



## Optical and Electrical Properties of Plasma Surface Treated Polymethylmethacrylate Films

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In the present study, the optical and electrical properties of Polymethylmethacrylate (PMMA) films have been investigated under the effect of direct current (DC) glow discharge plasma. PMMA films have been prepared and treated with low-pressure DC glow discharge plasma at different plasma conditions; mainly various working gas and plasma treatment times. The optical and electrical properties of the treated PMMA samples were measured and compared to the untreated one. Nitrogen, argon, and nitrogen-argon mixture have been used as working gases. Plasma parameters, such as the electron density, the ion density, and electron temperature, were measured, at the position of the PMMA films, using Langmuir probe. The results reveal that, the optical properties of PMMA were found to be more sensitive to N<sub>2</sub>- plasma treatment than Ar and N<sub>2</sub>-Ar gas mixture. The optical energy gap showed a decreasing behavior with increasing the treatment time of N<sub>2</sub> plasma, however, the band tail width and the electrical resistivity increased

**Keywords:** Surface modification, Refractive index, Electrical conductivity, Glow discharge, PMMA

### Introduction

Materials surface modification using various cold plasma techniques, has gained an enormous interest by scientists and research institutions since 1960 until now [1-3]. This method has an advantage over other surface modification techniques represented in the fact that; plasma, with its constituents, electrons, ions, free radicals and UV radiation, have sufficient energy to interact with the surfaces and change its physiochemical properties without affecting the bulk ones. Moreover, it is classified as a clean, fast and safe method. By controlling the plasma treatment conditions, such as the applied power, the treatment time, kind of gas and sample-electrode distance one can obtain the desired surface modification [4, 5].

Despite the excellent physical and chemical properties of polymers, that make them able to replace traditional materials such as metals, glasses and ceramics in many industrial applications, polymeric materials need, in most of their

applications, additional treatments. Cold plasma has been proven to be efficient in tailoring some polymeric features like, the surface hydrophilicity or hydrophobicity, roughness, crystallinity antimicrobial activity and biocompatibility to be suitable for different applications [6-13].

Polymethylmethacrylate (PMMA) of structure, [CH<sub>2</sub>-C(CH<sub>3</sub>).(COOCH<sub>3</sub>)], is optically clear (92% transmission, theoretical limit for normal incidence, in the visible region) through the visible wavelength range with very little ultraviolet absorption until 260 nm [14]. It is widely used for its bulk chemical, mechanical and optical properties in Plexiglass, contact lenses and electron beam lithography. In biomedicine it is applied as dental extender, blood filters, bone cement, plastic surgery fillers. It is hydrophobic, with a low surface energy, and does not swell in aqueous solution.

Polymeric materials, like PMMA, are known to be good insulators which makes them widely applicable in the field of electrical cables

insulation due to their high electrical resistivity. However, in other applications this may be an undesirable property. High resistivity may lead to high static charge problems in some applications such as, polymer films in the packaging industry. The high resistivity causes the buildup of static charges which has a harmful effect on sensitive electronic devices.

Enhancement of polymer conductivity by blending with another polymer or doping with metallic elements is a well-known technology [15-17]. It was proved that there is a direct proportionality between the dopant concentration and electrical conductivity [18-20]. However, irradiating a polymeric surface with a radiation such as DC glow discharge plasma may, also, enhance the polymer electrical conductivity. The reasons are interpreted [21] and, mostly, attributed to the alteration of the chemical composition of the surface as a result of the generation of functional groups such as, hydroxyl and carboxyl groups. Actually, these functional groups, which are generated by plasma, seem to act as dopants and have resulted in the improvement of the electrical conductivity.

The present investigation is devoted to study the influence of changing plasma conditions (nature of working gas and treatment time) on the optical and electrical properties of PMMA films. Pure PMMA were treated by plasma using three different gas environments namely, N<sub>2</sub>, Ar and their mixtures N<sub>2</sub>/Ar (60:40%). The effect of plasma treatment time on the same properties were also, investigated using N<sub>2</sub> as a working gas. Other plasma conditions, i.e., power, pressure and inter-electrode distance were kept constant.

### Material and Methods

The schematic diagram of the DC glow discharge cell used for surface treatment of the PMMA films was described elsewhere [22]. The setup consists of a Pyrex tube of (18 cm long and 13 cm diameter) sealed at both ends with aluminum sheets. Two plane circular copper plates of (5 cm radius) represent the two electrodes with a spacing of 7 cm. The two electrodes move axially forward and backward as a unit controlled by a handle outside the tube. The base pressure is attained into the tube at (10<sup>-3</sup> torr) by using a rotary pump (Edwards high vacuum pump, model ED 200) then the gas is allowed to let in the tube throw a controllable needle valve.

The PMMA films, (thickness of 0.175 mm), were prepared in the laboratory using the solution casting method. The polymer solution was prepared by dissolving the PMMA grains in toluene using a magnetic stirrer, then poured in a clean and dry glass Petri dish and let to dry at room temperature, for several days, until solvent free films were obtained. Then, it has been cut to obtain samples (of size 1.5x2 cm). The films of PMMA were supported on a glass holder and put in front of the cathode at the edge of the negative glow.

The plasma parameters, such as, electron and ion densities and electron temperature were determined using a cylindrical Langmuir probe as a diagnostic method. UV absorption spectra were measured using Perkin-Elmer Lambda 4BSpectrophotometer (190 - 990 nm). The AC electrical conductivity for samples was measured before and after exposure to plasma environment, by using the RLC bridge model (Fluka 3036 BM).

### Theory

#### Optical calculations

Generally, in ideal crystalline materials, no transition takes place in the energy gap. However, in amorphous materials, transitions may occur from occupied extended states of valence band to the empty tail states of conduction band and, from occupied tail states of valence band to empty extended states of conduction band. The density of states of localized states decays exponentially into the gap. Hence for transitions from localized to extended states and from extended to localized states, there is an exponential relation between absorption coefficient  $\alpha(\nu)$  and photon energy,  $h\nu$ , which obeys Urbach's empirical relation [23, 24]

$$\alpha(\nu) = \alpha_0 e^{-h\nu/E_u} \quad (1)$$

Where  $\alpha_0$  is constant,  $\nu$  is the angular frequency of the incident photon,  $h$  is Plank's constant and  $E_u$  is the band tail width of the localized states in the normally forbidden band gap. The optical band gap,  $E_g$ , associated with the amorphous nature of the material, can be obtained by applying the relation given by Davis and Mott [25-27]:

$$\alpha(\nu) = \frac{B(h\nu - E_g)^m}{h\nu} \quad (2)$$

Where  $B$  is a constant and  $m$  is an exponent equals 1/2, 3/2, 2, or 3 according to nature of the electronic transition, whether it is direct allowed or direct forbidden or indirect allowed or indirect forbidden respectively. The value of  $m$  can be estimated if one plot  $(\alpha h\nu)^{1/m}$  versus  $(h\nu)$ , which often yields a straight line fit to the absorption edge. In our case, the best straight-line fit was found for  $m=1/2$  corresponding to a direct allowed transition. An extrapolated  $(h\nu)$  at which  $(\alpha h\nu)^{1/m} = 0$  provides a value of the optical band gap  $E_g$  and  $E_u$  is the reciprocal gradient of the linear portion in the lower photon energy region of this plot.

Photon transmittance  $T$  and absorbance  $A$  are related to the optical absorption coefficient  $\alpha$  according to Lambert's law as

$$\ln\left(\frac{I_0}{I_t}\right) = \ln T$$

$$\alpha d = 2.303A \quad (3)$$

Where  $I_0, I_t$  and  $d$  are the incident intensity, the transmitted intensity, and sample thickness respectively. From equation (3), knowing  $A$ , one can obtain  $\alpha$  [26, 28]. The absorption coefficient  $\alpha$  is also related to the extinction coefficient  $K$  by the relation

$$K = \alpha \frac{\lambda}{4\pi} \quad (4)$$

where  $\lambda$  is the wave length of light. As the reflectance  $R$  is related to the transmittance  $T$  according to the relation:

$$T = (1 - R)^2 e^{-A} \quad (5)$$

one can obtain the value of the reflectance  $R$ . Substituting the value of  $R$  into the following equation, the refractive index  $n$  of the films can be determined [29]

$$n = \left(\frac{1+R}{1-R}\right) + \sqrt{\frac{4R}{(1-R)^2} - K^2} \quad (6)$$

#### Electrical calculations

The electrical conductivity,  $\sigma$  of polymers is attributed, mainly, to their conjugated structure. Conjugated polymers have alternating single and double carbon-carbon bonds along the polymer backbones. The carbon atoms along the backbone

are sp-2 hybridized, which leaves one unhybridized p-z orbital sticking up out of the plane of the polymer. The electrons in these pi-orbitals form a delocalized electron cloud, which is free to conduct.

Polymers are insulators possessing a very high resistivity of about  $10^{10} - 10^{16} \Omega m$ . One important property is the dielectric constant (or permittivity). The dielectric constant,  $\epsilon'$ , is the measure of the ability of a material to be polarized by an electric field, and closely related to the capacitance,  $C$ , i.e. the ability to store electric charge. Dielectric constant of materials often varies with applied frequency at constant temperature. The measured capacitance,  $C(\omega)$ , can be used to calculate the dielectric constant,  $\epsilon'$ , according to the following relation [18]:

$$\epsilon'(\omega) = C(\omega) \frac{d}{\epsilon_0 A} \quad (7)$$

The frequency dependence implies that the material doesn't respond instantaneously to an applied field upon polarization. Therefore, the response of the material to an alternating field is characterized by a complex permittivity defined as:

$$\epsilon^* = \epsilon' - i\epsilon'' \quad \text{with} \quad \epsilon'' = \epsilon' \tan \delta \quad (8)$$

Where  $\epsilon'$ , and  $\epsilon''$  are the real and imaginary parts of the permittivity, and  $\delta$  is the loss tangent. Using Equations (7 and 8), the AC conductivity,  $\sigma_{AC}$  can be calculated according to the following equation:

$$\sigma_{AC}(\omega) = \epsilon_0 \omega \epsilon'' \quad (9)$$

Where  $\omega$  is the applied angular frequency =  $2\pi f$ .

## Results and Discussion

### Langmuir probe measurements

After plasma ignition, the bias voltage and internal pressure were kept constant at 500 volt and 0.4 Torr respectively. Single Langmuir probe was used, as a diagnostic technique, to measure the axial values of the plasma parameters at the position of the films. Calculations of the electron density  $n_e$ , ion density  $n_i$  and electron temperature ( $T_e$ ) by using Langmuir probe can be obtained by equations given elsewhere [30]. The total flux of a given ionic species (ion bombardment of the surface)  $\Gamma_s$ , is also an important parameter in

plasma surface treatment; therefore, it has been calculated by Bohm criterion which can be applied to every ion species [31]

$$\Gamma_s = n_e \sqrt{\frac{kT_e}{m_i}} \exp(-0.5) \quad (10)$$

Table (1) gives the variations of the measured electron and ion densities, electron temperature and ion flux, at the position of the substrate, under the different plasma gas composition. The variation of plasma parameters, given in the table, had been discussed in a previous work [32].

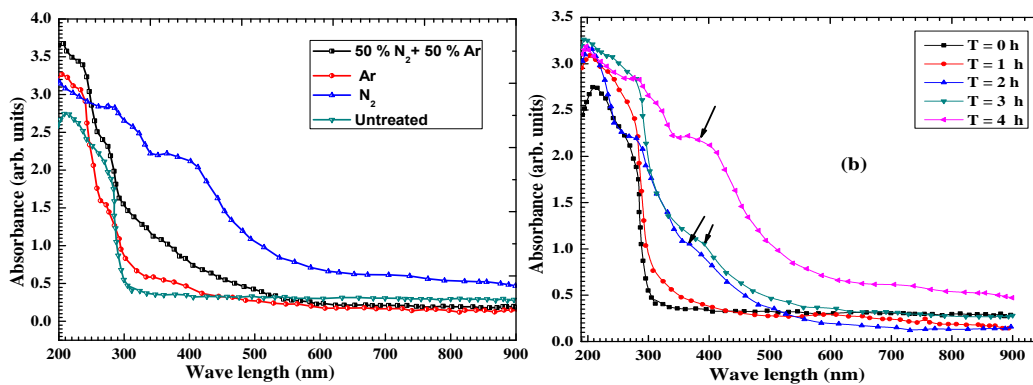
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#### Optical analysis

Fig. (1a) shows the absorption spectra for three samples of PMMA exposed to N<sub>2</sub>, Ar, and N<sub>2</sub>/Ar (60:40%) mixture for T=4hrs including the untreated sample. The spectral lines for samples treated with the three gases have nearly the same behavior as that for the untreated sample. The spectral lines have peaks of maximum absorbance in the range (190- 300 nm) corresponding to n→σ\* transition, as π→π\* and n→π\* transitions need relatively low energy and, hence, occur at higher wavelength [33]. Considerable increases in the UV absorbance for the treated samples were observed. Moreover, the spectral lines of the treated samples showed increased broadening, the sample exposed to N<sub>2</sub> has the most broadened line and its peak shifted to the shorter wavelength. These changes in the optical absorption indicates that the plasma irradiation induced some chemical and structural changes in the PMMA films. The spectral lines broadening may be attributed to the existence of more transitions from higher vibration levels of the ground state to the higher sublevels of the first excited singlet state [34].

**Table (1):** Variation of different plasma parameters as a function of plasma gas compositions

Gas composition	$n_e$ (cm <sup>-3</sup> )	$n_i$ (cm <sup>-3</sup> )	$T_e$ (eV)	$\Gamma_s$ (m <sup>-2</sup> s <sup>-1</sup> )
100% N <sub>2</sub>	3.422x10 <sup>9</sup>	2.205x10 <sup>10</sup>	4.59	2.66393x10 <sup>19</sup>
N <sub>2</sub> /Ar (60:40%)	4.439x10 <sup>9</sup>	3.177x10 <sup>10</sup>	4.27	3.315x10 <sup>19</sup>
100% Ar	6.81x10 <sup>9</sup>	5.35159x10 <sup>10</sup>	3.15	5.30002x10 <sup>19</sup>



**Fig. (1):** UV-visible absorption spectra of (a) (N<sub>2</sub>, Ar and N<sub>2</sub>-Ar plasmas) treated PMMA samples at exposure time 4 hours and (b) N<sub>2</sub> plasma treated PMMA

The UV absorption spectral lines for films exposed to N<sub>2</sub> plasma, for different exposure times, are represented in Fig. (1b). The spectral lines showed increasing absorbance and broadening as the treatment time increases. This behavior is similar to that reported for many polymeric materials exposed to different kinds of radiation such as, ion and electron beams, UV and Gamma radiation. Raia et al. [35] attributed these changes to the induced formation of conjugated dienes (-C=C-) groups through unsaturation of the PMMA chain. The peaks of absorption spectra that lie in the wavelength range 200-350 nm, showed an increasing intensity for a time duration of about 4 hrs after which the intensity starts to decrease. Mortazavi et al. [36] attributed this decrease to the loss of carbonyl compound (C = O) which may be due to the oxidative decomposition of the double bonds' plasma or etching effect on the surface of the substrate. The increase followed by a decrease in the UV-Vis absorbance peak for PMMA films upon plasma treatment, is similar to previously reported works for samples treated with Gamma and carbon ion radiation [35,37]. They attributed such behavior to the decrease in the number of radicals due to crosslinking.

The untreated PMMA films are clear and transparent. However, upon increasing the treatment time, their color gradually changed into light brown. The change in color was found to be accompanied by the appearance of a new absorption band in the region 350-450 nm, indicated by arrows in Figure (1b), attributed to  $\pi - \pi^*$  transitions. These bands were observed for films treated for 2 hrs. to be overlapped with the main absorbing band, and increased with increasing treatment time. The new band is an indication of the presence of a color center which

may be attributed to the generation of some types of radicals or electrons trapping during treatment [35]. A. Atta et al. attributed the change in color of PET, under argon plasma treatment, to the formation of hydrogenous carbon clusters [38].

The calculated values of the energy band gap;  $E_g$ , and band tail width,  $E_u$ , for the films upon exposure to the three gases are given in Table (2). It is clear that, values of  $E_g$  for all treated samples are smaller than that of the pristine one. However, the similarity of the values of  $E_g$  for treated samples indicates that, there is no preference for a gas over the other in this respect. Meanwhile,  $E_u$  showed increasing values for the three treated samples compared to the pristine one. However, the largest value, which is 0.880 eV, was found for the sample treated with N<sub>2</sub>/Ar gas mixture. The variations in the  $E_g$  and  $E_u$  for samples treated with nitrogen for different time intervals are shown in Fig. (2).  $E_g$  decreases from 3.87 to 3.07 eV and  $E_u$  increases from 0.266 to 0.817 eV with increasing treatment time. In Fig. (1a, b), the apparent shift of the spectral lines towards longer wavelength can be considered as an evidence for the existence of  $\pi$ -delocalization along the polymer chain and hence, smaller band gaps of the treated samples. Madhu et al. [39] interpreted the decrease of  $E_g$  for ion irradiated PMMA films as related to the cleavage of the C-C bond due to the effect of irradiation and dehydrogenation of polymer chains which, in turn, induces the formation of -C=C- bonds, then promote the delocalization of charge carriers. The increasing of  $E_u$  values with the plasma treatment may be attributed to the increase of the width of localized states which is an evidence for increasing the disorder in the PMMA films.

**Table (2): Variation of band tail width ( $E_u$ ) and energy band gap ( $E_g$ ) with different plasma gas compositions**

Gas composition	$E_g$ (eV)	$E_u$ (eV)
Untreated	3.87	0.266
100% N <sub>2</sub>	3.07	0.817
N <sub>2</sub> /Ar (60:40%)	3.06	0.880
100% Ar	3.09	0.768

The effect of the type of gas on the value of refractive index,  $n$ , is depicted in Fig. (3a). An increasing value of  $n$  was observed on treating the films with Ar, N<sub>2</sub>: Ar and N<sub>2</sub>. However, at  $\lambda = 440$

and 534 nm the values of  $n$  decreased for the films treated with Ar and N<sub>2</sub>: Ar respectively. The

decreasing effect due to Ar compared to pure N<sub>2</sub> and the mixture, may be attributed to the higher value of Ar ion flux compared to that of N<sub>2</sub>. As the higher ion flux leads to an increase in the etching rate of the films surface, which is related to the decrease in refractive index corresponding to the appearance of carbonized structure. Fig. (3) represents the variation of refractive index with wavelength for N<sub>2</sub>-plasma treated samples for different exposure times including the untreated one. The untreated sample showed nearly constant refractive index all over the range (400-900 nm), while as the treatment time increased the refractive index increases with decreasing the wavelength especially in the wavelength range (400-500) nm, which is in good agreement with the literature [36, 40]. The change in the refractive index can be attributed to localized density increases arising from photo-induced cross-linking [41]. From the chemical point of view, the increase in (*n*) at shorter wavelength is associated with the presence of small graphite islands with short chains of conjugated carbon double bond.

The extinction coefficient, *K*, over the absorption region (300-900 nm) for PMMA samples exposed to N<sub>2</sub>, Ar, and N<sub>2</sub>-Ar mixture are depicted in Fig. (4a), which shows that the value of *K* decrease as the wavelength increases, a common behavior with that one which is exposed to N<sub>2</sub>-plasma is of higher values. The resulting change in the values of *K* is an indicator for the change in the structural

nature of the treated films. The samples treated with N<sub>2</sub> and N<sub>2</sub>/Ar show weak attenuation in the spectral range (400-500 nm) which makes them applicable in the field of filters industry

The behavior of *K* with the exposure time was examined for samples treated with N<sub>2</sub> –plasma at two selected wavelengths (200 and 400) can be shown in Fig. (4b). According to the Figure the value of *K* is almost constant with increasing the treatment time in the low wavelength. However, at longer wavelength, it grows steadily with time.

### Electrical analysis

The surface AC conductivities,  $\sigma_{AC}$ , of our samples were measured at different treatment times. Fig. (5a) shows the variation of conductivity with frequency in the range (0.1–1 M Hz) for untreated and treated samples with the three discharge gases (Ar, N<sub>2</sub>, and Ar-N<sub>2</sub> mixture). The curves show a steady increase of conductivity with frequency for all samples. This may be attributed to the enhancement of charge carriers transition as the applied field frequency increase. However, the values of  $\sigma_{AC}$  for the three samples are less than that of the untreated one all over the frequency range. On the contrary, with the decreasing value of optical band gap, a remarkable decrease in the values of conductivity (within a narrow range) with increasing the exposure time can be seen Fig. (5b).

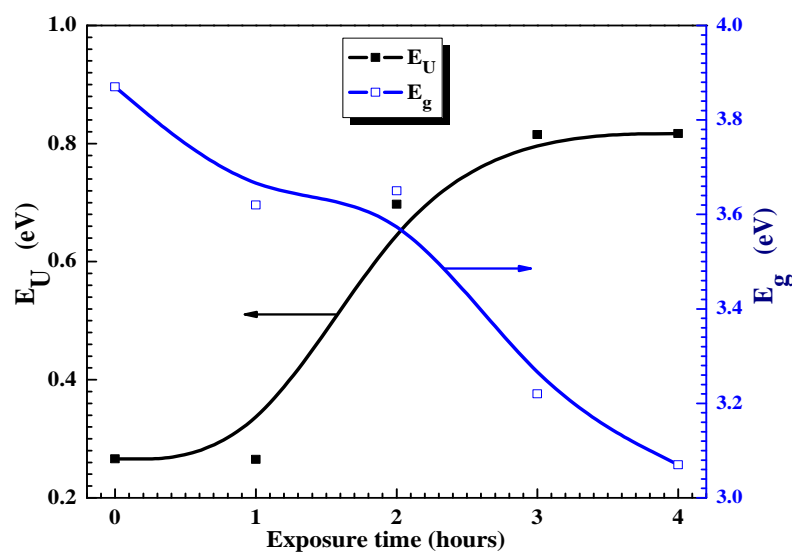


Fig. (2): Variation of band tail width and optical energy band gap of N<sub>2</sub> plasma treated PMMA samples at different exposure time.

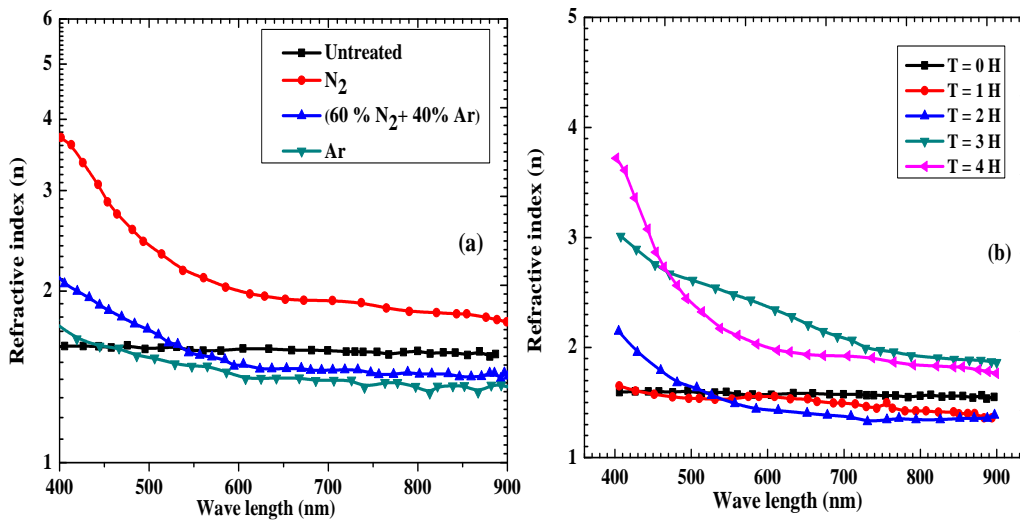


Figure (3): Variation of refractive index with wavelength of (a) (N<sub>2</sub>, Ar and N<sub>2</sub>/Ar plasmas) treated PMMA samples and (b) N<sub>2</sub> plasma treated PMMA samples at different exposure time

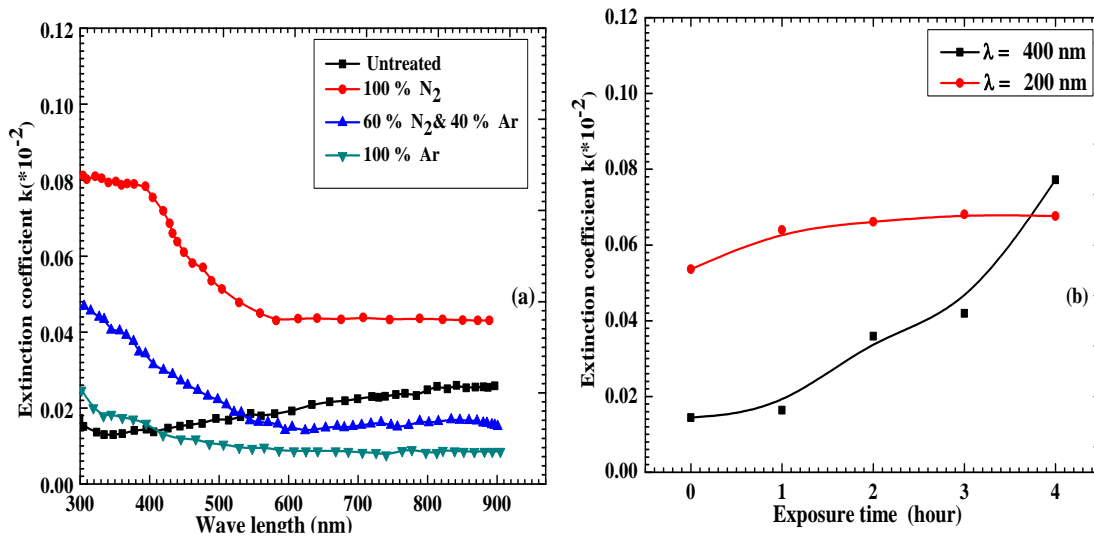


Figure (4): The extinction coefficient of (a) (N<sub>2</sub>, Ar and N<sub>2</sub>-Ar plasmas) treated PMMA samples as a function of wavelength and (b) N<sub>2</sub> plasma treated PMMA samples as a function of exposure time at two different wavelengths

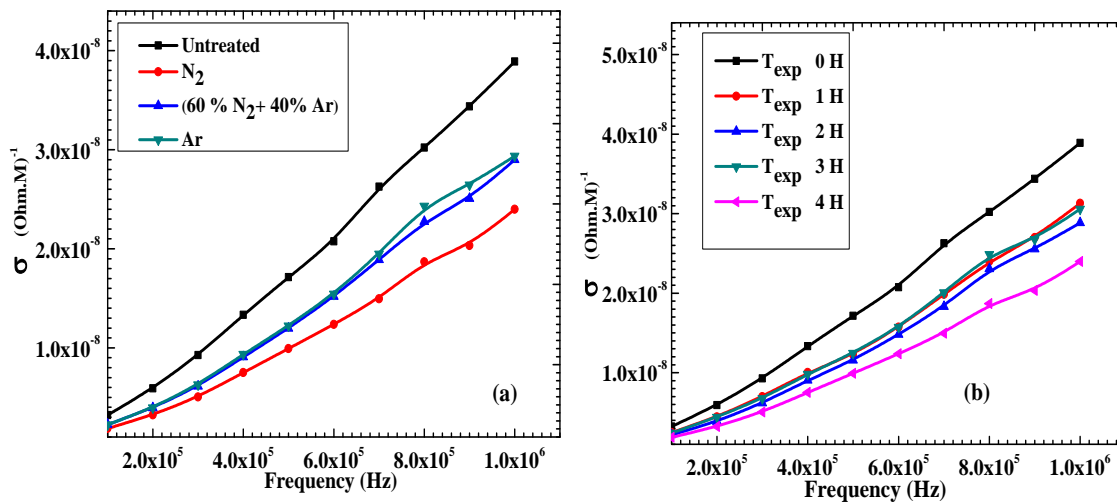


Figure (5): Variation of AC electrical conductivity of (a) ( $N_2$ , Ar and  $N_2$ -Ar plasmas) treated PMMA samples and (b)  $N_2$  plasma treated PMMA samples at different exposure time

According to the literature [42] they attributed the decrease of conductivity to the induced cross-linking at the surface of the films which results in the formation of some defects in the energy gap that, may be, acts as a barriers against the charge carriers, thus reducing the conductivity i.e. increasing electrical resistivity, the feature that make it excellent as a cable or wire insulators.

### Conclusion

Exposing polymer surfaces to low pressure plasma is a simple and effective way for modifying and improving both the optical and electrical properties. The experimental study in this research has been focused on modification of the polymethylmethacrylate (PMMA) surface using low pressure dc glow discharge plasma. The influence of various gas plasma treatments was investigated and compared. The effects of nitrogen, argon and nitrogen-argon mixture plasma treatments on the PMMA were discussed. The results of optical and electrical properties measurements and its dependence on the kind of discharge gas and time of treatment are summarized. The results showed that plasma treatment duration did give different degrees of influence on the properties of PMMA. The optical properties of PMMA were found to be more sensitive to  $N_2$ - plasma treatment than Ar and  $N_2$ -Ar gas mixture. The exposure of PMMA films to  $N_2$  plasma for different treatment times resulted in changes in the structural nature consequently, the optical and electrical properties of PMMA films.

The evidence for this is the changing values of optical parameters and Ac conductivity.

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