



Optical spectroscopy study for pulsed frequency powered atmospheric He plasma

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ABSTRACT

The atmospheric plasma has a great advantage over its vacuum counterpart for surface treatment such as cleaning and coating in its much less destructive nature. This nature makes the atmospheric plasma to function more like an ion carrier rather than a reaction hub for processes like ionization, dissociation, and excitation. The less destruction also preserves the integrity of molecular microstructures before deposition. Such a condition is particularly necessary for depositing macromolecules such as proteins, DNA/RNA in biomedical applications. In this study, we investigate the effects of the pulsed frequency of power and gas flow rates on the chemical compositions and morphology of atmospheric He plasma generated by a customized plasma system. Using an optical emission spectrometer, the spectra of He plasma in the air were quantitatively assessed for cases of different pulsed frequencies and He flow rates. In order to evaluate the capacity of He plasma, treatments on liquid (de-ionized water) and soft solid surface (spin-coated lactic acid films) were performed by direct contact. For the case of de-ionized water, the interaction between plasma and water is found to generate amount of OH radicals following the increase of pulsed frequency. For the case of spin-coated lactic acid films, the OH radicals generated by the plasma can cause the destruction of existing OH bonds in lactic acids to form water molecules during plasma treatment.

1. Introduction

Atmospheric plasma techniques developed rapidly in the past decade. High-performance atmospheric plasma harnesses the power of plasma for surface treatment such as cleaning and coating. Due to the nature of the operational environment, the atmospheric plasma functions less destructive to atoms or molecules than its vacuum counterpart in terms of ionization, excitation and dissociation. Such features imply many molecules or atoms uphold their original structures through the plasma zone, which is crucial if one would like to deliver these atoms or molecules directly onto the surface of a substrate by the plasma. The conservation of structures is particularly desirable for depositing macromolecules such as proteins, DNA/RNA in biomedical applications [1–4]. It is also useful in the task of surfaces activation and modification where the atmospheric plasma can be straightforwardly utilized for large-scale productions without complicate vacuum facilities. The roll-to-roll process for coating and etching metallic or polymeric surfaces is a typical example in aviation, marine, automotive and civil applications [5,6].

Besides the possible preservation of microstructures, atmospheric plasma also possesses some other important characters. For instance, it generates some short-lived reactive oxygen and nitrogen species (ROS/RNS), which provide the chance of various chemical reactions for anti-microbial or anti/pro-inflammatory, pro-apoptotic mechanisms [7–11]. Atmospheric plasma can be applied to in-vivo tests due to its low temperature [1]. On the front of plasma diagnosis, the optical emission spectrometers (OES) are ready to be deployed for the detection of radicals in plasma [12–15]. Perhaps the most importantly, most atmospheric plasma processes, the size of sample would not be limited by the dimension of the vacuum chamber.

In this study, we investigate the effects of the pulsed frequency of power and gas flow rates on the chemical compositions of atmospheric He plasma. A customized plasma system was set up and equipped with a pulsed frequency power supply, an optical spectrometer, x-y-z automated table, intensified charged coupled device camera, various flow controller, and pressure gauges. Our goal is to gain the insightful understanding of atmospheric He plasma via analyzing its optical spectra and further such knowledge to the control of He plasma for film

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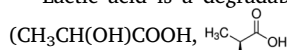
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deposition and surface treatments.

Since atmospheric plasma is mostly developed for in-situ processes where water is an inevitable contact body, the interactions with the aqueous environment by plasma are particularly important. To assess the interactions, we specifically conducted a contact treatment of de-ionized (DI) water by the He plasma. The recorded optical spectra near the water surface allow us to analyze possible reactions between the two entities and the capacity of our custom made plasma system.

Lactic acid is a degradable, organic compound with the formula



, CAS number: 79-33-4). In-room temperature, lactic acid is colorless liquid produced naturally. With a hydroxyl group adjacent to the carboxyl group, lactic acid is classified as an alpha-hydroxy acid (AHA). In the form of its conjugate base called lactate, lactate plays a role in many biochemical processes. For example, lactate made in muscle cells and red blood cells when the human body transforms hydrocarbons into energy. This transform is necessary when oxygen levels in tissues are low. Such circumstances are common particularly during an intensive exercise or having an infection or disease. The over-formation of lactate can cause a condition called “Lactic Acidosis” which can lead to some severe illness such as sepsis, heart attack or failure, severe lung disease or respiratory failure or even severe anemia [16,17]. In food industries, lactic acid is commonly used as a preservative in milk products and as a flavoring agent. In this study, we investigate the effects of treatment by atmospheric He plasma on lactic acid films. The effects were examined by possible changes in microstructures using Fourier-transform infrared spectroscopy (FTIR). One focus of the microstructural transitions in lactic acid is on OH radicals introduced by He plasma. Such change lactic acid could play an important role in the process of surgical septic applications.

2. Experiment

2.1. Setup of plasma system

The setup of the atmospheric plasma system is schematically shown in Fig. 1 where some details are described in the following. The power supply is variable pulsed frequency (RF) with the range of 5–80 kHz (You-Shang Technical Co., Taiwan). Both electrodes are enclosed in a quartz tube to avoid short-circuited or arching. The quartz tube also guarantees the plasma jet is securely ejected out at its tip. The optical spectrometer used in this study is AvaSpecULS2048L (Avantes, USA). It has optical range of 200–1100 nm with sensitivity in counts/μW per ms integration time 470,000. The integration time is 100 ms. This spectrometer is equipped with a 2048-pixel CCD linear array with onboard averaging sampling speed 1.1 ms/scan. During experiment, we retained all standard setups recommended by the manufacturer.

All gas flows are controlled by throttles and a flow meter. The purity of He used in this study is 99.999%. An intensified charge-coupled device camera (ICCD, DH-320 T, iStar, Andor Ltd., Belfast, UK) is also installed for the image capture on plasma jet.

2.2. Process parameter

Process parameters used for the test of atmospheric plasma are tabulated in Table 1. The system of plasma is operated at room temperature and the plasma carrier is solely He gas. Variable pulsed frequency (RF) is used to generate power under fixed voltage at 5500 V which is specified by the manufacturer limited by the current load 35,000 μA in the system.

The frequency of power is adjustable in our system and can be set up in the following way. First, the duty cycle ratio, which is the percentage of power-on time during one complete cycle, is fixed at 50%. In other words, the power-on and -off are equally split a single cycle. Then we varied the cycle time to be 200, 100, 40, 30 and 24 μs to obtain the

target frequencies 5, 10, 25, 33.3 and 41.7 kHz respectively. This is equivalent to say that the variation of power frequency is achieved by changing the periods of duty cycles.

2.3. Characterization

2.3.1. Plasma diagnostics and analysis

For the detection of excited species in the plasma, the optical spectrometer mentioned previously was employed. The data on emission from the spectrometer were analyzed afterward using the built-in software Avasoft® version 8.5.0.

In addition to the optical spectrometer, we also employed the ICCD camera to obtained images of particles of plasma ejected from the quartz tube. The images were recorded by the camera and processed by SOLIS® (Andor Ltd., Belfast, UK).

Numerical fitting by Gaussian curves for the spectra is implemented using the software Fityk 0.9.8.

2.3.2. Temperature measure

The temperature of atmospheric plasma is measured by Digital Thermo Tape® (Funakoshi Co., Ltd. Japan). This sample tape is electrically insulated and thus avoids arching when in contact with the plasma, which can be difficult to prevent if the thermal couple is used. The location of the measurement is kept at around 2.5 cm below the tip of the quartz tube where plasma ejected out.

2.4. Surface treatment

2.4.1. Interaction with water

Treatment of DI water for a short time (~2 min) is tested by employing 5 slm (standard liter/min) He plasma via a direct contact. This flow rate provides us strong enough spectra for analysis without excessively splashing and splurging water. As the plasma torch touches on the water surface, complex chemical reactions ensue immediately. To assess the effects of treatment, we followed the same approach to record the optical emission spectra near the water surface. The treatment yields stronger chemical reactions near the water surface and these reactions gradually diminish at the distance away from the surface. Therefore, as the location of measurements gradually moves away from the water surface, spectra weaken accordingly. Note that though different locations yield different intensities of peaks, the species of peaks are nevertheless almost identical to those in He plasma. A specific interest is on the peaks of OH radicals because they are noticeably produced by the interactions between plasma and water. Note that OH radicals are very active and short-lived (~10⁻⁹ s) [10,11], their existence is particularly important when the end application is toward biomedical purposes. For example, OH radicals can react with many volatile organic compounds (VOCs) by the removal of a hydrogen atom to form an alkyl radical (R·) as



Subsequently, the alkyl radical can rapidly combine with oxygen to become a peroxy radical as



The peroxy radical is an active chemical species and can be either a reductant or oxidant. The natural presence of peroxy radicals has a grand impact on the ozone and atmosphere [18–21].

2.4.2. Treatment on lactic acid films

(a) Preparation of Lactic Acid Film

Lactic acid films are prepared by spin coating (SWINCO SP-02, Power Assist Instrument Scientific Corp. Taoyuan, Taiwan) using lactic acid solution (L-(+)-Lactic acid, 88–92 wt%, 27,714 Sigma-Aldrich, US). Prior to coating, the glass substrate was treated by atmospheric He plasma (radio frequency 20 kHz, 5500 V) for a short time in order to

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