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Effect of Plasma Treatment on the Surface of Polyethylene Terephthalate with 50Hz Dielectric Barrier Discharge at Near-Atmospheric Pressure

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***Abstract*-- Exiguous reduction in pressure inside a normal Dielectric Barrier Discharge (DBD) reactor makes the discharge between the electrodes more uniform, allowing it to be preferably suited for material processing. The pressure of a DBD reactor was reduced to few orders of magnitude and the samples of polyethylene terepathalate (PET) were treated for varying time. Surface characterization of those samples were done using standard characterization techniques like contact angle measurement, Fourier Transform Infrared Spectroscopy (FTIR) and Optical Emission Spectroscopy (OES) followed by computation of polar and dispersive components of surface free energy. The outcomes of the experiments proved that the modification of surface properties via plasma treatment reaches to its saturation point after certain treatment time reducing the necessity of further treatment.**

***Keywords*- PET, DBD, Contact Angle, Surface Free Energy, OES, FTIR**

I. INTRODUCTION

Low-temperature plasmas; due to their non-thermal and non-equilibrium properties [1]; are particularly suited for textile processing [2] because most textile materials are heat sensitive. Other applications of cold plasma include food processing and treatment, water treatment, nonmaterial synthesis, medicine and many more [3, 4]. Low-temperature plasma treatments are either carried out at low pressure or at atmospheric pressure [5, 6]. While the low pressure plasma has become popular in the treatments of polymeric materials, the atmospheric pressure plasma has begun to grow as a good technique for textile processing [7]. Here we discuss about the near atmospheric plasma in which the pressure inside the reactor is slightly reduced and the discharge is produced [8]. Thus produced plasma is more uniform in comparison to the plasma produced at atmospheric pressure. Surface treatment with such uniform plasma is more reliable and consistent as it reaches homogenously over the surface being treated [9].

Industrial plasma treatments involve continuous and rapid treatment surfaces of huge quantity of samples every day. Reduction of treatment time of such individual samples by few milliseconds can finally result out to the reduction of treatment time of thousands of samples by few minutes. Determining optimal treatment time of polymer surface is therefore very important for industries, which is the primary objective of this research.

II. EXPERIMENTAL

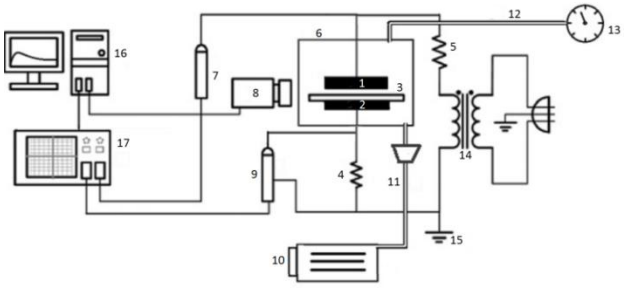

Figure 1: Experimental Setup

1. Circular upper electrode 2. Ground electrode
3. Dielectric sheet 4. Ballast Resistor
5. Resistor 6. Reaction Chamber
7. 10-times voltage Probe 8. ICCD Camera
9. Voltage Probe 10. Vacuum Pump
11& 12. Pipe 13. Pressure Gauge
14. High Volt. Transformer 15. Ground
16. Computer Interfacing 17. Oscilloscope

The schematic diagram of the experimental setup used in this research is as shown as in figure 1. Two copper electrodes are separated by a distance of 3.5mm and a gas plate of thickness 1mm is placed above the lower electrode. The electrode system is covered by a cylindrical polycarbonate chamber connected to a vacuum pump and an analogue pressure gauge. The working voltage of the experiment is set to 600V. The voltage across the electrodes is measured by a high voltage probe (PINTEK HVP-28HF) connected to a digital oscilloscope (Tektronix TDS2000) which then feds the data to a computer. A tiny circular hole is drilled through the chamber into which an optical fiber is inserted in order to avoid the loss of spectral information due to the light of some wavelength being absorbed by the polycarbonate chamber.

The sample of PET is placed over the dielectric between the electrodes thereafter, the vacuum pump is turned on and the pressure inside the chamber is reduced to about 40 torr. The power source is then switched on so that the discharge is produced between the electrodes. The treated sample taken for surface characterization which includes the measurement of contact angle using a contact angle Goniometer (Rame-hart Model 200) and FTIR analysis.

III. RESULTS AND DISCUSSION

A. Contact Angle Measurements

The static contact angle measurements are made before the treatment and immediately after the treatment by dropping $4\mu l$ of distilled water (H_2O) and glycerol ($C_3H_8O_3$) on the surface of the PET samples by using contact goniometer. Surface free energy (γ) was calculated in terms of polar (γ_p) and dispersive (γ_d) components with the use of Fowkes equation [10]. The addition of these two components gives the total surface free energy of the solid. The surface free energy is calculated from the Harmonic mean method using the contact angle [11].

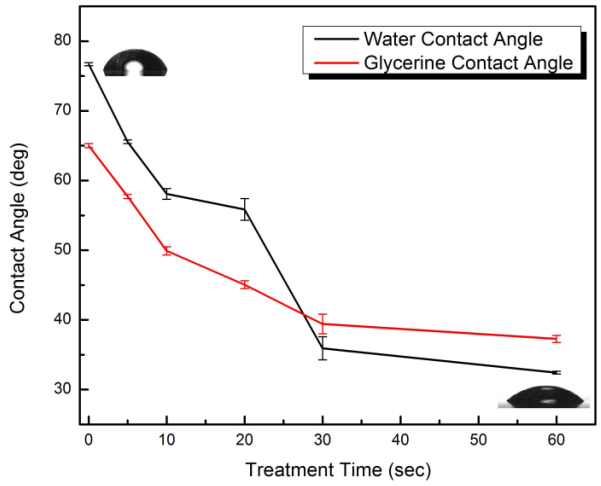

Fig. 2 Contact angle of PET surface as a function of treatment time for 50 Hz DBD at near-atmospheric pressure.

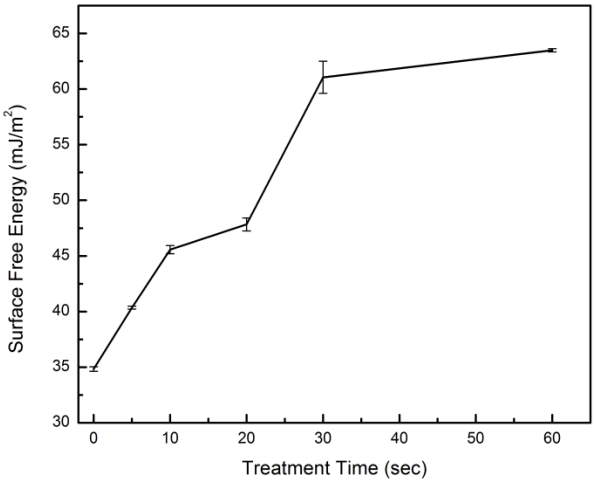

Fig. 3. Surface free energy ($\boldsymbol{\gamma}$) of PET surface as a function of treatment time for 50 Hz DBD at near-atmospheric pressure.

Figure 2 and 3 shows the variation of water contact angle and surface free energy of PET surface as the function of treatment time respectively. It is seen that on increasing treatment time there is a decrement in the contact angle which shows a strong increase in wettability [12] on the PET surface. The increase in surface free energy is due to the functionalization of the polymer surface with hydrophilic groups. It is found that the further treatment of PET does not lead to any significant changes in the contact angle and surface free energy. Figure 3 shows the

variation of ratio of polar and dispersive components of surface free energy as a function of treatment time. The increment in the value of the ratio signifies that there is a dominance of chemical change with respect to physical change on the surface of polymer sample. So, more treatment of sample is needed to get more chemical change on the surface of the PET polymer.

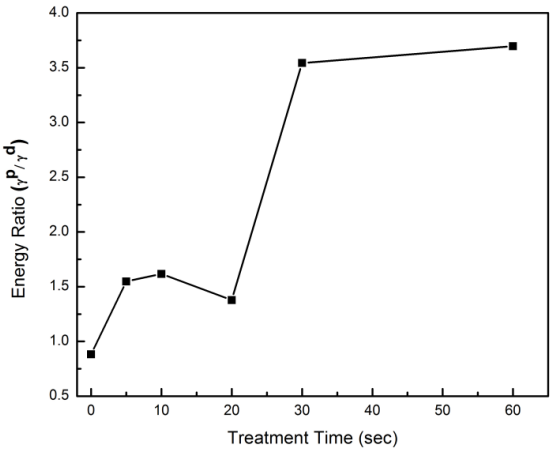

Fig. 4 Energy ratio as a function of treatment time for 50Hz DBD at near-atmospheric

B. Electron density by Stark Broadening Method

The value of electron density (n_e) has been calculated by using Stark broadening method [13] which arises from the interaction of atoms with the charged particles. The profile becomes symmetric and can be approximated by Lorentzian profile when the electron dominates. The Lorentzian plot is shown in the figure 5. The full-width half-maximum (FWHM) of stark broadening can be calculated using the formula.

$$\Delta\lambda_{stark} = 2 \times 10^{-11}(n_e)^{2/3}$$

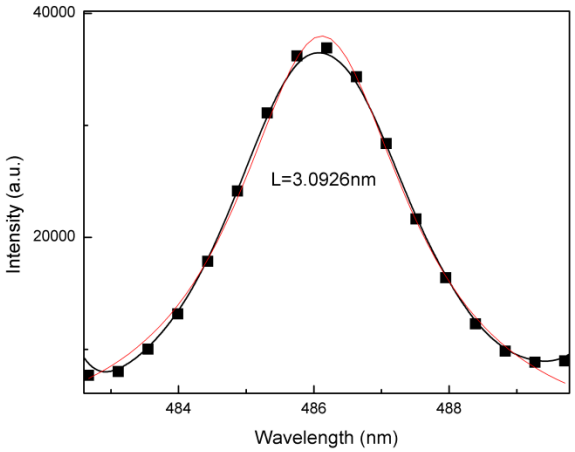

Fig. 5 Recorded line profile for $\Delta\lambda$=3.0926nm and its Lorentzian fit

For $\Delta\lambda$= 3:0926nm the shape of broadening line is Lorentzian and the calculated value of electron density (n_e) is $6.08\times10^{16}cm^{-3}$.

C. FTIR Analysis

The sample of PET treated at near-atmospheric condition for 5 sec and 5 min time duration along with the untreated sample produces the graph as shown in Figure 6. It is seen that after plasma treatment the less stable functional group like C=O, X=C=Y, C=X begins to disappear from the surface of the sample and only the ones with strong bonding like C=C remains on the surface.

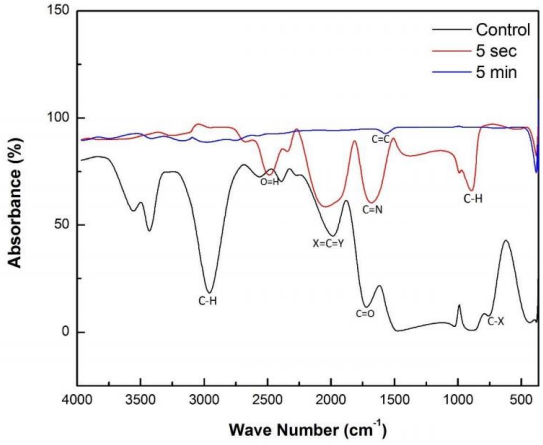

Fig. 6 PET treated at near-atmospheric condition along with the untreated sample

IV. CONCLUSIONS

The results of this research showed that a relatively homogeneous discharge can be achieved at near atmospheric pressure DBD. Samples of PET were treated for various intervals and it was seen that the surface properties of the sample were affected for certain time and ultimately gets saturated. FITR analysis shows that the plasma treatment causes the disappearance of less stable functional groups. Electron density of the near-atmospheric plasma was calculated using Stark broadening method. It was also seen that the change in surface properties of the treated sample reached its saturation point, elimination the requirement of further treatment.

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