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DEVELOPMENT OF A NEW DEVICE FOR APPLYING ATMOSPHERIC PRESSURE COLD PLASMA ON FOODS

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Abstract: *The studies on cold plasma, a non-thermal food processing technology, have been recently focused on extending the shelf life of foods and controlling pathogenic microorganisms since its antimicrobial activities were recognized. Atmospheric pressure cold plasma is a cost-effective and user-friendly technique since it does not require a vacuum environment and apparatus. In this study, a new device that produces atmospheric pressure cold plasma in large volumes has been designed and produced to treat various types and properties of food. The system can operate at three different modes, producing direct (Device mode-I) and indirect (Device mode-II) Volume Dielectric Barrier Discharge (VDBD) and Surface Dielectric Barrier Discharge (SDBD) plasma (Device mode-III). The system can be operated consecutively without any problems. The cold plasmas produced in three different modes were applied to different food types, and the foods were examined whether cold plasmas caused a noticeable change in sensory properties. The reactions caused by the system under different operating conditions were measured by optical emission spectroscopy, and the chemical interactions that the foods might be exposed to during plasma treatment were determined. The types of plasma particles that can be effective in food-plasma applications were discussed. The sensory analysis results showed that various foods such as chicken, fish, apple, and milk could be treated with atmospheric pressure cold plasma without causing significant changes in their sensory properties.*

Keywords: *Cold Plasma; Dielectric Barrier Discharge; Food Safety; Sensory; Quality.*

1. Introduction

Plasma, referred to as the fourth state of matter, occurs when high energy is applied to a gas. In other words, when any gas is energized, it ionizes, and free electrons and ion groups are formed. This state, generally named ionized gas, is called 'plasma' if it has a certain ionization rate. In the plasma state of matter, neutral atoms and molecules, ionized atoms and molecules, excited atoms and molecules, high-energy excited (metastable) atoms and molecules, electrons, radical particles (such as O, OH, NO), and photons are formed. Moreover, significant amounts of ultraviolet (UV) light and ozone are also produced. These plasma particles with different properties are used very effectively in lighting, medicine, food, and agriculture, as well as coating and electronic technologies. It has been observed that cold plasmas have antibacterial effects without producing any heat and are also effective on different microstructures such as fungi and biofilms

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[1]. Plasma can sterilize the air in air conditioners. Additionally, it can be used to treat bacterial and viral infectious diseases and to sterilize blood. It is also used in treating burns and wounds, oral-dental health and dental caries, different types of cancers, ulcers such as corneal ulcers, in the coagulation of blood for seamless surgery and various skin improvements such as wrinkles and brown spots. The recent application areas of cold plasmas are stem cells, cell mutation, reproductive cells, and neurology [2].

In recent years, it has been observed that cold plasma inactivates bacteria and viruses with the effect of oxygen and nitrogen radicals, UV radiation and charged particles. Thus, the studies have focused on using plasmas to produce safe food with high quality and extended shelf life. It has been reported that cold plasma can inhibit pathogen growth in cereals and vegetables [3], reduce spoilage microflora [4], decontaminate surfaces [5], delay metabolic activity [6], reduce peroxidase [7] and lipase – lipoxygenase activities [8]. In animal-based foods, cold plasma reported to inhibit pathogens [9-11], extend microbiological shelf life [12-15], and help to maintain quality [16-17].

Cold plasmas are considered strong alternatives to traditional food preservation methods due to their advantages, such as being effective at low, providing powerful antimicrobial effect, not producing toxic by-products, not causing significant damage to food products and being low cost [18]. In addition, plasma is a harmless and environmentally friendly technology without requiring any chemicals and leaving toxic residues [19]. Dielectric Barrier Discharge (DBD) is a system that successfully produces cold plasma and is safe for the people who apply it [20]. However, the equipment and treatment conditions must be validated on food products and target organisms to be commercially applicable [21].

It is important to investigate the effects of cold plasma technology in foods because it represents a potential technology for surface decontamination, shelf life extension, pathogen control and quality improvement. For the in vitro studies, it may be sufficient to generate cold plasma in a very small area, but for food studies, it is necessary to create it in an area where food can be placed comfortably. This study aimed to develop a plasma system with a sufficient volume suitable for treating foods with different types and sizes. A cheap, user-friendly and practical cold plasma device to be used in industrial applications was intended.

2. Materials and methods

2.1. Atmospheric pressure cold plasma device

The system designed and manufactured in this study consists of 2 cm thick polyethylene material. The system is 16 cm in height, 16 cm in width, 80 cm in length, with an internal volume of approximately 15 liters. A 4 mm-thick plexiglass cover that can move within the channels opened in the main body is placed on the front side of the chamber. The food can be placed in the chamber for cold plasma treatment by opening and closing this cover.

The device has been designed and manufactured to provide three different cold plasma environments. A direct plasma field (Device Mode-I) is formed on the right side of the device, and an indirect plasma field (Device Mode-II) is formed on the left side; meanwhile, helium gas is added into the chamber. The system has two copper electrodes with a width of 12 cm, length of 36 cm, and thickness of 1 mm. The electrode on the bottom base is placed in a plexiglass material (14 mm in width, 38 mm in length and 4 mm in thickness) to minimize the accumulation of charge on the system. Helium gas is filled from the back of the system between two parallel-placed copper electrodes.

Surface Dielectric Barrier Discharge (Device Mode-III) is created by removing the upper electrode of the device and replacing it with another electrode, consisting of a one mm-thick copper cable placed zig-zag on the plexiglass material. The dimensions of this electrode are 12 cm × 40 cm × 4 mm. Additionally, an alternating current (AC) power supply (30 kV and 20 kHz) was designed, produced, and used in the study. A 1000X high voltage probe was used for oscilloscope measurement of this power supply used during the study.

2.2. Sensory analysis of cold plasma-applied foods

Our device is designed and produced to expose foods to cold plasma to extend their shelf life or prevent pathogen growth. Regardless of the provided effect on shelf life or pathogen control, consumer acceptance of the product is the most important criterion for using relevant technology. For this reason, it is of great importance to know whether there is a sensory loss in foods exposed to cold plasma produced by our device. For this, different types of foods (chicken, fish, apple, and milk) were exposed to cold plasma for 2 minutes by the three different modes of our device and the effect of the process was examined by sensory analyzes. Ten panelists experienced in sensory evaluation participated in the sensory analysis. Food samples were coded with randomly generated 3-digit numbers and presented to the panelists in white plates. The sensory panel was carried out under daylight in a well-ventilated room. It was ensured that panelists made the sensory evaluation with a physical distance to prevent their interaction. Color, odor, texture, and taste parameters were evaluated. For the taste parameter, the cooking process of the chicken meat (breast) and fish samples (red mullet, *Mullus barbatus* Linnaeus, 1758) was done in odorless glass jars using the bain-marie method. The apple (*Malus domestica*, Borkh., 1803) and milk (UHT-cow milk) samples were tested at room temperature, without any heating process. The classification proposed by [22] was used in sensory evaluation (9: excellent, 8: very good, 7: good, 6: above average, 5: moderate, 4: below average, 3: bad, 2: very bad, 1: extremely bad).

During the sensory panel, the panelists also asked, "Would you buy this food?" The options "I definitely don't buy (1 point), I probably don't buy (2 points), I may or may not buy (3 points), I probably buy (4 points), I buy definitely (5 points)" options were presented [23-24]. This test aimed to determine whether the cold plasma application significantly changes the purchasing preference of the consumer. Sensory evaluations were also performed on control samples that were not treated with plasma. The SPSS program (SPSS 16.00, Chicago, IL, USA) was used for the statistical analysis. The significance level was chosen as $P < 0.05$.

3. Results and discussion

3.1. Atmospheric Pressure Cold Plasma Device

The pressure of the gas used for plasma production is the most important parameter affecting the plasma temperature. It is quite easy to produce cold plasma even when a voltage of 1-5 kV is applied as direct current (DC) at pressures as low as 10^{-2} mbar. However, at least 20-30 kV high voltages are needed to produce plasma at high pressures around 1 atmosphere. These high voltages result in the occurrence of thermal plasma. The electrode design and the voltage frequency used to produce cold plasma at high pressures are important [25]. The electrode design of the system produced in this study and the applied voltage frequency constitute the original aspect of the study. The system has been specially designed to produce cold plasma at atmospheric pressure in an area large enough for food applications. In the Dielectric barrier discharge, one of the electrodes is covered with an insulator, preventing the generation of secondary electron emission in DC Glow discharges. Thus, the formation of thermal plasma is prevented. Accordingly, our system produces Dielectric Barrier Discharge (DBD) between the electrodes, thanks to the insulating material coating of the lower electrode. The electrodes of our device prevent secondary electron emission, and the type of voltage we use is another important parameter that prevents the heating of the plasma.

The production of cold plasma at atmospheric pressure is highly dependent on the type of gas used and the electrode design of the power source [10]. Since the effects of different types of gas on foods can vary, the system is designed to work by sending gas from outside. %99.999 pure helium can be sent to our device using the gas flow meter with sockets and a pneumatic hose from the He gas tank regulated with a regulator. The gas flow meter is mounted on the system and can measure the gas flow between 0-15 liters/minute. When the average gas flow is set as 8-10 liters/minute, the distance between the electrodes as 5-6 cm and the electrodes are powered, a visible plasma is generated (Figure 1). The plasma formed between the copper plates on the right side of the device can be applied directly to the food placed in this area (Direct plasma field, Device Mode-I). The plasma products formed can be transported to the left side with the help of the fan in the middle of the system and can be applied indirectly to the food placed on this side of the device (Indirect plasma field, Device Mode-II). The helium gas, continuously sent to the system, also supports the transport of the particles produced by the plasma generated between the electrodes from the right to the left side (Figure 1). Thus, the plasma particles indirectly affect the food placed in the area far from the electrodes on the device's left side. Indirect plasma application is defined as exposing the sample to the indirect effect of plasma by leaving a distance between the sample and the plasma source. It is stated that the effects will be different from that of the direct plasma [26-27].

Plasma produces many different particles such as invisible electrons, ions and radical particles, and photons. When plasma is applied indirectly to a food sample, the effects of invisible particles produced in the plasma are in question. Determining the indirect effect of cold plasma is important for industrial applications. Because generating the plasma in large areas is more expensive due to high energy requirements. When plasma is applied to large-sized foods in commercial applications, each part of the food may not fit in the plasma field (a large fish, food parcel, fish box, etc.). So, it is important to know the effects provided by the indirect plasma to understand the effects on the food parts that do not directly enter the plasma field. Our device works with Helium gas in Device Mode-I (Direct plasma) and Device Mode-II (Indirect plasma). Since gas-operated systems may not be preferred in industrial applications due to additional costs and the need to use gas, the other mode (Device Mode-III) was designed since it works with atmospheric air. Accordingly, the upper electrode of the device is removed, and another electrode is placed on it, on which a copper cable of 1 mm thickness is placed in a zig-zag shape.This mode of our device (Device Mode-III) creates Surface Dielectric Barrier Discharge (SDBD) with atmospheric air, thanks to this specially designed electrode (Figure 2). Figure 2a shows the electrode design, Figure 2b shows its assembled state, and Figure 2c shows the Surface Dielectric Barrier Discharge Plasma formed in purple color.

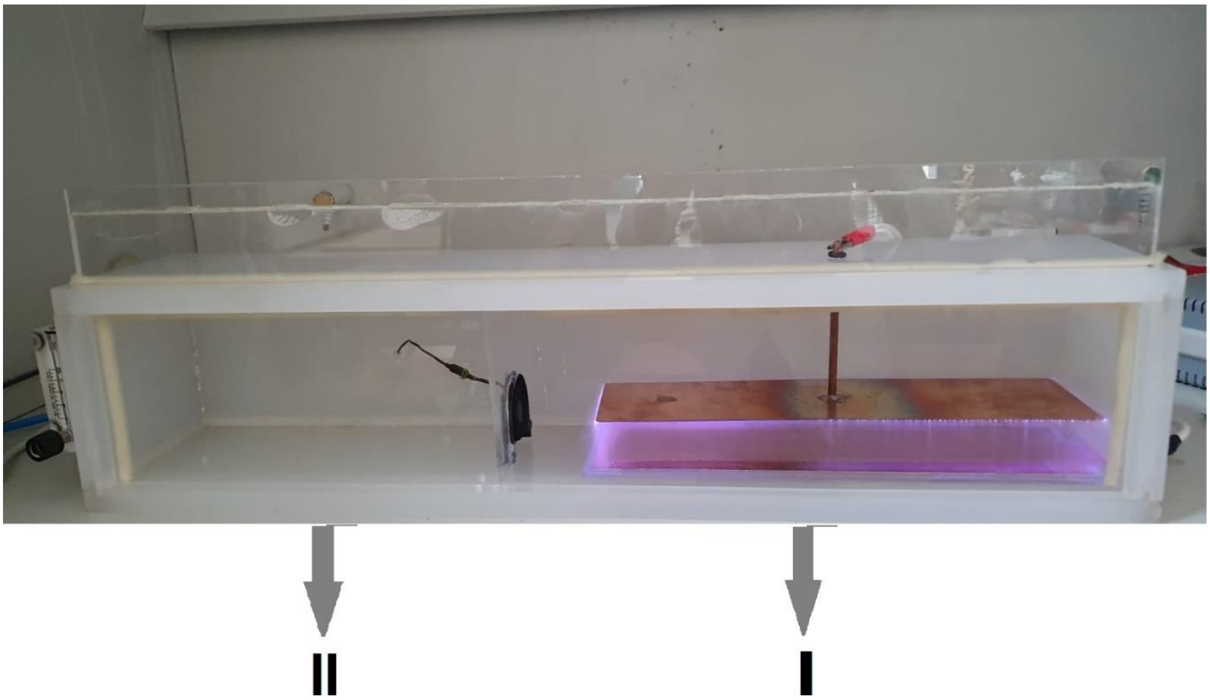
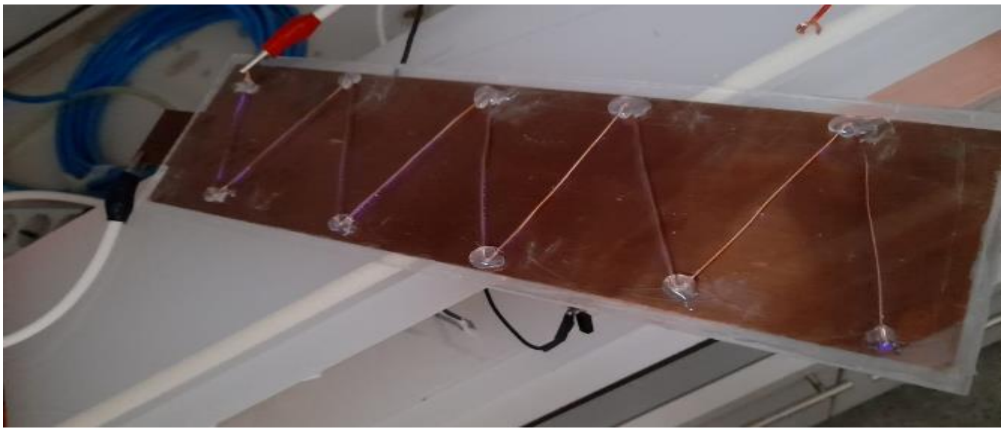
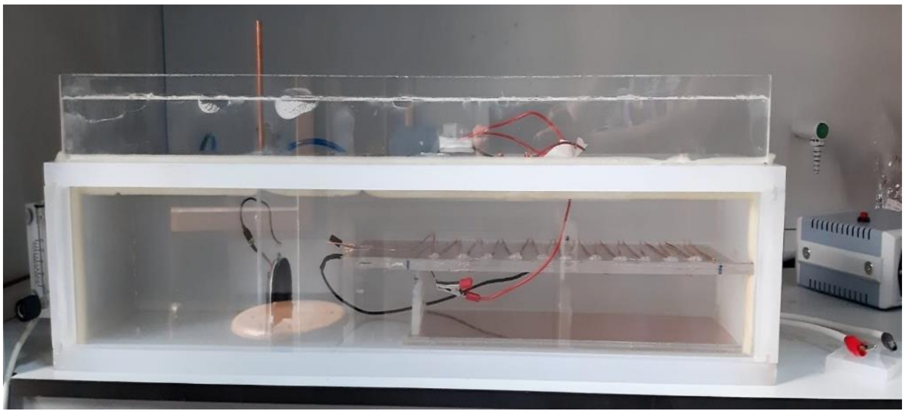


Fig.1. Large Volume Atmospheric Pressure Cold Plasma System, Direct (Device Mode-I) and Indirect (Device Mode-II) cold plasma fields



2. a



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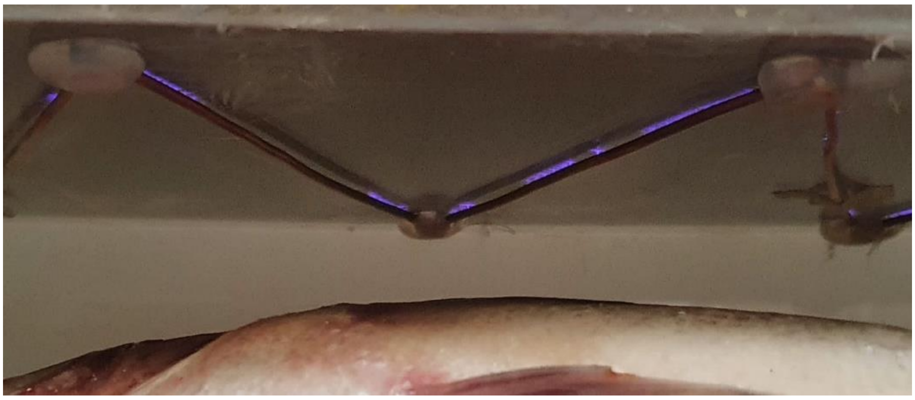

2. c

Fig. 2. Device Mode-III, Surface Dielectric Barrier Discharge (SDBD) with atmospheric air
(**2a**. Electrode design of Surface Dielectric Barrier Discharge, **2b**. mounted electrode on the system, **2c**. surface dielectric barrier discharge plasma formed in purple color)

3.2. Application of cold plasma to various foods with the designed device

Apple, fish (red mullet), milk and chicken (breast) were exposed to cold plasma produced by different device modes. Their sensory characteristics such as taste, smell, texture, and color were evaluated. As a result of all cold plasma applications (Direct-I, Indirect-II, SDBD-III), it was observed that all food samples preserved their sensory-like properties. None of the cold plasma treatments caused a significant change in the sensory scores of the samples, and all foods retained their sensory acceptability (Table 1).

Table 1.
Sensory evaluation of foods treated with cold plasma by three different modes of the device

Sensory test scores (Mean ± SD)	Control	Direct Plasma	Indirect Plasma	SDBD Plasma
Apple	8.16±0.37 [a]	8.16±0.37 [a]	8.00±0.50 [a]	8.00±0.50 [a]
Fish (Red mullet)	7.28±0.84 [a]	7.56±0.71 [a]	7.32±0.95 [a]	7.28±0.94 [a]
Milk	8.32±0.56 [a]	8.36±0.49 [a]	8.36±0.49 [a]	8.24±0.66 [b]
Chicken (breast)	7.76±0.52 [a]	7.68±0.56 [a]	7.64±0.57 [a]	7.60±0.58 [ab]

[a,b]: *The same letters on the same row indicate that the statistical difference is not significant (P ≤0.05).*

Panelists were also asked, "Would you buy this product?". All cold plasma-treated foods were evaluated as "probably" or "definitely" would buy. Thereby, it has been concluded that plasma-treated foods kept their marketable - preferable properties (Figure 3). None of the panelists gave the answers "I definitely don't buy", "I probably don't buy" or "I may or may not buy".

When the food is placed between the electrodes, the UV light, ozone, radical oxygen (O), radical hydroxyl (OH), excited and ionized particles and electrons produced by the plasma affect it. The high electromagnetic field applied between the electrodes is also effective on food. Particles produced in plasma, especially UV light, ozone, radical oxygen (O), radical hydroxyl (OH) and excited particles, create an important antibacterial effect on the food with which they interact [28]. Therefore, the use of cold plasma to increase the shelf life of various foods or to control pathogens constitutes a new and important area of study. Although the applied process will provide significant benefits in terms of food safety, quality or shelf life, food preference for the consumer is usually based on sensory properties, and consumer acceptance is indispensable for the food to be sold. For this reason, it is important whether cold plasma causes a noticeable sensory change in the food to which it is applied.

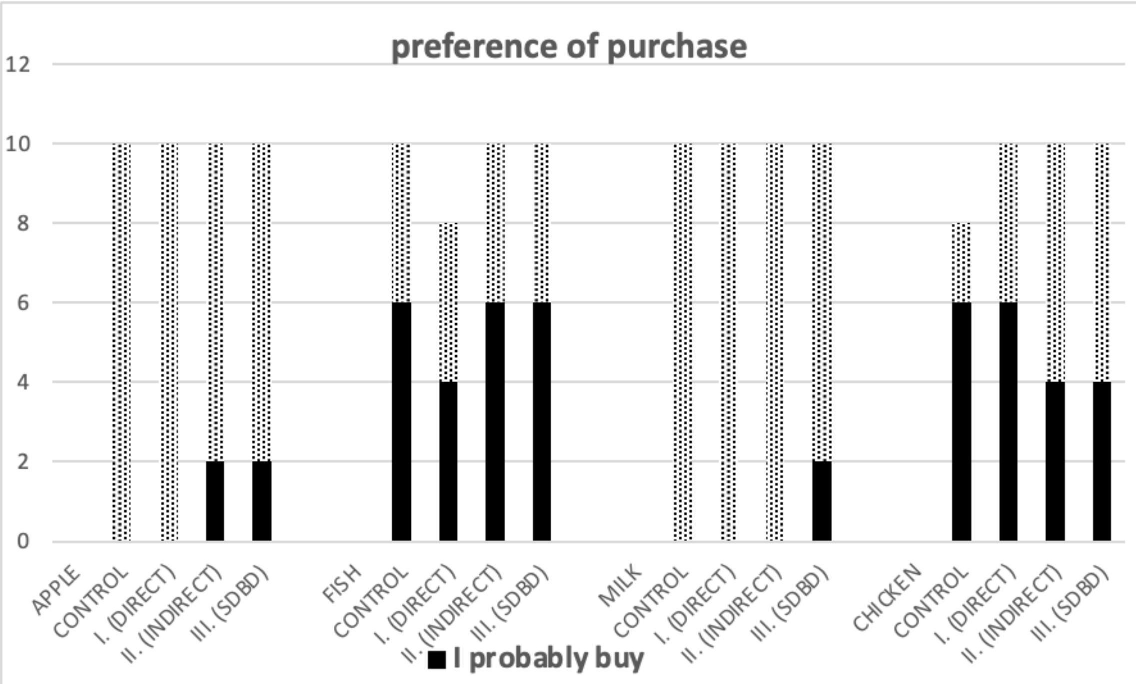

Fig. 3. Response of panelists to the question "Would you buy this product?"

In literature, there are few studies on sensory changes of foods treated with cold plasma. Chiper & al. [29] stated that the cold plasma applied to the fish in the package filled with Ar/CO_2 mixture deteriorates the sensory quality as the processing time increases but did not share the sensory analysis findings. Kim & al. [30] observed significant reductions in sensory parameters of bacon treated with plasma for 5 and 10 minutes. On the other hand, Chen & al. [31] reported positive effects of atmospheric cold plasma treatment (1 min.) on the sensory properties of chub mackerel. Choi & al. [16] applied cold plasma to dried squid for 1, 2, and 3 minutes and stated that there was no significant difference in sensory scores after treatment. In another study, no change in sensory properties was reported in frozen pork meats treated with cold plasma, while plasma application caused some sensory changes in fresh samples [32]. In our study, the cold plasma applied by three different modes of our device for 2 minutes did not cause a noticeable change in the sensory properties of apple, chicken, fish, and milk. Therefore, our device can successfully apply atmospheric pressure cold plasma to different food types.

3.3. Plasma Characterization of Atmospheric Pressure Cold Plasma System

Reactive Oxygen Species (ROS) and Reactive Nitrogen Species (RNS) are called free radicals. Both ROS and RNS are key regulators of processes such as metabolism, growth, development, and cell death. The production of various ROS and RNSs has been confirmed to have antibacterial activity. Plasma can produce ROS/RNS in the presence of atmospheric oxygen and nitrogen. Ultraviolet (UV) radiation, charged particles, energetic ions, reagents such as ROS and RNS can be generated using air as the working gas. Among ROS, ozone, atomic oxygen, singlet oxygen, superoxide, peroxide, and hydroxyl radicals are thought to participate in bacterial inactivation [28]. The generation of cold plasma in the environment can trigger oxidation, nitration, hydrolysis, and amination reactions that cause bacterial death [33]. Plasma generates reactive oxygen and nitrogen species, disrupting the membrane integrity and thus damaging the bacteria [34]. The particles produced in the plasma generated by our device are circulated in the chamber with a fan. In this manner, the food placed far from the plasma generating area can still be exposed to the plasma products.

Analyzing the properties of the plasma system and the types of plasma particles is a crucial process to determine the operating conditions and possible effects of plasma particles on food samples [35]. Thus, the plasma reactions that lead to positive effects in the applied food can be detected. By finding the most efficient plasma reaction, the system can be used very efficiently. Suppose any particle in the plasma creates a positive change in sample quality dominantly. In that case, plasma production conditions can be adjusted to produce more particles of that type. If any particle produces an undesirable effect on the sample, it can be minimized by adjusting the system parameters. Therefore, a spectroscopic study was observed to determine the plasma particle types produced by our system.

Table 2 presents the main chemical reactions and particle types produced by any plasma system operating with helium gas at atmospheric pressure. Although it may be known from the reaction rate coefficients which of these reactions occur, they may differ according to the characteristics of the plasma system. For this reason, spectroscopic measurements of the produced plasma are decisive about the reactions in the plasma system [35].

Optical emission spectroscopy results from our atmospheric pressure cold plasma system provide information about these reactions and particle types. In Figure 4, the optical emission spectrum of the helium plasma produced at atmospheric pressure without any food in the system is given. The spectrum was taken with the Ocean Optics USB2000+ device. When helium gas plasma is produced at atmospheric pressure with our device, transitions at 380 (N_2), 391 (N_2^+), 399 (N_2), 405 (N_2), 419, 427 (N_2^+), 434, 470, 587 (He), 668 (He), 707 (He), 728 (He) ve 777 (O) nm wavelengths are observed (Figure 4).

Table 2.

Important reactions that can occur in Helium plasma at atmospheric pressure [35].

Interaction	Reaction
Helium atom interactions	$He + e^- \rightarrow He^* + e^-$ $He + e^- \rightarrow He^+ + 2e^-$
Helium excited atom interactions	$e^- + He^* \rightarrow He + e^-$ $He^* + e^- \rightarrow He^+ + 2e^-$ $He^* + 2He \rightarrow He_2^* + He$ $He^* + He^* \rightarrow He_2^+ + e^-$ $He^* + He^* \rightarrow He^+ + He + e^-$ $He_2^* + M \rightarrow 2He + M$ $2He^* \rightarrow He_2^+ + e^-$ $2He_2^* \rightarrow He_2^+ + 2He + e^-$
Helium ion interactions	$He^+ + e^- + e^- \rightarrow He + e^-$ $He^+ + e^- + e^- \rightarrow He^* + e^-$ $He_2^+ + e^- + e^- \rightarrow He + He + e^-$ $He_2^+ + e^- \rightarrow He^* + He$ $He_2^+ + e^- \rightarrow He + He$
Water interactions	$H_2O + e^- \rightarrow H + OH + e^-$ $H_2O + O^* \rightarrow 2OH$ $He^* + H_2O \rightarrow OH^* + H + He$ $He^* + H_2O \rightarrow O^* + H_2 + He$ $He^* + H_2O \rightarrow OH + H^* + He$
Nitrogen interactions	$N_2^+ + e^- \rightarrow N_2$ $He^+ + N_2 \rightarrow N^+ + N + He$ $He^+ + N_2 \rightarrow N_2^+ + He$ $He_2^+ + N_2 \rightarrow N_2^+ + He + He$ $He^* + N_2 \rightarrow N_2^+ + He + e^-$ $He_2^* + N_2 \rightarrow N_2^+ + 2He + e^-$ $He_2^+ + N_2 \rightarrow N_2^+ + He_2^*$ $N^+ + e^- + He \rightarrow N + He$
NO interactions	$NO^- + He \rightarrow NO + He + e^-$ $NO^+ + O_3^- + He \rightarrow NO + O_2 + O + He$
Oxygen interactions	$e^- + O_2 + He \rightarrow O_2^- + He$ $O^- + O_2 + He \rightarrow O_3^- + He$ $O^+ + N_2 + He \rightarrow NO^+ + N + He$ $O + O + He \rightarrow O_2 + He$ $He^+ + O_2 \rightarrow O^+ + O + He$ $He^+ + O_2 \rightarrow O_2^+ + He$ $O + O_2 + He \rightarrow O_3 + He$ $O^+ + e^- + He \rightarrow O + He$

The excited particle transitions were given on the corresponding peaks. These transitions are standard and are provided collectively at NIST (www.nist.gov). As expected, since Helium plasma is generated in the atmospheric environment, a large number of nitrogen reactions occur, and therefore nitrogen ions and excited particles are present in the plasma. Helium peaks appear at standard wavelengths as expected. At 777 nm, radical oxygen is seen. In this case, the food treated in our atmospheric pressure cold plasma device will interact with the nitrogen, helium, and radical oxygen atoms.

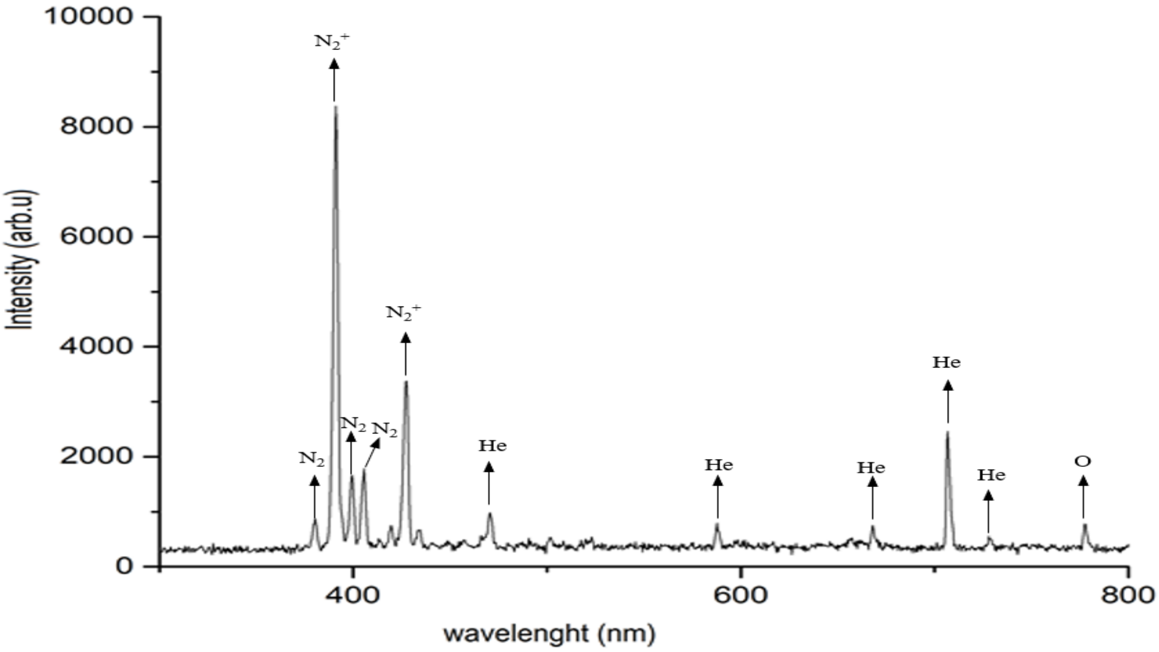

Fig. 4. The optical emission spectrum of atmospheric pressure cold plasma system helium plasma

Our atmospheric pressure cold plasma device uses atmospheric air as gas to produce Surface Dielectric Barrier Discharge (SDBD) at the Device Mode-III. In this case, the optical emission spectrum from the plasma is given in Figure 5. As seen in Figure 5, the produced atmospheric pressure air plasma emits radiation at 297 nm wavelength of NO gas; and 315 nm, 337 nm, 353 nm, 357 nm, 374 nm, 379 nm, 398 nm and 405 nm wavelengths of the nitrogen molecule. As is seen, almost all of them are below 400 nm, which can be considered ultraviolet radiation. Our system produces ultraviolet with very high antibacterial activity at Device mode-III, working with atmospheric air. In addition, when the system operates using helium gas (Device modes I and II), radical oxygen with a very high antibacterial activity is formed (777 nm) (Figure 4). When the system works with atmospheric air at Device mode III, it produces NO, a radical with high antibacterial activity (Figure 5). In both cases, all these formations are important for sterilization and inactivation. The results achieved in food applications can be evaluated in terms of interaction with the particle types produced by the plasma.

The system can work for 30 minutes without interruption, and it can work throughout the day if 15-minute applications are made consecutively. While our device was operating in all three modes, measurements were made with an ozonometer, and it was determined that the created plasma contained ≥1 ppm ozone. Considering ozone above one ppm is enough for antibacterial effect [36], one may conclude that the efficacy of cold plasma may increase due to this high amount of ozone.

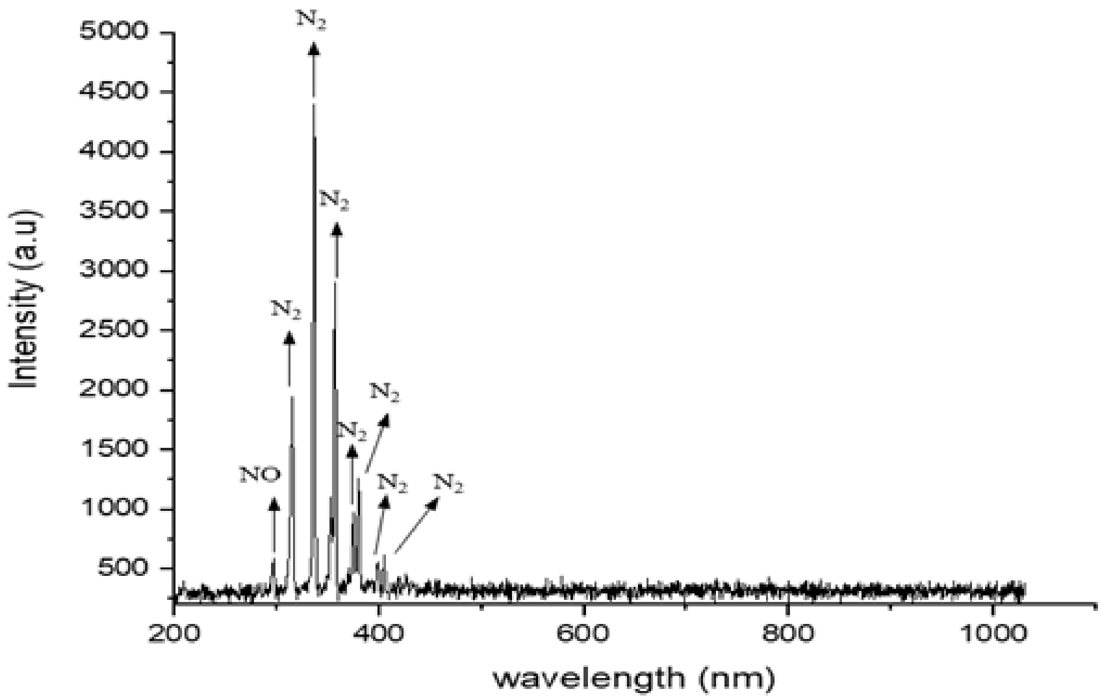

Fig. 5. The optical emission spectroscopy in a Surface Dielectric Barrier Discharge (SDBD) plasma system

4. Conclusion

A new device that produces cold plasma at atmospheric pressure with a sufficient volume has been designed and manufactured to apply cold plasma to foods. The system can produce Volume Dielectric Barrier Discharge and Surface Dielectric Barrier Discharge plasmas. The device can be operated in three different modes. These three modes of the device can provide direct or indirect plasma application to foods, work with helium gas or create plasma from atmospheric air without needing an additional gas. The system can work for 30 minutes without interruption, and it can work throughout the day if 15-minute applications are made consecutively. In this study, by taking the optical emission of the plasmas produced in the device, the effective plasma types produced in the plasma are revealed, and their possible effects in food applications are discussed. Treatment of various foods such as chicken, fish, apple and milk with atmospheric pressure cold plasma applied by three different modes of our device for 2 minutes did not cause any significant change in sensory characteristics. It was concluded that this system would be a suitable device for applying plasma to foods to control pathogen growth or extend shelf life.

5. Acknowledgment

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6. Conflicts of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be constituted as a conflict of interest regarding the publication of this paper.

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