ground electrode, such as for example plasma jets impinging on different surfaces. In this case, it may be beneficial measuring the current directly on the high voltage line between the power supply and the plasma source (Peeters and Butterworth, 2019; Sarani et al., 2011; Simoncelli et al., 2019).

As shown in the electrical characterization setup, the electronic probes are usually connected to an oscilloscope, namely an instrument which graphically displays the varying electrical signals as a function of time. To accurately acquire signals when using an oscilloscope, some practical recommendations must be taken into account. First of all, doing much use of the oscilloscope's full scale is always suggested, but care should be taken to avoid exceeding the maximum range of the oscilloscope (signal over-ranging). Furthermore, the investigated total time for each acquisition must be sufficiently long to visualize at least 3-4 complete applied voltage periods. In this way, the acquired signals can adequately represent the generic electrical behavior of the plasma source, especially for plasma sources whose operation is not perfectly stable over time. Moreover, the number of points used to discretize the signals must be properly selected. As expected, the greater the number of points, the more precisely the acquired signal will represent the real signal. On the other hand, a greater number of points will require a greater computational effort to process the data. For this reason, the chosen number of points generally derives from an applicationdependent cost-benefit analysis. As a final remark, signals must be acquired a sufficiently large number of times (always >3) to ensure the statistical significance of the result.

Through the oscilloscope, the signals can be acquired, saved, and then post processed using software able to perform simple mathematical operations (e.g. Excel and Matlab). In the standard method, the total dissipated discharge power (P), averaged over the number (n) of applied voltage periods (T), is determined directly from the measured voltage, V (t), and current, i(t), using the following formula (Peeters and Butterworth, 2019; Pipa and Brandenburg, 2019):

$$P = \frac{1}{nT} \int_0^{nT} V(t) \, i(t) \, dt$$
 (1)

To achieve an accurate discharge power value, when using numerical methods to calculate this integral, it is fundamental to ensure that the integration of the acquired data is performed over exact multiples of the applied voltage period. The results in terms of dissipated power are typically expressed in watts (e.g. 50 W) and presented along with the corresponding standard deviations or standard errors resulting from the repeated measurements (e.g. 50.0 ± 0.5 W).

For those plasma sources for which the key parameter to interpret the gas phase chemistry is represented by the surface power density, e.g. surface dielectric barrier discharges (SDBDs), the formula presented above can be modified to include the active discharge surface (A) (Simoncelli et al., 2019):

$$P = \frac{1}{A} \frac{1}{nT} \int_0^{nT} V(t) \, i(t) \, dt$$
 (2)

When discussing the power dissipated in the discharge, it is worth to also introduce the concept of duty cycle (DC). This parameter defines the fraction of time in which the voltage signal is provided by the power



Fig. 3. Schematic representation of the typical electrical characterization setup related to the Lissajous method.

supply: during the on-time, power is dissipated in the discharge, while during the off-time the power is null (Lieberman and Lichtenberg, 2005). Since it denotes a simple strategy to modulate the dissipation of power in the discharge, the duty cycle is frequently used to prevent possible undesired complications deriving from the continuous application of a voltage signal, like for example the overheating of the plasma source or of the sample being treated. In the calculation of the power dissipated in the discharge, the presence of a duty cycle must be always taken into account. If the total time investigated is sufficiently long to include the presence of complete cycles showing both on-time and offtime windows, the formula (1) can be used as is. Otherwise, it is a common practice to focus the electrical measurements within an on-time window and then correct the formula (1) as shown below (Laurita et al., 2021):

$$P = DC \frac{1}{nT} \int_{0}^{nT} V(t) \, i(t) \, dt$$
(3)

4.2. The Lissajous method

First introduced by Manley in 1943 (Manley, 1943), the Lissajous method is an alternative method to calculate the discharge power dissipated in dielectric barrier discharge (DBD) plasma sources (Pipa and Brandenburg, 2019). Despite being less straightforward than the standard method, this method is widely used in literature, thus deserving a dedicated section in this context (Biganzoli et al., 2014; Fang et al., 2012; Hołub, 2012; Kakaroglou et al., 2015; Jiang et al., 2013; Kostov et al., 2009; Lieberman and Lichtenberg, 2005; Nikiforov et al., 2011; Bisag et al., 2020; Tyata et al., 2013).

The schematic representation of the typical electrical characterization setup associated to the Lissajous method is shown in Fig. 3, where the "black box" refers in this case to a generic DBD plasma source. As can be seen from the schematic, this method requires the addition of a monitor capacitor (C_m) between the reactor and the ground and the measurement of the voltage across it, $V_m(t)$, through a low voltage probe (typically with a 10:1 attenuation).

The presented modification in the electrical characterization setup allows to calculate the instantaneous charge on the monitor capacitor Q (t) by applying the relationship in Eq. (4):



Fig. 4. Schematic of the experimental setup for optical absorption spectroscopy.

(4)

$$Q(t) = C_m V_m(t)$$

Since the current through the monitor capacitor must be identical to the current through the plasma source (because of the series of the elements in the circuit), the charge measured on the monitor capacitor is used in this method to gather information on the charge deposited on the dielectric surfaces of the DBD plasma source and, consequently, on its associated power consumption (Ashpis et al., 2017; Peeters and Butterworth, 2019). Indeed, the average dissipated discharge power can be derived as the area of the closed loop created by plotting the measured charge, Q(t), as a function of the applied voltage, V(t), the so-called Lissajous figure (or Q-V diagram), multiplied for the inverse of the applied voltage period (Ashpis et al., 2017; Peeters and Butterworth, 2019). This concept is expressed in Eq. (5) where the circulation represents the Lissajous figure:

$$P = \frac{1}{T} \oint V(t) dQ(t) \tag{5}$$

The relationship between area and dissipated power is valid for any shape of the Lissajous figure, whether this resembles a perfect parallelogram like in the most ideal situation (Falkenstein and Coogan, 1997; Manley, 1943), whether it significatively differs from a parallelogram as often happens in practice (Jiang et al., 2013; Kuhnhenn et al., 2016; Pipa et al., 2013; Zhang et al., 2018; Kriegseis, 2011). When an ideal Lissajous figure is obtained, the capacitances associated to the different phases of the working principle of the DBD plasma source can be derived as the slopes of each side of the fig. (Peeters and Butterworth, 2019; Pipa and Brandenburg, 2019). The possibility to gather additional electrical information from a simple Q-V diagram is one of the main reasons behind the widespread use of the Lissajous method.

To successfully apply the Lissajous method, a proper monitor capacitor must be selected. Typically, to limit the effects of the addition of an element in the electrical circuit, C_m is chosen to be large compared to the capacitance of characteristic of the plasma source without plasma discharge (sometimes referred as C_{cell}), whose value can be estimated for example using an LCR-meter (Ashpis et al., 2017; Brandenburg, 2017). As a rule of thumb, a C_m of 100:10000 times C_{cell} is generally appropriate: if C_m is too small, the voltage across the capacitor will exceed the maximum voltage range of the oscilloscope, thus leading to signal overranging, while if C_m is too large the amplitude of the voltage across the monitor capacitor will become very small, and the resulting low signal-to-noise ratio will become problematic. Moreover, class 1 ceramic capacitors are preferred, as they have relatively low parasitic inductances

and parasitic capacitances (Peeters and Butterworth, 2019).

To close, it must be pointed out that the considerations made in terms of surface power density and the duty cycle for the standard method, along with the related opportune modifications in the formulas, can be extended also to the case of the Lissajous method.

5. Optical absorption spectroscopy

As already mentioned, the OAS technique exploits the property of atoms and molecules to absorb photons at certain wavelengths, enabling the measurement of the absolute concentration of a species within a given volume (Fantz, 2006; Moiseev et al., 2014). This technique has several strengths, among which being quantitative, calibration-free, non-intrusive, real-time and relatively simple to perform. From a practical point of view, an OAS analysis setup consists of three main elements: a light source, a measuring cell, and a detector. The light source must be chosen according to the chemical species whose concentration is to be measured; to date, the most commonly used photon sources are broad-spectrum lamps, LEDs, and lasers. The measuring cell is a closed volume inside which is the gas to be analysed; this cell must have optical accesses that are transparent to the radiation emitted by the light source. The radiation emitted by the light source passes through the measuring cell and reaches at the detector after being partially absorbed by the sampled gas. Depending on the light source used, different detectors can be adopted; a spectrophotometer coupled with a photomultiplier is a common choice: the spectrophotometer allows to focus the analysis to specific wavelengths, shielding the rest of the incident radiation, and it intensifies the signal which can then be read through an oscilloscope.

A typical setup for absorption spectroscopy of a plasma source (measuring cell) is schematically represented in Fig. 4. Here a lamp characterized by a broad-band spectrum from UV to NIR radiation is used as a source of a light beam which is made parallel trough optical fibres and fused silica lens after passing through the plasma source, the beam is collected into a spectrometer to spectrally resolve the light beam in the UV, VIS, and near-infrared (NIR) regions. A photomultiplier tube connected to a fast oscilloscope is used as the detector. The PMT amplification factor must be kept constant for all acquisitions to guarantee the comparability of results collected in different runs of the experiment; for similar reasons, between two consecutive measurements it is good practice to flush the measuring cell to avoid the accumulation of plasma products.

The species concentrations can be quantified from absorption measurements by employing the Lambert-Beer law:



Fig. 5. PASS schematic.

$$\frac{I}{I_0} = e^{(-L\sigma n)} \tag{6}$$

where I/I_0 is the ratio between the initial light intensity I_0 and the light intensity I after an optical path length L, n is a concentration of absorbers. The absorption cross-section σ is a function of the wavelength ($\sigma = \sigma(\lambda)$).

Absorption cross-sections of the chemical species present in a plasma can normally be found in literature in specific publications or in dedicated databases, see for reference the database 'The MPI-Mainz UV/VIS Spectral Atlas of Gaseous Molecules' (Keller-Rudek et al., 2013). As reported by Moiseev et al. (Moiseev et al., 2014) it is of paramount importance to accurately choose the wavelengths at which absorption measurements are to be taken. In order to obtain the most accurate measurement possible, it is necessary to choose as the wavelengths for the tests, those in which the difference between the absorption of the species of interest and the other absorbing species present in the gas to be analysed is maximised.

For the same reason, it is not always possible to find a wavelength suitable for measuring the concentration of a particular reactive species, for example: HNO_3 has an absorption cross-section that is always lower than that of O_3 in the UV-VIS range, which does not permit direct analysis by OAS. In the frame of food applications, the most interesting species that can be investigated are O_3 , NO_2 and NO_3 .

In general, when *N* species absorb at the same λ_j wavelength the Lambert-Beer equation can be re-written as:

$$\frac{I}{I_0}\Big|_j = e^{\left(-L\sum_{i=1}^N \sigma_{i,j} n_i\right)}$$
(7)

where $\sigma_{i,j}$, that represents the absorbing cross section of the *i*-species at λ_j , and the ratio between the light intensities *I* and I_0 is referred to the *j*-wavelength. Solving it for the determination of the concentration *n* of the *k*th species:

Table 2	
PASS operating	conditions.

Category	Operating condition	
Electrical	Voltage	6 kV
	Frequency	22.5 kHz
	Current	-
	Duty cycle	10 %
Treatment	Mode	Indirect treatment
	Duration	30 min
Process gas	Composition	Air
	Flow rate	Static
Treated material	Туре	-
	Weight or volume	-

$$n_{k} = -\frac{1}{L\sigma_{kj}} ln \left(\frac{I}{I_{0}} \right|_{j} \right) - \sum_{i \neq k}^{N} \frac{\sigma_{ij}}{\sigma_{kj}} n_{i}$$
(8)

where the absorbance A at the *j*-wavelength is expressed by:

$$A_{j} = ln \left(\frac{I}{I_{0}} \right|_{j}$$
(9)

A more useful form of Eq. (8), that links the total absorbance *A* with concentration of each absorbing component, is:

$$\sigma_{1,j}n_1 + \sigma_{2,j}n_2 + \ldots + \sigma_{N,j}n_N = -\frac{1}{L}A_j$$
⁽¹⁰⁾

Therefore, the concentration of *N* different species can be determined through *N* measurements of the absorbance at *N* different wavelengths *j* through the following linear system of equations (Eq. (7)):

$$\begin{bmatrix} \sigma_{11} & \dots & \sigma_{1N} \\ \vdots & \ddots & \vdots \\ \sigma_{N1} & \dots & \sigma_{NN} \end{bmatrix} \begin{pmatrix} n_1 \\ \vdots \\ n_N \end{pmatrix} = -\frac{1}{L} \begin{pmatrix} A_1 \\ \vdots \\ A_N \end{pmatrix}$$
(11)

or synthetically, using i (i-species) and j (j-wavelength) pedixs:

$$[\sigma_{i,j}][n_i] = -\frac{1}{L}[A_j]$$
(12)

A practical case of OAS analysis is illustrated in *Chapter 6.3*, along with the choice of wavelengths and their associated cross sections.

6. Characterization of a surface dielectric barrier discharge

A practical example of the description and characterization of a plasma source employed in food processing studies is here reported. This chapter is aligned with the proposed approach aimed at adopting a common language for the description of sources and experimental setups and at providing a bulk of significant data sufficient to enable the reproducibility of performed experiments.

6.1. Plasma source and operating conditions

- . .

The plasma process presented here is designed to test the decontamination efficacy of an indirect CAP treatment, which therefore sees no interaction between plasma and the material being treated; the only plasma components responsible for microbial inactivation are the reactive species created within the treatment volume.

A schematic of the plasma system adopted in this study, Plasma Assisted Sanitation System (PASS) developed by AlmaPlasma srl, is shown in Fig. 5 (Foligni et al., 2022b; Maccaferri et al., 2023; Molina-Hernandez et al., 2022). PASS is composed of an SDBD plasma source, a



Fig. 6. PASS.



Fig. 7. Schematic representation of the electrical characterization setup.

treatment chamber, a cooling system, and a high-voltage generator (see Fig. 6). The SDBD consists of a mica dielectric layer (2 mm thick) interposed between 4 liquid-cooled high voltage (HV) aluminium electrodes and 4 grounded electrodes (AISI 316 L mesh). The treatment chamber is made of PVC and has two optical accesses (quartz windows) to allow optical spectroscopy; when the SDBD is placed on the top of the treatment chamber a 17.5 L closed volume is defined. The operating conditions employed in the present example are reported in Table 2.

CAP was homogenously generated on the surface of the mesh on an area of around 420 cm^2 . The device, operating in environmental air (relative humidity in the range 20–40 %), was driven by a sinusoidal

generator (by AlmaPlasma srl, Bologna, Italy).

6.2. Electrical characterization

The electrical characterization setup used for this example is schematically reported in Fig. 7.

The liquid-cooled electrodes of the plasma source are connected to a sinusoidal high voltage power supply, operated at a peak voltage of 6.3 kV and frequency of 23 kHz, while the rhomboid mesh is grounded. The plasma discharge is generated between the holes of the mesh in static air.

The applied voltage (*V*) and the current (*i*) are measured using a high voltage probe (Tektronix P6015A, attenuation 1000:1), located on the high voltage cable between the power supply and the plasma source, and a current probe (Pearson 6585), situated on the grounded cable. The charge (*Q*) is evaluated measuring the voltage across a monitor capacitor of 15.3 nF (connected in series with the plasma source) by means of a low voltage probe (Tektronix P6139A, attenuation 10:1). All the corresponding waveforms are recorded using a digital oscilloscope (Tektronix DPO4034, 350 MHz, 2.5 GSa/s), in which a total acquisition time of 400 µs (i.e. around 9 complete voltage periods) and number of points of 100,000 (sample rate: 0.25 G samples per second) are set.

The average discharge power dissipated over the period is determined using both the standard and the Lissajous method according to the formulas (1) and (5) presented in *Chapter 2*. Eighteen replicates are involved in the power calculation; hence, the results are shown as mean value \pm standard deviation.

Fig. 8 shows sinusoidal current and voltage waveforms (related to four complete applied voltage periods) representative of the electrical steady-state behavior of the plasma source operating. These waveforms represent a useful tool to check the operating conditions set at the high voltage generator (e.g. peak voltage and associated frequency).

During the applied voltage period (around 44 μs), the presence of discharge activity can be clearly distinguished by the appearance of multiple spikes (more clearly observable in the zoom proposed in Fig. 8) of nanosecond duration in the current waveform. The average discharge power calculated with the standard method directly from the measured current and voltage is 200.3 \pm 15.4 W.

The Lissajous figure obtained in the aforementioned operating





Fig. 8. Current and voltage waveforms at 6.3 kV and 23 kHz.



Fig. 9. Lissajous figure at 6.3 kV and 23 kHz.

conditions is reported in Fig. 9.

As can be observed from Fig. 9, the Lissajous figure exhibits the "almond-like" shape frequently observed in SDBD plasma sources (Pipa and Brandenburg, 2019). This shape differs from a perfect parallelogram, therefore the figure cannot be used to gather information about the plasma source characteristic capacitances. Nonetheless, it allows to calculate the dissipated discharge power as its area multiplied for the frequency, leading to a value of 201.1 \pm 9.6 W. This result is perfectly in agreement with the one obtained using the standard method, corroborating the validity of the measurements.

6.3. OAS analysis

The setup for OAS is schematically represented in Fig. 4. A 255 nm LED and a 400 LED were used as light sources. The light beam was focused by means of a fused silica lens (50.8 mm of focus length) to achieve a parallel beam passing under the mesh to investigate the

Table 3

Absorption cross-sections in cm^2 of the species of interest at each selected wavelength. ()* are values estimated by interpolation due to insufficient data to perform an average.

Selected wavelength	O ₃ cross-section	NO ₂ cross-section
$\begin{array}{c} 253.0 \pm 1,2 \text{ nm} \\ 400.0 \pm 1,2 \text{ nm} \end{array}$	(1.12 ± 0.02) E-17 (1.12 ± 0.08) E-23	(1.1 ± 0.3) E-20 (6.4 ± 0.2) E-19

plasma afterglow; the beam is then collected into a 500 mm spectrometer (Acton SP2500i, Princeton Instruments) and spectrally resolved in the UV, VIS and near infrared (NIR) regions. OAS acquisitions were performed using a grating with a resolution of 150 mm^{-1} and setting a width of 10 µm for the inlet slit of the spectrometer. A photomultiplier tube (PMT-Princeton Instruments PD439) connected to a fast oscilloscope (Tektronix MSO46) was used as detector to allow for fast acquisitions (time resolution of 40 ms). The PMT amplification factor was kept constant for all acquisitions. In order to ensure identical initial conditions, the discharge chamber was opened and flushed for 300 s with fresh air prior to each measurement. The quantitative evaluation of the species concentrations from absorption measurements was performed according to the theory described in *Chapter 5*.

The wavelengths selected to perform the study and the corresponding absorption cross-sections for O₃ and NO₂ as absorbers are reported in Table 3. These wavelengths were defined, in accordance with Moiseev (Moiseev et al., 2014), to maximise the absorption of the molecules of interest (O₃ and NO₂) while minimising the contribution, and thus the disturbance, of other absorbing molecules. The cross sections $\sigma_{i,j}$ for O₃ and NO₂ molecules are reported in Table 3.

For all experiments, the length of optical path L is 25 cm and the contributions of background radiation and spontaneous plasma emission were duly taken into account in the data processing, subtracting them from the acquired values of I and I_0 .

The temporal evolutions of O_3 and NO_2 are reported in Fig. 10. More details on the use and of the potential of OAS for the characterization of SDBD devices can be found in (Simoncelli et al., 2019b).

7. Conclusions

Cold atmospheric plasmas (CAPs) are a promising technology for



Fig. 10. Temporal evolution of O3 and NO2 concentrations during a PASS treatment.

non-thermal food treatment, with possible roles in the biodecontamination of pathogens to reduce spoilage and increase shelflife, in the enhancement of nutritional properties and in food functionalization.

The diversification of the scientific base researching the field of CAP food processing is instrumental for its development, but at the same time poses a communication challenge: a common terminology and sensibility to mandatory information/data should be adopted to guarantee the replicability of performed experiments and a systematic and synergistic development of the field. From the point of view of researchers with a plasma physics/chemistry/engineering background, most published papers on the topic do not provide enough information on plasma devices and properties to compare results across laboratories and to properly assess and, potentially, replicate the experiments.

In this paper, basic principles of CAP technology and its application in food processes are reviewed and recommendations for providing a schematic and exhaustive description of a CAP process are proposed. Relevant parameters of widespread use for describing plasma discharges are also introduced, along with characterization techniques suitable for their measurement; in this regard, electrical measurement and optical absorption spectroscopy are discussed in detail as relatively accessible means to detail important physical and chemical aspects of plasma discharges.

It is our hope that this viewpoint paper can contribute to a standardization of the description of CAPs used in studies on food processing, with the aim of accelerating the expansion of the knowledge base towards the commercial exploitation of the technology.

CRediT authorship contribution statement

Filippo Capelli: Writing – original draft, Investigation, Data curation, Conceptualization. **Giulia Laghi:** Writing – original draft, Investigation, Data curation, Conceptualization. **Romolo Laurita:** Writing – review & editing, Resources, Conceptualization. **Nevena Puač:** Writing – review & editing. **Matteo Gherardi:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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