

Research Article

Studies on Modification of Surface Properties in Polycarbonate (PC) Film Induced by DC Glow Discharge Plasma

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The polycarbonate film (PC) surface was treated using glow discharge low-pressure air plasma. The modified surface was characterized by contact angle, FTIR, XRD, AFM, and XPS analysis. The surface-modified samples were further investigated using T-peel test for technical applications. The surface energy of the sample was estimated by measuring contact angle. The results show that, after plasma treatment, the root mean square (RMS) roughness of PC film was gradually increased with exposure time. Plasma treatment modified the chemical composition of the polymer surface and it made the surface to be highly hydrophilic. It was found that the air plasma treatment increases the polar component of PC film.

1. Introduction

Polymers are generally macromolecules formed by the repeated linking of large number of small molecules. Polymers are widely used in automobile, defense, electrical, and computer components and so forth [1]. Nowadays organic polymers are usually replaced by traditional engineering material such as metals, glasses, and ceramics because of their good characteristic features in physical and chemical aspects [2]. The low-surface energy of polycarbonate results in poor adhesion of additional coatings, which leads the manufacturers to face more technical challenges [3]. Because of their superior performance and low-cost the polymer-based materials are used in many areas. To modify polymer surfaces low temperature, low-pressure plasma is commonly used [4]. In modern days, various methodologies were developed to convert polymer surface for enhanced wettability, adhesion, and so forth, [5]. To modify the surface properties of polymers, low-pressure cold plasma treatment is commonly used as a dry process. The effect of plasma treatment depends on internal and external parameters like type of plasma (DC, RF, or microwave), the discharge power density, pressure, and

flow rate of the gas or gas mixture and exposure time [6]. The surface of the polymer is activated during plasma treatment, which brings about the chain session of the existing groups on the surface of the polymer and creates new functional groups such as $-OH$ and $-OOH$ [7, 8]. Air plasma is used to increase polar functional groups (hydroxyl, carboxyl, ether, carbonyl, etc.) which can successfully increase the surface free energy of the polymer [9]. In the recent years the surface treatment of PC was performed by many researchers to make suitable polymer surfaces for adhesion [10]. In this work PC films were treated with DC glow discharge air plasma under different exposure times with an intension of improving the intrinsic low-surface properties. The difference in hydrophilicity of plasma-modified PC film was characterized by measuring the contact angle as a function of treatment time. The surface morphology of the modified PC film was analyzed using atomic force microscopy (AFM). The change in crystallinity of the plasma-modified PC film surface was investigated by the X-ray diffraction (XRD) analysis. The chemical bonding on the surface of the film and functional groups were found using XPS analysis. The surface adhesion properties of surface-treated PC were widely studied

TABLE 1: Operating parameters for plasma processing.

Discharge potential	—	400 V
Discharge power (input)	—	10 W
Pressure	—	0.2 mbar
Exposure time	—	1–25 min
Electrode separation	—	3 cm
Plasma gas	—	Air
Samples	—	Polycarbonate film

using T-peel strength measurement for technical applications.

2. Experimental Setup and Methodology

Polycarbonate films of $160\ \mu\text{m}$ thickness were cut into $5\ \text{cm} \times 5\ \text{cm}$ sections for plasma treatment. PC films were ultrasonically washed in acetone and with distilled water for 15 minutes and then dried before plasma treatment. DC glow discharge plasma of low pressure was generated in a glass chamber of 29 cm length and 10 cm internal diameter size. Vacuum of 10^{-3} mbar was maintained inside the chamber using a vacuum pump. Required vacuum was maintained using fine control gas needle valve. Pirani gauge was used for pressure measurement. Circular-shaped electrodes made of aluminum with a diameter of 5 cm were fixed inside the chamber. The electrodes were separated by a distance of 3 cm. Air was used as the reactive gas. High-tension dc power supply of 1.5 KV was used. The PC films were placed perpendicular to the discharge axis between the parallel electrodes using a holder. The discharge potential and base pressure were 400 V and 0.2 mbar, respectively. The operating parameters are listed in Table 1.

The weight of the sample was measured before and after the plasma treatment, to estimate etching effects on the surface layers of PC films using a microbalance and the loss noticed was plotted against exposure time. The plasma-etching effect expressed by weight loss was calculated using the following expression [11]:

$$\text{Weight loss (\%)} = \left[\frac{(W_{\text{ut}} - W_{\text{pt}})}{W_{\text{ut}}} \right] \times 100, \quad (1)$$

where W_{ut} and W_{pt} are the weight of untreated and plasma-treated samples, respectively.

The contact angle is defined as the angle between a solid-surface and tangent of a liquid-vapour interface of a liquid drop. The hydrophilicity of a solid-surface is usually expressed in terms of wettability that can be estimated by contact angle measurements. It is a simple and convenient method to determine the surface wettability. Contact angles are influenced by interfacial tension, roughness, and molecular orientation in the polymer material.

The angle of contact was measured using sessile drop method and surface energy was estimated. The liquids water and glycerol with known γ^p (polar component) and γ^d (dispersive component) were used for calculating the surface energy of PC film. The height (h) and radius (r) of the liquids

TABLE 2: Surface energy of liquids.

Liquid	γ_1 (mJ/m ²)	γ_1^d (mJ/m ²)	γ_1^p (mJ/m ²)
Distilled water	72.8	21.8	51.0
Glycerol	64.0	34.0	30.0

were measured by using microscope and the contact angle was calculated using the following equation [12]:

$$\text{Contact angle } (D) = \frac{\sin^{-1}(2rh)}{(r^2 + h^2)}. \quad (2)$$

Three readings were taken at different places of the sample surface and an average was determined. The error in the measurement of contact angle was found to be $\pm 2^\circ$. Similarly, the contact angle measurements were carried out with respect to glycerol. The values of polar and dispersive components of testing liquids are given in Table 2.

The polar and dispersive components of the surface energy of the polymer film surface were calculated using the Fowkes approximation [13]

$$\gamma_1(1 + \cos \theta) = 2(\gamma_1^d \gamma_s^d)^{1/2} + 2(\gamma_1^p \gamma_s^p)^{1/2}, \quad (3)$$

where θ is the contact angle of testing liquids, γ_1 is the liquid surface tension, and γ_1^p and γ_1^d are the polar and dispersive components of the test liquids. Similarly, the solid-surface tension (γ_s) is expressed in terms of its polar and dispersive components

$$\gamma_s = \gamma_s^p + \gamma_s^d. \quad (4)$$

The adhesion work W_{adh} , a quantity related to the surface wettability, was estimated using the relation

$$W_{\text{adh}} = \gamma_1(1 + \cos \theta). \quad (5)$$

The surface polarity (P) of the plasma-treated polymer films was estimated using the expression

$$P = \frac{\gamma_s^p}{(\gamma_s^p + \gamma_s^d)}, \quad (6)$$

where γ_s (mJ/m²) is the total surface energy of the polymer film, and γ_s^p and γ_s^d (mJ/m²) are the polar and dispersion components of surface energy of the polymer film [14].

FTIR spectra were taken for untreated and plasma-treated PC samples to analyze the variation in absorption bands. The crystalline structure of the polymer films was investigated using XRD. The polymer crystallinity was evaluated from the ratio of the diffraction peak areas before and after the plasma treatment.

The surface morphology of PC films was analyzed by atomic force microscopy (AFM). The difference in root mean square (RMS) of the vertical Z-axis value, within the area of observation, was noted as the change in surface roughness

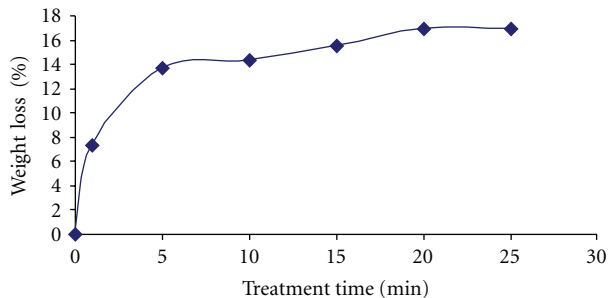


FIGURE 1: Treatment of air plasma results in loss of weight of PC film.

of the plasma-treated polycarbonate film. The RMS can be calculated using the following equation [15]:

$$\text{RMS}_{xy} = \left[\sum_{xy=1}^N \frac{\left((Z_{xy} - z_{\text{average}})^2 \right)}{(N^2)} \right]^{1/2}, \quad (7)$$

where Z_{average} is the average Z-axis value within the observed area, Z_{xy} is the local Z-axis, value and N indicates the number of points observed. Every surface-roughness value was calculated as the average of minimum 10 measurements, in the different areas of observation on PC film surfaces.

XPS spectra for untreated and plasma-treated PC films were taken to estimate the variation in surface elemental composition [16].

To study the effect of plasma treatment on adhesion, that is, to understand the effect of hydrophilic groups on bonding strength, a standard T-peel test was carried out using CRE machine at a rate of 100 mm/min at room temperature. For the test a transparent adhesive tape of 5 cm width was pasted over a length of 17 cm on the PC film. T-peel test was carried out after fixing one end of the sample in one jaw and the adhesive tape with a piece of paper adhered to it in another jaw. The bond strengths were reported as force of peel per unit length of sample width [17, 18].

3. Result and Discussion

3.1. Weight Loss Study. The interaction of ions, electrons, and energetic species of neutral atoms with the surface of polymer films causes rapid removal of low-molecular contaminants such as additives, processing acids, and adsorbed species. The process is called plasma-etching. After plasma-etching, ablation of polymer chain starts. This is either due to the physical removal of molecules or fragments or the breaking up of bonds, chain scission, and degradation processes [19]. This causes loss in the weight of the film. Figure 1 shows that the treatment of air plasma results in loss of weight of PC film, which increases with treatment time. The amorphous region of the PC surface undergoes more etching process than the crystalline region [20–22]. Plasma treatment removes etchable amorphous material from the top layer of PC surface retaining the bulk properties which reduces the etching rate.

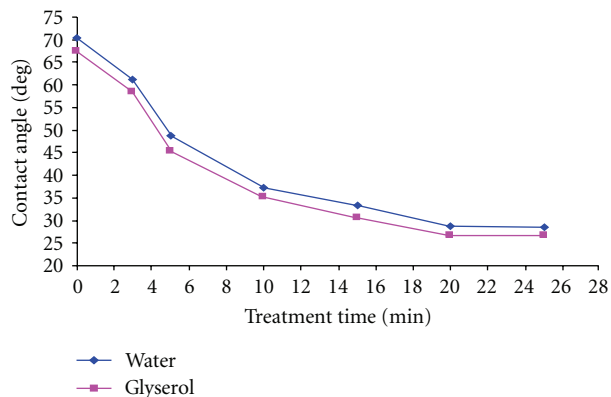


FIGURE 2: Variation of contact angle with respect to treatment time.

3.2. Contact Angle, Surface Energy, and Work of Adhesion. The variation in contact angle of PC films for different treatment times and for different test liquids is shown in Figure 2. It shows that the contact angle on the untreated surface is 70.3° and 67.3° for distilled water and glycerol, respectively. The contact angle values considerably reduced after the plasma treatment even for short exposure times (3 min) shifting them to lower values 61.2° and 58.5° for water and glycerol, respectively. The contact angle values do not change significantly with longer exposure times although slightly smaller values are observed for exposure times in the 25-minute range.

The PC film surface becomes more hydrophilic due to plasma treatment and the decrease in contact angle is relative to the rate of chemical changes taking place.

The adsorption characteristics of PC surface depend on the adhesion work. It controls all the physical interfacial changes happening on the polymer surface. The work adhesion (W_{adh}) and polarity were calculated using (5) and (6). It was observed that work adhesion W_{adh} and polarity of the untreated PC films were 97.3 mJ/m² and 0.702. After 25-minutes of plasma treatment the values increased to 136.72 mJ/m² and 0.803, respectively.

Figure 3 shows a plot of surface energy γ_s from the measured contact angles on the PC surface as a function of exposure time. Plasma treatment appears to increase the surface energy upto 25 minutes exposure time. Similar trend was observed for the polar component (γ_s^p), it was mainly due to the incorporation of polar groups such as CO, COO, and OH [23, 24]. The properties such as wettability, adhesion strongly depend upon the surface energy.

3.3. FTIR Analysis. FTIR spectra were taken for PC samples both untreated and plasma-treated as shown in Figure 4. The characteristic absorption bands as observed from the untreated sample were identified as (A) C–O stretching vibration, (B) C=O stretching vibration, and (C) CH₃ stretching vibration. The absorption bands C–O and C=O increase due to plasma treatment which also create unsaturated –C=C– bonds.

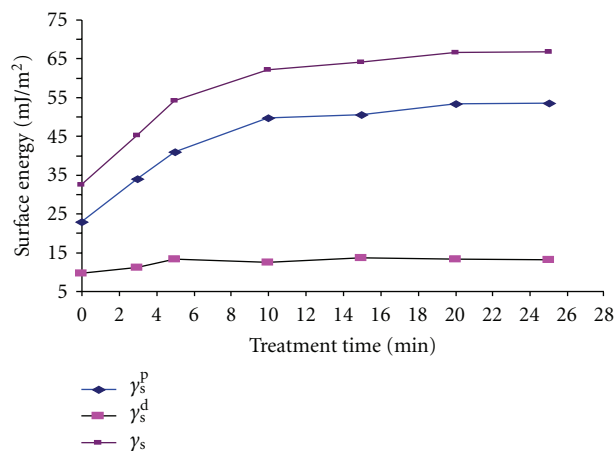


FIGURE 3: Surface energy of PC film with respect to treatment time.

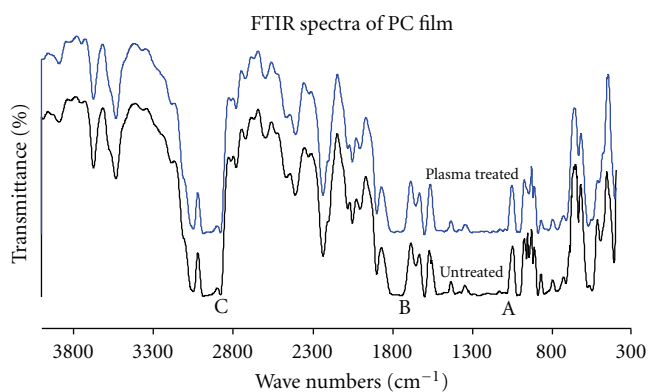


FIGURE 4: FTIR spectra of untreated and treated PC film.

3.4. XRD Results. XRD spectra were used for analyzing the crystalline structure of PC film. The ratio of diffraction peak areas before and after plasma treatment was used for the analysis of crystal structure. The peak was more intense for the treated film due to the increase in crystallinity. There were no major changes in the shape or position of the diffraction peaks after plasma treatment (Figure 5 and Table 3).

3.5. Surface Analysis: AFM Results. The surface modification as well as the change in morphology of the PC film was measured by AFM analysis. Figure 6 shows the AFM images of the untreated PC film and treated film exposed to air plasma for different time duration. Figure 6(a) shows the surface of the untreated PC film which is comparatively smooth. Figures 6(b), 6(c), and 6(d) show that the surface roughness of the PC film increased after plasma treatment which indicates the improvement in wettability and bonding strength.

It was observed that the values of root mean square (RMS) roughness increase when treatment time increases as shown in Figure 7. The plasma treatment removes few monolayers from the surface of polymer film increasing the roughness which in turn improves wettability and bonding strength. The adhesion depends strongly on the type of polar

TABLE 3

Pos. [$^{\circ}2\theta$.]	Height [cts]	FWHM [$^{\circ}2\theta$.]	D-spacing [\AA]	Rel. Int. [%]
Before plasma treatment				
17.0848	162.40	4.0000	5.18576	100.00
After plasma treatment				
17.0320	561.29	4.0000	5.20171	100.00

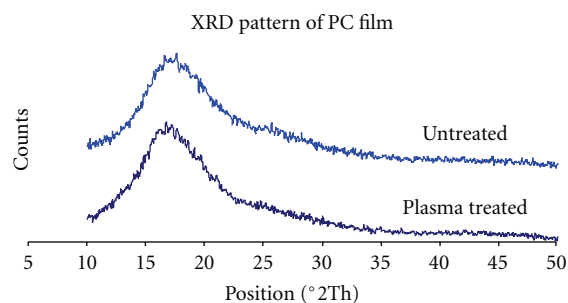


FIGURE 5: XRD spectrum of untreated and plasma-treated PC films.

functional groups attached to the plasma-modified PC surface.

3.6. XPS Analysis. The XPS spectrum of the plasma-treated and untreated PC film is shown in Figures 8(a) and 8(b). It was observed that C1s and O1s were dominant components. After plasma treatment, the intensity of O1s peak increases and C1s peak decreases in the surface of the PC film. The inclusion of the oxygen-containing polar groups onto the polymer surface may be the reason for the increase in hydrophilic property. Since the PC film was treated in air plasma, some nitrogen-containing group was added onto the surface. For the untreated PC film, there is no peak due to N1s but for the plasma-treated, there is a small peak. The atomic concentration of the N1 element increases.

Increase in oxygen content and decrease in carbon concentration were observed on the PC surface. Figures 9(a) and 9(b) show the C1s and O1s peaks, respectively, of the high resolution XPS spectra of the untreated PC sample. The amounts of chemical bonds present in the film were in proportion to the area of individual peaks. Figures 10(a) and 10(b) show the C1s and O1s peaks of polycarbonate sample treated with air plasma.

The C1s are represented by three Gaussian functions which correspond to the different bonding states 284.5 eV aromatic C–H, 285.0 eV aliphatic C–H, C–C, and 286.24 eV aromatic C–O correlated in Figure 9(a). The O1s were separated in two components corresponding to the binding energy 531.72 eV C=O and 533.01 eV C–O as shown in Figure 9(b).

The plasma-treated PC films showed additional C1s peaks at 287.54 eV, 288.24 eV which may be due to C=O/O–C–O, O–C=O, and O–CO–C groups, respectively [25]. After plasma treatment, the C–C groups decrease, C–O and additional oxygen-containing functional group increases with

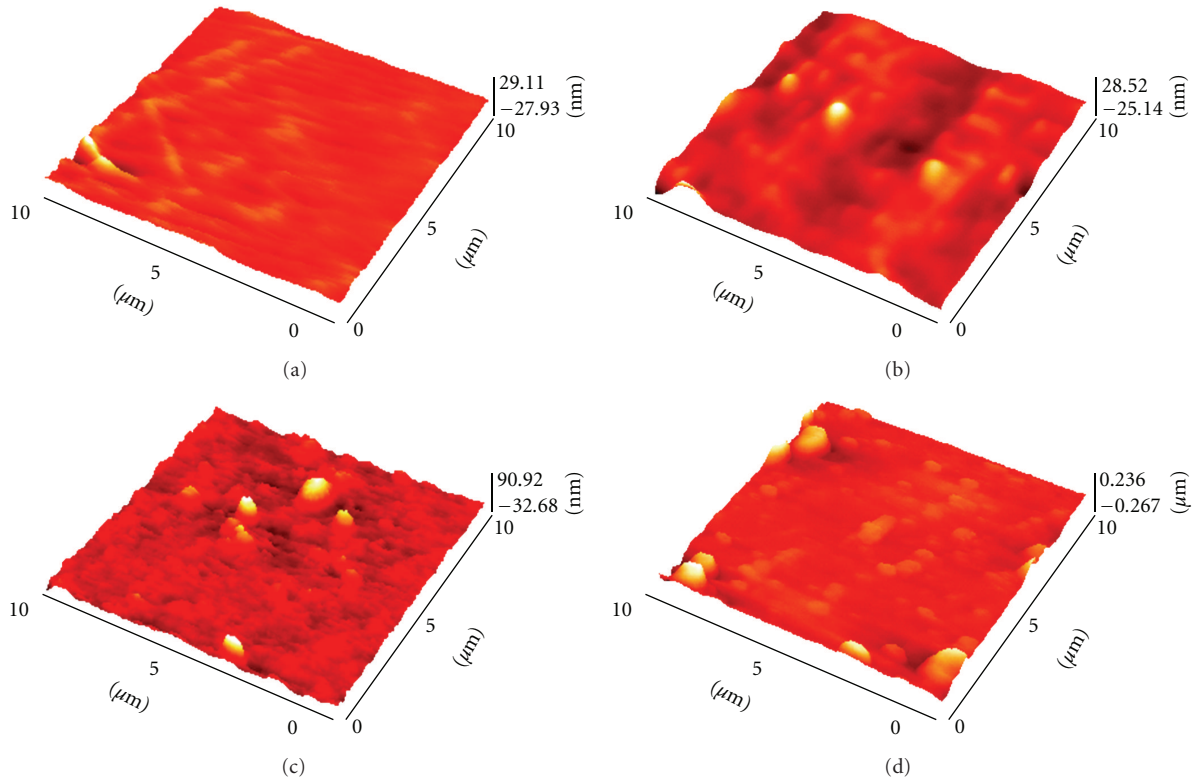


FIGURE 6: AFM image of (a) untreated (b) 3 min treated (c) 5 min treated and (d) 10 min treated PC films.

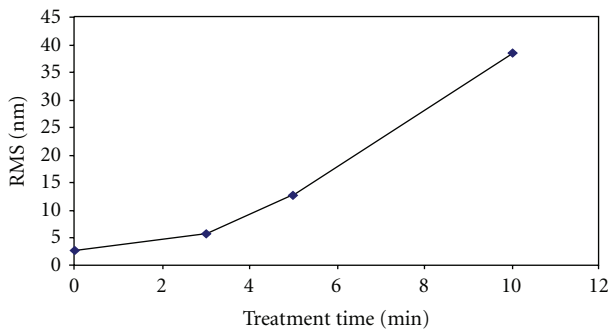


FIGURE 7: Variation of RMS value with respect to treatment time.

respect to treatment time. These polar groups are responsible for the increase in the surface hydrophilicity of the PC films.

3.7. Adhesion Analysis. Both the plasma-treated and untreated samples were tested to understand the effect of hydrophilic groups on bonding strength using T-peel test. For the untreated PC film, the peel strength was noted as 2 N/cm and for the 10-minute plasma-treated PC film, the peel strength was 3 N/cm, which indicates the increase in bond strength due to plasma treatment. The plasma treatment of polymeric surface is commonly believed to be effective because it creates wettable polar surfaces on which the adhesive may spread spontaneously and thus provide extensive interfacial contact. The treatment of polymer film in a plasma environment incorporates hydrophilic groups, which contribute to the

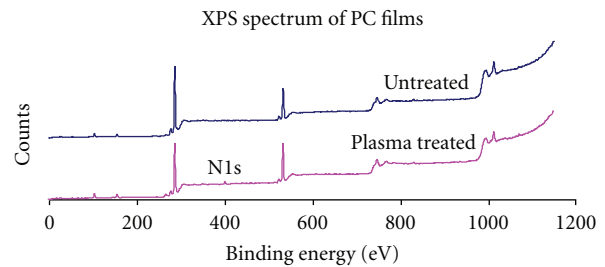


FIGURE 8: XPS spectrum of untreated and plasma-treated PC film.

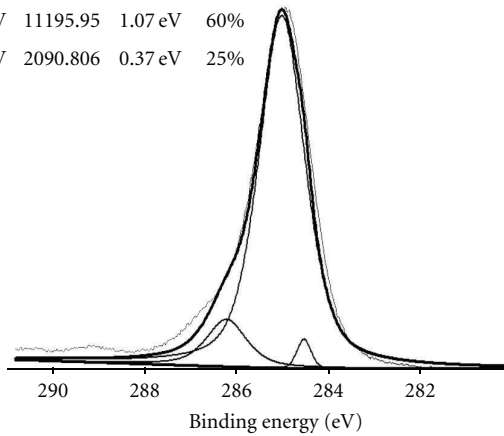
increase in wettability. As a result, the adhesion layer spreads on the surface more easily. Moreover, when these functionalities come into contact with adhesive material, it forms a weak bond due to Van der Waal's force. This force of attraction between the plasma-treated polymer surface and adhesive material contributes to the absorbed increase in bonding strength. AFM images reveal the increase in surface roughness and hence the increase in effective surface area due to plasma treatment. There was mechanical anchoring of adhesive on the surface of the PC film [26].

4. Conclusion

Low-pressure soft plasma treatment has been used to modify the PC film surfaces. It was found that the plasma treatment modified the surfaces both in chemical composition and morphology. The plasma treatment increased polar

Untreated PC C1s

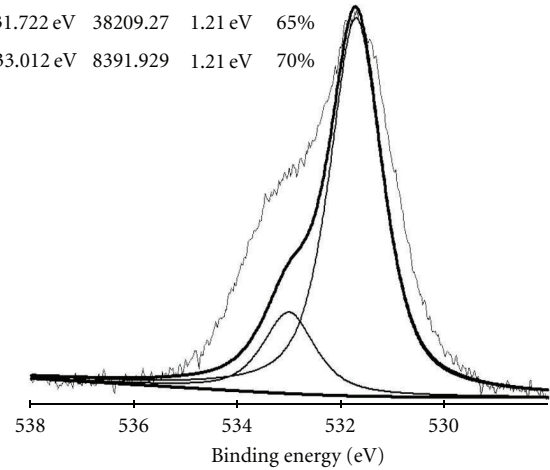
Peak	Position	Area	FWHM	GL%
0	285 eV	90962.23	1.23 eV	48%
1	286.242 eV	11195.95	1.07 eV	60%
2	284.502 eV	2090.806	0.37 eV	25%



(a)

Untreated PC O1s

Peak	Position	Area	FWHM	GL%
0	531.722 eV	38209.27	1.21 eV	65%
1	533.012 eV	8391.929	1.21 eV	70%

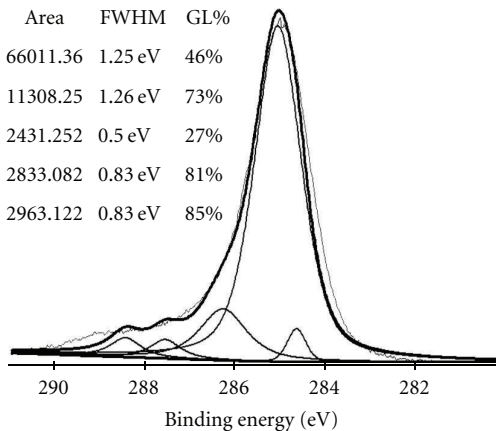


(b)

FIGURE 9: XPS spectra of (a) C1s peak (b) O1s peak of untreated PC film.

Treated PC C1s

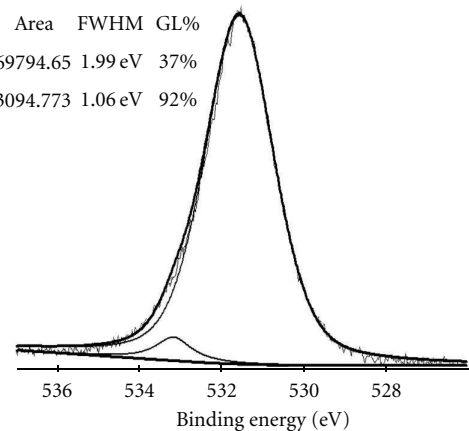
Peak	Position	Area	FWHM	GL%
0	285.012 eV	66011.36	1.25 eV	46%
1	286.242 eV	11308.25	1.26 eV	73%
2	284.592 eV	2431.252	0.5 eV	27%
3	287.542 eV	2833.082	0.83 eV	81%
4	288.432 eV	2963.122	0.83 eV	85%



(a)

Treated PC O1s

Peak	Position	Area	FWHM	GL%
0	531.556 eV	69794.65	1.99 eV	37%
1	533.166 eV	3094.773	1.06 eV	92%



(b)

FIGURE 10: XPS spectra of (a) C1s peak (b) O1s peak of plasma-treated PC film.

functional groups onto the surface of the PC film causing decrease in contact angle and increase in surface energy. The FTIR spectra showed the increase in absorption bands due to plasma treatment. AFM and XRD characterization studies showed increase in surface roughness and increased crystallinity on the PC film. XPS analysis detected polar groups on the PC film. The plasma treatment enhanced the bond strength of the PC surface as indicated by T-peel strength test. This proves that the adhesion can be improved by plasma treatment. All the changes in PC surface made the films more hydrophilic.

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