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# Feasibility study of atmospheric-pressure plasma treated air gas package for grape's shelf-life improvement

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ABSTRACT

Recently, atmospheric-pressure plasmas have been studied for many types of microorganism sterilization in the food science because of its abundant active agents, including atomic oxygen, ozone, radicals, thermal energy, and UV radiation. In this work, a dielectric barrier discharge (DBD) plasma source generated by a 30 kHz low-frequency pulse power supply was used to treat air gases filling pouches. Electrical and spectroscopic investigations were performed to characterize plasma properties. Fourier transform infrared spectroscopy (FTIR) and an ozone detector were used to analyze the compositions of plasma treated air gases injected into the grape storage pouch. In addition, the effects of plasma on fruit qualities were probed through measurements of brix degree (%), acidity (pH), and mold enumeration. Microbiological analysis showed that filling the pouch with plasma treated gases allowed better storage conditions of fruits without degradation in fruit quality.



## 1. Introduction

Agricultural products are easily influenced by spoilage microorganisms including viruses, fungi, yeast, and bacteria [1–3]. These spoilage microorganisms can lead to deterioration in quality or spoilage of agricultural products during distribution or storage. In addition, due to the higher respiration rate and/or higher water activity of fresh foods, the fast quality deterioration and short shelf-life result in tremendous economic losses after harvest [4–8]. It is estimated that about 33 billion dollars of agricultural products in the United States are lost every year due to the spoilage [4]. For example, grape is one of important fruits, especially for Mediterranean diet consumed almost all year, but considered highly perishable fruit due to the severe quality losses after harvest because of weight loss, color changes, rapid softening and ripening [5,6]. In addition, grape is known as very sensitive to decay during storage by various fungi [5,6]. In order to reduce the loss of those agricultural products by removing the microorganisms, many methods such as pesticide, insecticide, modified atmosphere packaging (MAP), and plasma treatment have been developed and applied. Although conventional chemical methods can effectively kill harmful microorganisms, these methods remain environmental hazards that are toxic to humans [7,8]. The MAP can extend a product's shelf-life due to lower respiration rates and delay ripening of fruits by altering the oxygen and carbon dioxide [9]. However, the products stored by MAP method can be damaged due to high humidity and long storage during the wholesale stage and also accumulate solid waste because of its package material [8,9]. For these reasons, alternative methods such as the use of non-equilibrium atmospheric-pressure plasmas are still developing for improving shelf-life [10].

Plasma, the fourth state of matter, is ionized gas that emits light and contains balanced positively and negatively charged particles, free radicals, and reactive neutrals [11–13]. Plasmas have been applied to diverse processes such as semiconductor processing, coating, plasma polymerization, and biomedical applications [11–15].

Especially, atmospheric-pressure air plasma have been intensively studied to generate ozone because of its strong decontamination effect [7,16,17]. In addition, potent oxidants such as oxygen atoms and ozone generated by plasmas can penetrate into the cells

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of microorganisms to attack DNA molecules [2]. Since cell membranes are composed of proteins and lipids, proteins can react with reactive oxygen species (ROS) at the cellular level to alter ammonia side chains and protein structure [18,19]. These changes result in functional alterations that disturb cellular metabolism [18,19]. Based on these plasma effects on bio-cells, the application of plasma for food industries are promising [16–21]. Although plasma showed better decontamination effects on cells, it is also important to minimize thermal damage to fruit tissues by plasmas. Because most plasma treatment sources directly touch fruits and foods, the electrical and thermal damages caused by plasma should be considered [22–24]. In this work, therefore, an atmospheric-pressure plasma source was developed to flow plasma treated gas while avoiding direct contact between plasma and fruits and its effects on grape properties and shelf-life were evaluated. Electrical and Optical spectroscopic measurement were also performed to estimate the fundamental properties of the plasma. Fourier transform infrared spectroscopy (FTIR) was used to analyze the air characteristics after plasma treatment and fruit storage.

## 2. Experimental setup and methods

### 2.1. Samples preparation

Fresh grape bunches (Campbell Early) were purchased from a local market (Jeonju, Republic of Korea) and stored under refrigerated conditions at 4 °C for 1 h until the experiments. No bunches were washed prior to experiments. Grapes were stored in a Tedlar® gas sampling bag (C-type Dongbang Hightech) filled with air gas and plasma treated air gas, respectively. The gas sampling bag was made of Tedlar® polyvinyl fluoride (PVF) film (DuPont). Since the permeability of oxygen and nitrogen which are main components of air are reported as small as 3.2 cc/(100 $in^2$)(24 h)(atm)(mil) and 0.25 cc/(100 $in^2$)(24 h)(atm)(mil), respectively, the gas permeation of the bag is negligible in this work. The number of grapes was controlled as 15 in a bunch. Each experimental condition was conducted 3 times. All packaged fruits were also stored at room temperature.

### 2.2. Plasma generation and characterization

Fig. 1(a) presents the schematics of the atmospheric-pressure dielectric barrier discharge (DBD) plasma source and experimental setup. The DBD plasma source for air gas treatment was comprised of a cylindrical copper rod as an inner electrode (diameter: 3 mm, length: 110 mm) which was encircled by a dielectric tube (Quartz, outer diameter: 6 mm, length: 120 mm) and a copper coil electrode covering the tube. The coil electrode encircling the quartz tube was electrically grounded and the inner electrode was connected to a high-voltage source (APPP-020 EESYS). The high voltage was generated through an amplifier in the power supply while the high output voltage, $V_{out}$, was controlled by input dc voltage, $V_{in}$. Compressed air gases were fed inside the quartz tube with 15 liter per minute (lpm). Plasma treatment was conducted at atmospheric pressure, which took 5 s to fill the bag. Plasma treated air gases were collected into a Tedlar® gas sampling bag having a length of 16 cm and a width of 27.5 cm with or without grapes. The distance from the end of coil electrode to the inlet of Tedlar® bag was fixed as 60 mm which relatively long distance insure the minimization of the electrical and thermal damages caused by direct plasma contact.

To measure the plasma voltage and current, two electric probes (1000:1 voltage probe, PPE 20KV LeCroy and 10:1 voltage probe, PP 009 LeCroy) were connected to the inner electrode and a resistor (50 Ω), respectively. Electrical signal sampling was performed with

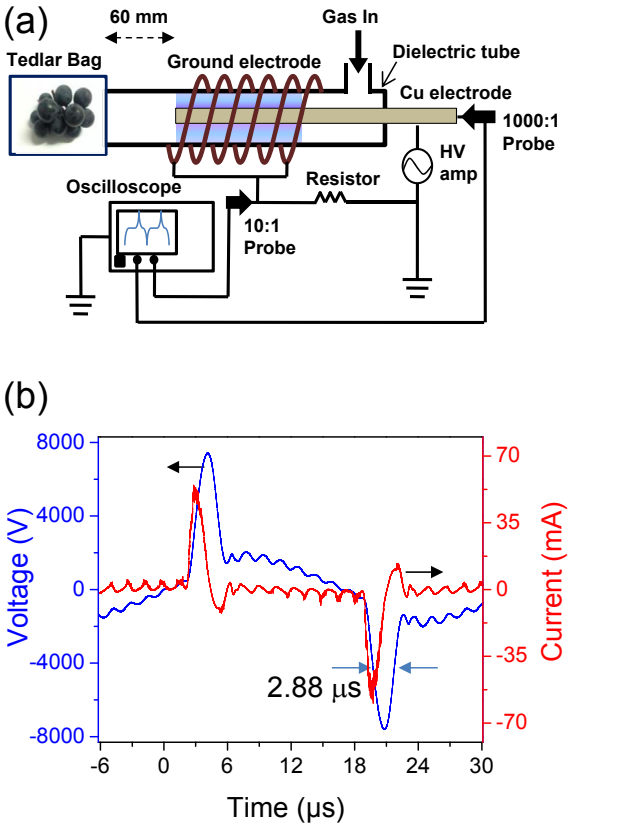


**Fig. 1.** (a) Schematic diagram of experimental set-up for atmospheric-pressure dielectric barrier discharge system and (b) waveforms of applied voltage and current at input voltage of 120 V and air gas flow rate of 15 L/min.

an oscilloscope (WaveSurfer44MXs-B LeCroy). Optical emission spectroscopy (OES) was conducted to measure the plasma emission spectra using a spectrometer (SCT-320 Princeton Instruments, 600 g/mm) equipped with a charge coupled device (CCD, PIXIS400B Princeton Instrument).

### 2.3. Gas and microbiological analysis

FTIR (FT/IR-6300 JASCO) signals were obtained from 4000 to 400 $cm^{-1}$ to measure gas compositions in spectra intensities. The background spectrum was collected by maintaining resolution at 0.07 $cm^{-1}$. After the background scan, air gas and plasma treated air gas were fed to the sample holder and analyzed. Ozone concentrations in the Tedlar® bag were investigated by ozone sensors (UV-106M Ozone Solution and A-22 ECO SENSORS) with units of parts per million (ppm).

To confirm the storage improvement of grapes by plasma treatment, grapes were stored in a pouch filled with air and plasma treated air gas at room temperature. The three sample bags having 15 grapes from a bunch were prepared for the same experimental condition. The 3–5 g collected both from the pulp and skin of the grapes stored in a sample bag were transferred to a sterile conical tube and homogenized for 1 min using a vortex mixer (KMC-1300V Vision Scientific) with 1:10 distilled water. Experiments on microbiological analysis for one sample bag were carried out at 5 times in each condition and average value with its standard deviation were used in analysis results. Decimal serial dilutions were carried out with distilled water. A total of 1 ml of each diluted solution was inoculated on yeast and mold count plate Petrifilm™ (3M Microbiology, USA). The plates were incubated for 72 h at 25 °C. The