DEVELOPMENT OF LOW COST ATMOSPHERIC PRESSURE PLASMA JET FOR NANO- AND BIO- TECHNOLOGY APPLICATIONS

ELFA RIZAN BINTI RIZON

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I dedicated this thesis to my family for supporting me with a lot of love and affection in my journey to become a successful person

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ABSTRACT

The development of cold atmospheric plasma (CAP) can overcome many limitations of low pressure plasma. Low temperature plasma is widely used in the semiconductor and medical industry for cleaning purposes. However, the process is time consuming because low pressure plasma requires vacuum environment. This renders the system unable to sustain continuous processes. Cold atmospheric plasma opened up new possibilities providing the ability to expose plasma to liquous samples, three-dimensional samples and enabling in-situ treatment. In this study several plasma configurations were constructed. The configuration that ignited stable plasma was the atmospheric pressure plasma needle jet (APPNJ). During optical measurements, energetic particles, Ar atoms, and other elements within the ultraviolet (UV) range such as secondary nitrogen, oxygen, and hydrogen were detected. The optimum treatment time, 60 s was obtained from several materials surfaces treated by APPNJ. Treatment time of 60 s, successfully enhanced surface wettability of microscope slide glass from 51.07° to 4.46°, polypropylene from 99.70° to 35.43°, and fluorine doped tin oxide from 59.06° to 9.9°. The wettability of Aluminium (Al) thin film was enhanced from 72° to 0°. The topography studies indicated that treated Al thin film surface roughness reduced by 2.86 $X10^{-3}$ nm but the grain size was unaffected. The structural properties of the Al thin film showed no significant difference after plasma treatment, according to the full width at half maximum (FWHM) value of 111 orientations of Al thin films, both of which had the same value (0.3542 deg). The liquous sample of the *Escherichia coli* (*E. coli*) was exposed to direct (1.5 cm gap) and indirect (2.0 cm gap) plasma treatment. In the direct treatment, a total inactivation of *E. coli* was achieved after 100 s of plasma treatment. Indirect treatment of the same duration deactivated 54% of the bacteria. Overall, the purported abilities of CAP have been proved to be true and now await application in a variety of fields.

ABSTRAK

Perkembangan plasma atmosfera sejuk (CAP) dapat mengatasi banyak penghadan plasma tekanan rendah. Plasma suhu rendah digunakan secara meluas dalam industri semikonduktor dan perubatan untuk tujuan pembersihan. Walau bagaimanapun, proses itu memakan masa kerana plasma tekanan rendah memerlukan persekitaran vakum. Ini menjadikan sistem tidak dapat mennyediakan proses suapan selanjar. Plasma atmosfera sejuk membuka kemungkinan baru yang memberikan keupayaan untuk mendedahkan plasma kepada sampel cecair, sampel tiga dimensi dan membolehkan rawatan *in-situ*. Dalam kajian ini beberapa konfigurasi plasma telah dibina. Konfigurasi yang menyalakan plasma stabil ialah jet plasma jarum tekanan atmosfera (APPNJ). Semasa pengukuran optik, zarah bertenaga tinggi, atom Ar, dan unsur lain yang terletak dalam julat ultraviolet (UV) seperti nitrogen sekunder, oksigen, dan hydrogen telah dikesan. Masa rawatan optimum, 60 s diperolehi dari rawatan APPNJ kepada beberapa permukaan bahan. Masa rawat selama 60 s, kebolehbasahan permukaan permukaan kaca mikroskop berjaya dipertingkat dari 51.07° hingga 4.46°, polipropilena dari 99.70° hingga 35.43°, dan fluorin dop stanum oksida dari 59.06° hingga 9.9°. Kebolehbasahan filem tipis Aluminium (Al) ditingkatkan dari 72° hingga 0°. Kajian topografi menunjukkan bahawa kekasaran permukaan filem tipis Al yang dirawat berkurangan sebanyak 2.86 $X10^{-3}$ nm tetapi tidak memberi kesan kepada saiz bijian. Sifat struktur filem tipis Al tidak menunjukkan perbezaan selepas rawatan plasma, mengikut nilai lebar lengkap separa maksimum (FWHM) filem tipis Al orientasi 111, kedua-duanya mempunyai nilai yang sama (0.3542 deg). Sampel cecair *Escherichia coli (E. coli)* terdedah kepada rawatan plasma secara langsung (jarak 1.5 cm) dan tidak langsung (jarak 2.0 cm). Dalam rawatan langsung, penyahaktifan menyeluruh *E. coli* telah dicapai selepas rawatan plasma 100 s. Pada tempoh yang sama rawatan tidak langsung bakteria dinyahaktifkan 54 %. Secara keseluruhan, keberangkalian kebolehan CAP telah terbukti benar dan kini menanti aplikasinya dalam pelbagai bidang.

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LIST OF SYMBOLS AND ABBREVIATIONS

µL	-	Microlitre
µs	-	Microsecond
°C	-	Degree celcius
a.u.	-	Arbitary unit
AC	-	Accelerating current
ACD	-	Allergic contact dermatitis
AFM	-	Atomic force microscope
Al	-	Aluminium
AP-PECVD	-	Atmospheric pressure plasma enhanced chemical vapour deposition
APPJ	-	Atmospheric pressure plasma jet
APPNJ	-	Atmospheric pressure plasma needle Jet
Ar	-	Argon gas
CAP	-	Cold atmospheric plasma
CFU	-	Colony forming units
CFU/g	-	Colony forming units per gram
CFU/ml	-	Colony forming units per mililitre
cm	-	Centimeter
cm^{-3}	-	Electron density
Co^{60}	-	Cobalt-60
Cs^{137}	-	Cesium-137
Cu	-	Copper
CVD	-	Chemical vapour deposition
DBD	-	Dielectric barrier discharge
DHCP	-	Dental healthcare personnel
DI	-	Dionized water
DNA	-	Deoxyribonucleic acid

E. coli	-	Escherichia bacteria
EtO	-	Ethylene oxide
eV	-	Electron volt
FTO	-	Fluorine-doped-oxide
H_2O	-	Water vapor
He	-	Helium
Hz	-	Hertz
IR	-	Infrared
K	-	Kelvin
keV	-	Kilo electron volt
kHz	-	Kilohertz
kV	-	Kilo volt
kVp-p	-	Kilo volt peak to peak
L/min	-	Litre per minutes
LP-PECVD	-	Low pressure plasma enhanced chemical deposition
LTE	-	Local-thermal equilibrium
mA	-	Milli ampere
mg	-	Milligram
Min	-	Minutes
MLP	-	Multilayer perceptron
mm	-	Millimetre
MPT	-	Microwave plasma torch
MW	-	Microwave
N_2	-	Secondary nitrogen
nm	-	Nanometre
Non-LTE	-	Non local-thermal equilibrium
O	-	Oxygen atom
O_2	-	Oxygen
OES	-	Optical emission spectroscope
OH	-	Hydroxide
PET	-	Polyethylene terephthalate
PP	-	Polypropylene
PVD	-	Physical vapour deposition
RF	-	Radio frequency

ROS	-	Reactive oxygen species
Rq	-	Root mean square roughness
Si	-	Silicon
SPLNN	-	Single layer perceptron neural network
T_e	-	Electron temperature
T_g	-	Gas temperature
T_h	-	Heavy particle temperature
T_i	-	Ion temperature
Torr	-	Gas pressure
UV	-	Ultraviolet
V	-	Voltage
VA	-	Volts-amps
Vac	-	Voltage accelarating current
Vin	-	Input voltage
W	-	Watt
WCA	-	Water contact angle
XRD	-	X-ray diffraction

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CHAPTER 1

INTRODUCTION

Plasma can be regarded as the fourth state of matter after solid, liquid and gas, accounting for about 99.99% of matter present in the universe [1]. It can be categorized into natural plasma and artificial plasma [2]. Examples of natural plasma include stars, nebulae, interstellar particles, the sun and many more. Meanwhile, artificial plasma can be created by giving gas enough external energy for its atoms to ionize, thus leading to an increase in temperature (that generally exceeds 10^5 K). The temperature of the plasma can be lower than 10^5 K if a mechanism for ionizing gas to be low in recombination and mass density exists [1]. Plasma technology is widely utilized in our daily life in items such as the fluorescent bulb, plasma television, cell phone, arc welding and more. Although plasma is a highly contained charged particle and strongly affected by electric and magnetic field, it is electrically neutral from macroscopic point of view [3,4].

1.1 An overview

The proposed cold atmospheric plasma (CAP) is one of the low temperature plasma that is highly effective because it contains ultraviolet light, energetic ions, and radicals during the process that can modify surface adhesion and remove microorganisms [5,6]. Previous work showed that plasma in high-pressure environment enhances the number of energetic atoms and particles [7]. However previous studies show that the efficiency and performance of the CAP is a crucial part to be determined because it is relies on the power supply, configuration and type of working gas of the designated atmospheric pressure plasma [8,9]. Hence, this study proposes a comprehensive study of the in-house low cost CAP. In order to obtain the CAP performance, several treatment process on selected material surfaces and mediums have been conducted to have an effect in nano- and bio-technology. This was because CAP has a unique emphasis on the mechanism of surface activation and bacteria biofilms [10,11].

1.2 Problem statements

The emergence of plasma technology has become a topic of great interest. Its wide applications in material processing, surface cleaning, and etching have been implemented in small to huge scale processes [12]. In the earlier years, the manipulation of plasma was obtained in a high-end vacuum chamber due to easy generation of stable plasma in a low pressure environment [13]. The ultimate inspiration to move from low pressure plasma to CAP was to overcome the limitations of low pressure plasma such as the inability to provide a continuous process, and the inability to provide crucial exposure space, especially three-dimensional objects [3]. Plasma generation in an atmospheric environment introduced several challenges. Plasma had to be in the state between glow to arc transition where the plasma could easily be turned to arc transition. The working gas in arc transition plasma can reach temperatures up to several thousands of Kelvin (K) when the electrons achieve thermal equlibrium with the heavy particles ($T_e = T_{h,}$; T_e = electron temperature; T_h = heavy particle temperature or sensible temperature) [14]. Plasma in this condition is not favorable as it can shrink the possibility of plasma exposure in low temperature application [3].

Nowadays, a lot of effort has been put into obtaining plasma in an atmospheric environment. Several types of high pressure plasma such as glow discharge, corona discharge, dielectric barrier discharge, plasma jet and microwave plasma have proposed [15–17]. The instability of the plasma generated in the atmospheric pressure plasma have caused variations in plasma discharge. In addition, the type of power supply also affects the performance of the atmospheric pressure plasma discharge [18]. A thorough of investigation is needed to fully understand the mechanism of each type of plasma in regards to its application.Hence, this study is embarked on deeper understanding of a low cost atmospheric pressure plasma, and the discovery of methods that can enhances the generations of plasma in atmospheric environment.

1.3 Objectives of the study

This study embarks on the following main objectives:

(i) To design and develop the CAP.

(ii) To evaluate the distribution of energetic atoms, ions and particles in CAP system using optical emission spectroscopy (OES).

(iii) To determine the performance of CAP system for nano- and bio- technology applications.

1.4 Scopes of the study

The following constraints are the scopes for this project for successful completion:

(i) Design and develop the low cost cold atmospheric pressure discharge system.

(ii) Argon is used as working gas for CAP applications.

(iii) Measurement of plasma temperature using infrared (IR) thermometer.

(iv) The surface modification of microscope slide glass, polypropylene (commercial food container) and fluorine doped tin oxide (FTO) thin film with treatment time of 5 s, 10 s, 20 s, 40 s and 60 s.

(v) The thin film deposition of 30 mg Aluminium (Al) 99.97% (industrial grade) on silicon substrates by using a thermal evaporator.

(vi) The wettability testing and aging effect of Al thin film after plasma treatment for 5 s and 60 s for up to 3 days.

(vii) The characterization of optimum treatment time of 60 s by evaluating the water contact angle, x-ray diffraction (XRD) and atomic force microscope (AFM) of Al thin film.

(viii) The microbial *E. coli* isolated from wastewater (UTHM sewage).

(ix) The exposure time required for *E. coli* inactivation at 5 s, 10 s, 20 s, 40 s, 60 s, 80 s and 100 s.

1.5 Outline of the thesis

This thesis contains five chapters. The first chapter provides the overview, objectives, and the limits of the project. The second chapter shows literature on previous efforts that have contributed to this project and its potential applications. Next, in the third chapter, the synchronization and flow of this project will be discussed in detail. The fourth chapter will present the result and discussion of the study in CAP development. Last but not least, chapter five houses the summary of the outcome, the conclusion, and proposals for future work.

CHAPTER 2

LITERATURE REVIEW

The production of plasma in an atmospheric environment has been highly sought since discovering of low pressure plasma by Irving Langmuir (1881-1957) [19]. The advantages of CAP greatly benefits many industries such as those related to raw food product, biomedical, and engineering production [20,21] This is because CAP has a simple operating set-up, can operate in an atmospheric environment, and operated at low temperatures. However, a deeper understanding of CAP fundamentals is needed to develop a complete CAP system that can be transferred from research into market technology. This study is an effort towards this direction.

2.1 Plasma physics

The term 'Plasma' was introduced by American chemist, Irving Langmuir in 1927. During his studies on electric discharge and their fluid characteristics at General Electric Research and Development Centre, he found an ionized gas that could be manipulated by a magnetic field [19]. The most well-known natural plasma in the earth's atmosphere is lightning [22]. Besides natural plasma, plasma also can be generated. Both natural and man-made plasma occur at differing in temperatures, electron densities, and pressures. Plasma as the fourth state of matter is composed of ionized gases as shown in Figure 2.1. Through increasing energy by means of heating, matter can change its state from solid to liquid then to a gaseous state. From there, higher energy levels can be obtained by applying and increasing external energy, whereby the gas molecules separate and form freely moving charged particles; electrons and ions form a quasi-neutral particle whereby the plasma is electrically conductive, and the density of positive and negative charges are in a balanced state [23].

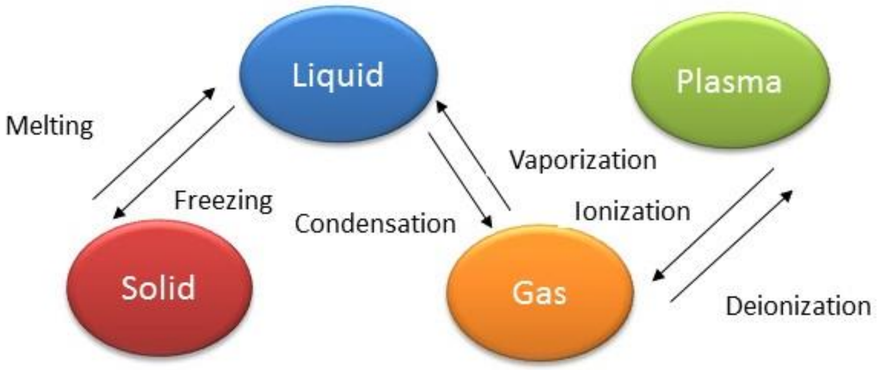

Figure 2.1: Solid state of matter [24]

The accelerated and collision of electrons with other bodies will provide further excitation, dissociation, and process reactions that lead to the multicomponent nature of plasma such as electrons, ions, excited molecules, and neutrals as shown in Figure 2.2. The plasma gas temperature ranges from room temperature to solar temperature. The electron temperature ranges from 1 eV – 20 keV, while electron densities range from 10^6 – 10^{18} cm^{-3} [25].

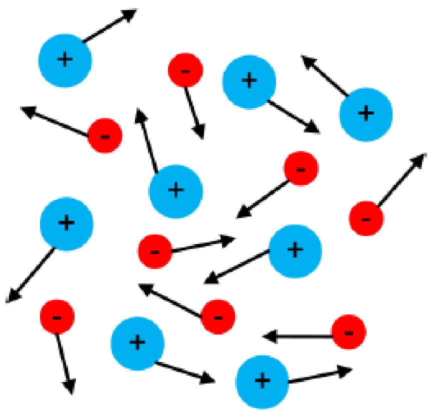

Figure 2.2: Quasi-neutral state of plasma [23]

Plasma can be classified into high-temperature plasma and low-temperature plasma [26]. Sometimes, these are also referred to local-thermal equilibrium (LTE) and non-LTE plasmas. The highly energetic electrons have much higher temperatures than heavy particles such as ions and atoms that have a relatively low

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