

Synergistic Effect of Atmospheric Pressure Plasma Pre-Treatment on Alkaline Etching of Polyethylene Terephthalate Fabrics and Films

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Abstract Dyeing of PET materials by traditional methods presents several problems. Plasma technology has received enormous attention as a solution for the environmental problems related with textile surface modifications, and there has been a rapid development and commercialization of plasma technology over the past decade. In this work, the synergistic effect of atmospheric pressure plasma on alkaline etching and deep coloring of dyeing properties on polyethylene terephthalate (PET) fabrics and films was investigated. The topographical changes of the PET surface were investigated by atomic force microscopy (AFM) images, which revealed a smooth surface morphology of the untreated sample whereas a high surface roughness for the plasma and/or alkaline treated samples. The effects of atmospheric pressure plasma on alkaline etching of the structure and properties of PET were investigated by means of differential scanning calorimetry (DSC), the main objective of performing DSC was to investigate the effect of the plasma pre-treatment on the T_g and T_m . Using a tensile strength tester YG065H and following a standard procedure the maximum force and elongation at maximum force of PET materials was investigated. Oxygen and argon plasma pre-treatment was found to increase the PET fabric weight loss rate. The color strength of PET fabrics was increased by various plasma pre-treatment times. The penetration of plasma and alkaline reactive species deep into the PET structure results in better dyeability and leaves a significant effect on the K/S values of the plasma pre-treated PET. It indicated that plasma pre-treatment has a great synergistic effect with the alkaline treatment of PET.

Keywords: atmospheric pressure plasma, synergistic effect, alkaline treatment, polyethylene terephthalate materials, K/S value, surface roughness

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(Some figures may appear in colour only in the online journal)

1 Introduction

Polyethylene terephthalate (PET) materials dominate the world synthetic polymer industry. Also the PET polymer molecule has no reactive chemical functionalities, such as carbonyl, hydroxyl and carboxylic for a dyeing reaction mechanism, due to the hydrophobic nature and the compact molecular structure of PET^[1,2] which is causing environmental problems for its hydrophobicity and poor dyeing performance etc. Generally it is a predicament to dye PET materials with dyes without such functional groups. The demand for environmentally friendly processes has led to intensive research into the development of new plasma pre-treatment techniques to improve the alkaline etching and deep coloring of PET materials with less energy requirements, chemicals and cost. Surface properties of materials such as wettability, chemistry, charge and topography^[3] are of great importance in textile appli-

cations in fabric handle or dyeing. The most desirable property of PET is its good mechanical attributes due to its exceptional tensile strength, abrasion resistance and resilience^[4].

Plasma treatment is considered as a dry process since it does not require the use of water or chemicals^[5,6]. In addition, plasma is highly efficient for increasing the surface energy and able to change the surface properties and consequently improve its dyeability^[7,8]. One of the typical plasma discharges that operate at atmospheric pressure is the dielectric barrier discharge (DBD)^[9,10]. The DBD is used to modify the surface properties of natural and synthetic polymers, thereby improving their hydrophilicity, adhesion properties and wettability^[11–13]. Plasma treatments do not involve handling hazardous chemicals and thus there are no problems of effluents and their treatment. Environmentally friendly surface modifications of PET materials by plasma do not affect the bulk properties but only im-

prove its wettability [14,15].

In a wet chemical approach to surface modification, a material is treated with liquid reagents to generate reactive functional groups on the surface, and this well-established method does not require specialized equipment being carried out in most laboratories. Alkaline hydrolysis is one of the most rapid methods to modify the chemical and physical characteristics of the surface [16]. The nucleophilic attack of a base on the electron-deficient carbonyl carbon of the ester linkage causes chain breaking along the polymer chain, producing carboxylate and hydroxyl polar end-groups. The increased surface polarity leads to better wettability. However, PET has weak resistance to strong bases when treated with the alkaline solution, it can be broken down into its monomers, terephthalic acid and ethylene glycol, through a hydrolysis reaction with alkaline; these shortcomings reduce the range of its application, especially in the technical area [17].

Therefore, our main objective in this work was to investigate the effect of oxygen/argon plasma pre-treatment on 4% alkaline etching at 95 °C, to create functional groups of PET fabrics, were carried out using the dielectric barrier discharge (DBD), which has proven to be efficient in the surface treatment to reduce the degree of degradation. The next step was coloring treatment of PET fabrics and films with disperse dye to investigate the changes in dyeability. UV-visible spectrometer and color measurement spectrophotometer for determination of dye uptake and K/S value respectively were observed with increasing weight loss and treatment times. The evaluation of the surface morphology, mechanical properties, dyeability, and fastness properties furnished evidence that plasma pre-treatment enhanced the dyeability and sufficiently lowered the detrimental effects of alkaline treatment of PET materials.

2 Experiment

2.1 Materials

A plain woven polyethylene terephthalate (PET) fabric with the fabric density of 86.1 g/m² was used for the experiment and films of 200 μm in thickness were selected. The fabrics and films were washed in distilled water for 40 min at 100 °C, 60 °C and dried in 90 °C, 70 °C for 1 h respectively. The cleaned samples were chopped into the dimension of 6×6 cm² and then weighted. Oxygen and argon (purity 99.99%) were bought from the Shanghai Cheng Gong Gas Industry Co., Ltd. Sodium hydroxide 99% from (Xin Jiang Tian Cheng Corporation Limited, China) was used as such, no further purification was needed. C.I. Disperse Blue 183, with λ_{max} 530 nm was procured from the Zhejiang Longsheng Group Co., Ltd, (Lonsen), was used as received and its molecular structure is shown in Fig. 1. All the experiments were carried out using distilled water.

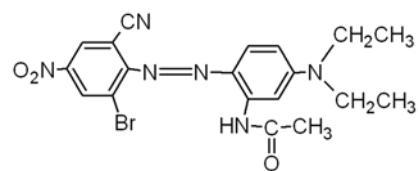


Fig.1 Structure of the Blue dye (C.I. Disperse Blue 183)

2.2 Atmospheric oxygen and argon plasma treatments

A dielectric barrier discharge (DBD) plasma system is used for the surface treatment in this study. Shown in Fig. 2(a), the discharge is composed between two parallel copper electrodes with a diameter of 60 mm, which are covered with a very thin Al₂O₃ ceramics plate and the electrode gap is 3 mm. Shown in Fig. 2(b) are the discharge voltage and current waveforms, the discharge current displays the glow-like regular multiple pulse discharge peaks. It is believed that the generated discharge characterization AC power in the frequency of about 20 kHz was applied between the upper and grounded electrode. The plasma system was evacuated first for 10 min and then filled with a mixture of oxygen and argon for 5 min. The flow rate ratio of O₂/Ar = 10:1. The pumping was stopped and the filling of the mixed gases was continued until to the atmospheric pressure. The discharge started and the fabrics and films were treated for a certain of duration of time. The discharge images are taken by the ICCD (Andor i-star DH734). The exposing time was 25 μs with a 250 gain and 10 times accumulated.

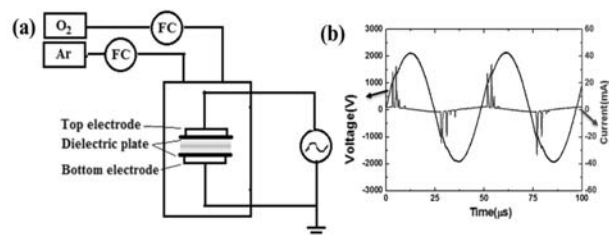


Fig.2 Experimental setup of (a) the DBD discharge plasma system and (b) the *I-V* curve of a typical discharge voltage and current waveforms at 20 kHz

2.3 PET fabric etching by alkaline solution

Alkali solubility values of untreated and plasma treated fabrics and films at optimum condition were realized using a concentration of alkaline solution of 4% w/w. The liquor to sample ratio was 50:1. The alkaline solution was heated and then the sample was added and immersed into a water bath; the beakers were kept in the bath at a constant temperature of 95 °C for various times of 5 min, the alkaline profile is shown in Fig. 3. At the end of the designated time, the fabrics and films were removed and rinsed several times with distilled water, then washed for 15 min and followed by a drying process in an oven set at a temperature of 100 °C and 70 °C for 30 min respectively. Each of the treated

samples was weighed in order to determine the extent of the damage during the alkaline treatment.

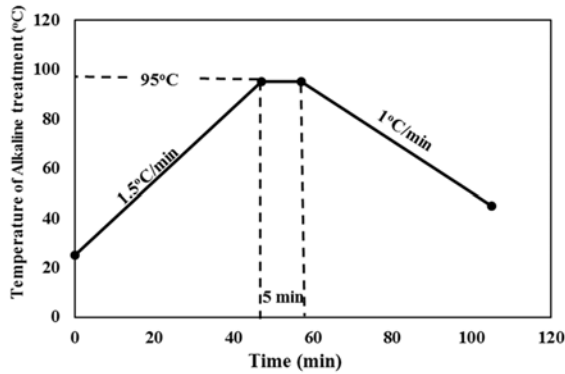


Fig.3 Profile of the alkaline curve

2.4 Dyeing of pre-treated PET fabric

2.4.1 Dyeing procedure

Dyeing was carried out in a dye bath containing 2% o.w.f shade in distilled water without any auxiliaries and a liquor ratio with the disperse dyes of 1:50. The dyeing profile is shown in Fig. 4. The maximum temperature is held constant at 100°C and kept at this temperature for 60 min. After finishing the dyeing process, the dyed samples were rinsed with water, fully washed and dried in air. The solution was used to determine the dye uptake.

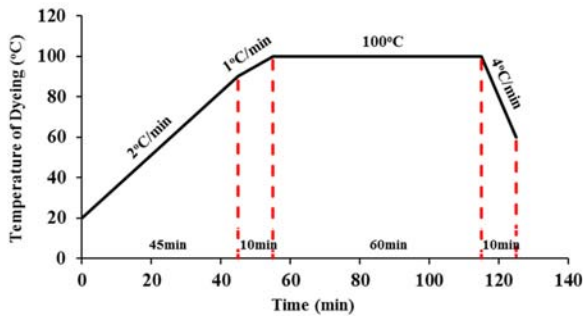


Fig.4 Profile of dyeing curve

2.4.2 Determination of dye uptake

To determine the amount of dye uptake, samples were removed from the dye bath; the refuse dye liquor was put into a 50 mL flask and 5 mL was used per measurement, and the wavelength at the maximum absorbance (530 nm) for the dyes (C.I. Disperse Blue 183) was applied to measure the absorbance of the dyed solution. The percentage of dye uptake (E) was calculated using Eq. (1):

$$E = \frac{A_0 - A_t}{A_0} \times 100, \quad (1)$$

where A_0 is the absorbance of the dye solution at 0 min, and A_t is the absorbance of the dye solution at t min.

2.4.3 Dyeability analysis

The reflectance of the cleaned dyed samples was measured with a spectra flash SF-600 plus (Datacolor CO., USA) color measurement spectrophotometer. The dye absorbance was adopted by measurement of the average of four reflectances, taken at different positions on the dyed fabric; the range of measurement was 360-700 nm and the reflectance at the wavelength of maximum absorption λ_{\max} was used to calculate the color yield of the dyed sample by applying the Kubelka-Munk [18] equation:

$$K/S = \frac{(1 - R)^2}{2R}, \quad (2)$$

where R is the decimal fraction of the reflectance of the dyed fabric, K is the absorption coefficient of the substrate, and S is the scattering coefficient of the substrate. The percentage increase in the color strength (I) was obtained from the following equation:

$$I = \frac{(K/S)_1 - (K/S)_0}{(K/S)_0} \times 100, \quad (3)$$

where $(K/S)_1$ is the color strength of the dyed treated sample and $(K/S)_0$ is the color strength of the dyed untreated sample.

2.4.4 Color fastness properties

Color fastness was evaluated according to the respective international standards. The specific tests used were ISO 105-F10 for color fastness to washing and ISO 105-B02 for color light fastness.

2.5 Fabric surface characterization

2.5.1 Atomic force microscopy (AFM)

Atomic force microscopy (AFM) (Multimode Nanoscope IIIa, Digital Instrument, USA) was used to examine the surface morphology of the PET sample before and after the plasma pre-treatment on alkaline. The roughness of the PET sample was analyzed using a computer. Two parameters, such as the mean square root of roughness (RMS) and the average roughness (Ra) were calculated from the following Eqs. (4) and (5) [19]:

$$RMS = \sqrt{\frac{\sum_{n=1}^N (Z_n - \bar{Z})^2}{N - 1}}, \quad (4)$$

$$Ra = \frac{\sum_{n=1}^N |Z_n - \bar{Z}|}{N}, \quad (5)$$

where RMS represents the roughness of the standard deviation from the mean height, Ra represents the roughness of the mean deviation from the mean height, N is the number of data points in the image, n and $\bar{\cdot}$ are the pixel locations on the AFM image, Z_n is the height value at n locations, and \bar{Z} is the height value of the center plane.

2.5.2 Contact angle wetting experiments

The contact angle measurements of individual PET fabrics and films was performed using a MALSI vision Inspection and Measurement CO, Ltd. Drop Meter A-200. The test unit utilizes drop image advanced software for resolving all contact angle measurements and surface energy analysis. The 25×25 mm PET specimens were cut, and the measurements were taken immediately after placing the water drop contact on the surface.

2.5.3 Tensile strength analysis

Tensile strength testing of the PET fabric and film was carried out on a YG065H Series Strength Tester (Laizhou Electron Instrument Co. Ltd). All tensile tests were performed at standard room temperature. At least five samples were tested for each batch. The samples had dimensions of 50 mm in width and 240 mm length, and the average values were reported. Tensile testing was done on the basis of two sets of test specimens: one in the warp and other in the weft direction. The tensile stress-strain curves were obtained by determining the engineering stress and engineering strain according to the standard test procedure (ASTM D5035).

2.5.4 Thermal properties

The melting point temperatures and heat of fusion of all the PET materials were determined by using a differential scanning calorimeter (DSC) (Perkin-Elmer Corporation, USA). The samples were dried under a vacuum at 80 °C for 12 h to remove any residual moisture. All samples in the range of 5 mg were placed in the standard aluminum sample pans, and the scans were carried out at a heating rate of 10 °C/min under flowing nitrogen at a flow rate of 50 mL/min. The samples were kept for 2 min at 285 °C to eliminate the thermal history. Then the samples were cooled to below 0 °C at a cooling rate of 10 °C/min. The instrument was calibrated using pure indium.

3 Results and discussion

Fig. 5 shows the change in the weight loss of PET fabrics and films treated with alkaline without plasma pre-treatment, plasma initiated in O₂/Ar mixture and plasma pre-treatment on 4% alkaline etching w/w at 95 °C. The change in weight is deduced from the difference in the weight of the fabric or film before and after treatment with alkaline, plasma and a combination of both alkaline and plasma. Alkali treatment of PET fabrics at 5 min only shows the least weight loss, which means degradation was weak with minimal at a short treatment time and less roughening of the surface was reported. However, after using 3 min plasma pre-treatment, the weight loss rate was noticeable for only 5 min alkaline treatment, but alkali treatment of PET film at 5 min only shows a better value than the fabric.

The weight loss rate of PET treated by O₂/Ar plasma and alkaline has been more than the total amount of the fabric and film by the two treatments separately, which shows the synergistic effect of plasma pre-treatment on the alkaline etching treatment in both PET materials. According to the statistical evaluations there are important differences with respect to gas type, time and temperature.

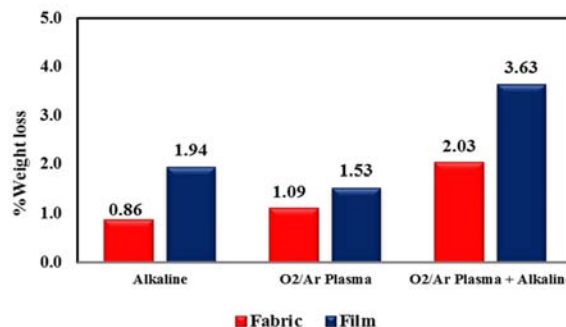


Fig.5 Weight loss of PET fabrics and films effects of plasma pre-treatment on alkaline etching, concentration 4% at 95 °C

Moreover AFM analysis gave information on the plasma induced surface chemical and topographical modifications. Fig. 6 shows AFM images of PET fibers surface and films before and after alkaline and plasma treatment. As shown in Fig. 6(a1, b1) and (a2, b2), the untreated samples show a relatively flat surface and the samples treated by plasma for 3 min show a small extent of surface roughness and edges of a hill, uniformly distributed on both the fiber (fabric) and film PET surface. Fiber and film surfaces in Fig. 6(a3, b3) of the alkaline treated PET samples at 5 min show a few rises to pits and craters. While samples in Fig. 6(a4, b4) that were pre-treated by plasma for 3 min and etched with 4% alkaline for 5 min at 95 °C showed a rather broad and smooth wavy surface as a result of the alkali reaction. The number of ridges on the surfaces is influenced by the plasma pre-treatment and alkaline etching time, but in the film surface for short treatment times the amorphous regions are more easily removed to create a fine structure consequently leading to a decrease in the surface roughness. Longer alkaline treatment times caused the detachment of PET crystallites, which led to a decrease in the size of surface undulations but increased their number.

Table 1 shows surface wettability (Water Contact Angle). Measuring of the contact angle was difficult because the water droplets spread immediately and deposited on the surface of the PET fabric. However, it was done by putting dye solution drops on different areas of each sample in our other publication.

To compare the wettability of plasma and alkaline treated PET fabrics and films, their respective contact angles were measured. Table 1 shows the dynamic contact angles as a function of the flow rate and exposure time. Contact angles of the fabrics are shown when a water droplet was placed on these fabrics, which spreads

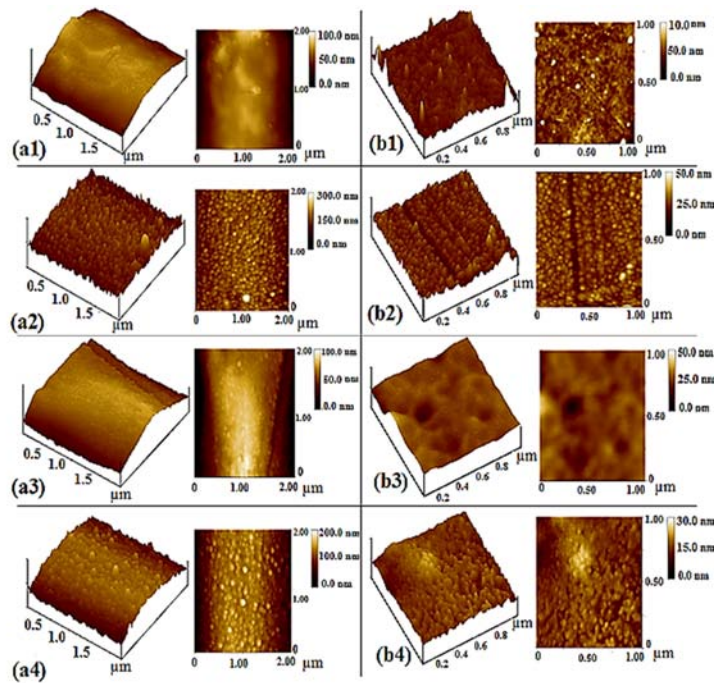


Fig.6 AFM images of PET fabric and film respectively. (a1, b1) untreated, (a2, b2) treated in O₂/Ar plasma, (a3, b3) treated by alkaline 4% at 95 °C, (a4, b4) pre-treated by O₂/Ar plasma and 4% alkaline etching at 95 °C

Table 1. The effect of pre-treatment passes on contact angle of PET fabrics and films

	Wettability Time	PET	
		Fabrics	Films
Untreated	0	CA: 90.6 ± 5.5°	CA: 86.4 ± 0.4°
Treated by plasma only	3min	CA: 10.2 ± 0.5°	CA: 37.7 ± 0.1°
Treated by alkaline only	5min	CA: 17.2 ± 1.7°	CA: 45.4 ± 3.1°
Treated by plasma + alkaline	3min + 5min	CA: 0.0 ± 0.0°	CA: 25.4 ± 0.0°

completely within less than 2 s. In the untreated sample the water droplet does not entirely spread, but the treated PET fabrics show a static water contact angle (<90°), indicating that the fabric is wetted by water easily. The contact angles of treated PET films decreased after plasma treatment, alkaline treatment and a combination of plasma pre-treatment on alkaline etching. The wettability of plasma treated PET films increased with an increase of exposure time and different alkaline treatment times compared with the untreated samples, this is attributed to an increase in the surface free energy.

For mechanical properties: to study the changes in the tensile strength and elongation to break properties and the effect of O₂/Ar plasma pre-treatment on alkaline etching of the fabrics and films, the testing was carried out in both the weft and warp directions, the results are shown in Fig. 7. It can be seen that there is a loss in the average tensile strength after pre-treatment with plasma for 3 min and treatment with alkaline for 5 min. It can also be seen that the load at the break and the elongation at the break decreased after the plasma pre-treatment. This decrease is believed to be closely related to the inter-frictional forces induced by the plasma pre-treatment. Furthermore, higher discharge current or longer treatment time during plasma treatment may lead to a loss in tensile properties due to excessive etching.

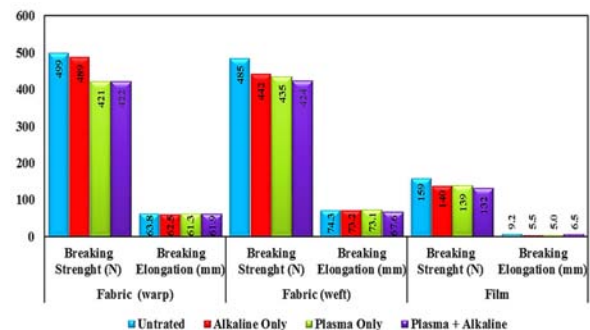


Fig.7 Variations of breaking load and elongation at different levels of fabric weft density, weave structure and of film

DSC studies were performed on PET fabrics and films before and after the plasma pre-treatment on alkaline, in which the heat flow is measured as a function of temperature. The main objective of performing DSC was to investigate the effect of the plasma pre-treatment

on the T_g and T_m . Fig. 8 shows the DSC thermograms of untreated, different plasma pre-treated, and alkaline etched PET fabrics and films. It was found that DSC thermograms of plasma pre-treated PET fabrics and films exhibited no significant changes regarding the melting point in comparison with the untreated PET fabrics and films. In addition, the thermograms do not reveal any significant features that are attributable to the surface modifications. This may be due to the limited penetration depth of the plasma and the short alkaline etching times adopted in the study; thus the obtained results confirm that plasma pre-treatments do not affect the bulk properties of PET surface.

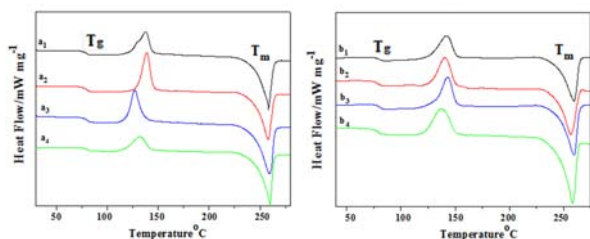


Fig. 8 DSC thermograms respectively of PET fabrics and film. (a1, b1) the untreated, (a2, b2) treated in O_2/Ar plasma, (a3, b3) treated by 4% alkaline at $95^\circ C$, (a4, b4) pre-treated by O_2/Ar plasma and 4% alkaline etching at $95^\circ C$

3.1 Effect of pre-treatment on dyeing properties

PET fabrics and films were treated in two steps. In the first one oxygen/argon plasma was used to endow the surface with moieties of polar groups ($-C=O$, $-COOH$ and $-C-OH$), increase the surface free energy and enhance the fabrics and films wettability. The following expected reactions occurred at the surface of the PET. Oxygen/argon plasma treatment generates polymer chain scissions of the ester bonds by high energy electrons and photons. These chain scissions create a large amount of very reactive chain ends, such as free radicals, which then react easily with the reactive species present in the oxygen/argon plasma such as OH radicals, excited molecular oxygen O_2 and H^+ radicals. At the chain ends, these reactions create some oxidized groups. In the second step, the hydrolysis of PET by alkali metal hydroxides, the hydroxide ions attack the electron-deficient carbonyl carbons, resulting in the production of hydroxyl and carboxyl end groups at the PET surfaces. Further reaction between OH^- ions and $-C-$ breaks the macromolecular chain and produces $-COOH$. Fig. 9 shows the dyeing takes place in a little of the PET surface, directly demonstrating a mechanism of dyeing by single molecules. The simple chemical structure of the dyes often results in bright colors. Dyes with a simple molecular structure can often be prepared with a minimum of contaminating isomers and by-products that tend to dull the color. Inducing a chemical reaction between a fiber, film and an adsorbed dye molecule is another significant way of producing the dyeing of good washing fastness. Dye molecules that

have been adsorbed on the PET surface diffuse into the interior by a relatively simple mechanism, the rate of diffusion of the dye is directly proportional to the concentration gradient at that point through unit area transverse to the direction of diffusion at any point in the surface. As the small amount of dissolved disperse dye diffuses into the surface, additional dye dispersed in solution is dissolved, until the disperse dye is nearly completely exhausted onto the fiber and the film. The adsorbed dye diffusion in PET is considered as a rate process depending on the spontaneous appearance; at a suitable position in the non-crystalline regions of the network of molecular chains, it has a hole large enough to accommodate the dye molecules. The hole includes $-COOH$ and $-C-OH$ groups, which are anionic groups that attract cationic disperse dye.

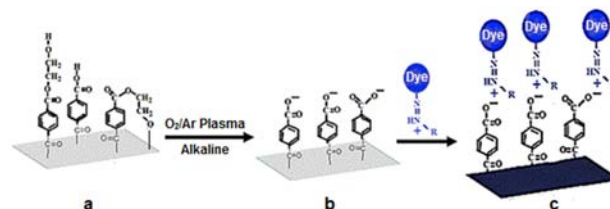


Fig. 9 The mechanism of PET surface (a) untreated (b) pre-treated before dyeing (c) pre-treated after dyeing by blue disperse dye

The use of O_2/Ar plasma treatment on 4% alkaline etching for 5 min at $95^\circ C$ can improve the transport of dye molecules into the structure of PET fabrics and films, which eventually improved dyeability. Fig. 10 shows the K/S value of the PET samples and the dyeing properties of PET fabrics and films exposed to plasma pre-treatment at 0 min and 3 min on alkaline etching at 5 min. As already shown in AFM observation, a rough surface with small edges of a hill, uniformly distributed on the fiber was created by plasma treatment. Also the alkaline treated at 5 min shows a few rises to pits and craters and through plasma pre-treatment for 3 min and 4% alkaline etching for 5 min at $95^\circ C$ shows the broad and smooth wavy surface consequently the RMS roughness value increases with PET fibers; therefore, the rough surface with hydrophilic polar groups leads to the enhancement of the absorbability and deep penetration of the dyestuffs covered around by the surface-active agent with hydrophilic end groups into the fiber surface. The increase of the K/S values is progressive when the treatment time was increased and it is clear that K/S values and color strength increase obviously after plasma only or alkaline treatment together, in the fabric more than in the film, which is dependent on the polymer structure and properties.

Furthermore, the UV-visible absorption spectra used of the dye uptake values for the untreated and the pre-treated samples are shown in Fig. 11. The dye uptake of the pre-treated samples is more than that of the untreated samples. Thus, the dyeing studies showed that the K/S values and the subsequent dye uptake values of the pre-treated samples significantly increased compared with the untreated PET fabrics and films.

These changes in dyeing behavior reflect the changes in the fabrics and films structure that have been affected by O₂/Ar plasma pre-treatment on alkaline etching at 95 °C.

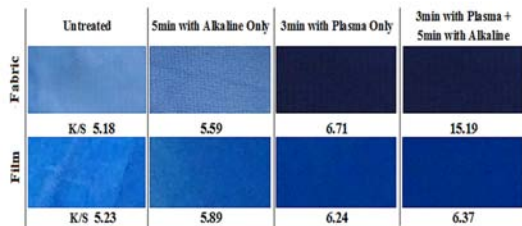


Fig.10 Photographs of dyed PET fabrics and films samples, and *K/S* values with plasma pre-treatment on alkaline etching and dyed at 100 °C for 60 min

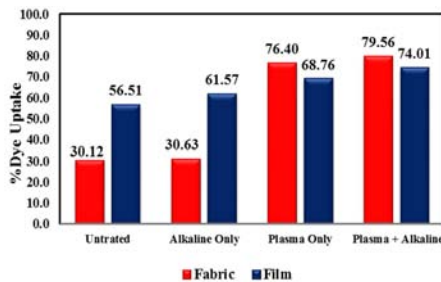


Fig.11 Dye uptake values of PET samples with plasma pre-treatment at 3 min and 5 min alkaline etching and dyed at 100 °C for 60 min

3.2 Fastness properties of dyeing PET with blue disperse dye

The summarized results of O₂/Ar plasma, 4% alkaline at 95 °C pre-treatment dyed PET samples, present the fastness properties to washing for dyed samples that were assessed by the change in shade of the sample fabrics as compared to a standard multifiber fabric. The samples were examined side-by-side adjacent to the standard fabric. The results were almost analogous on both fabrics and films tested for washing and light fastness for both untreated and pre-treatment samples. This result implies that pre-treatment had no effect on the color fastness.

4 Conclusion

Plasma pre-treatment increased the number of active sites on the PET surfaces compared to non-pretreated samples and decreased the detrimental effects caused by alkaline etching. The change in the surface morphology as shown by AFM imaging proved that the plasma pre-treatment increased the roughness of the PET surfaces. DSC results revealed that the melting temperature remained unchanged for both treated and untreated PET fabrics and films. However, the strength at the break and the elongation at the break decreased. This study illustrates a simple and economic method for enhancing the binding efficiency of O₂/Ar plasma pre-treatment on alkaline etching of the PET surfaces. The

dyeability and both washing and light fastness of PET samples were enhanced by pre-treatment and changing the dyeing procedure. The color strength increases of the treated dyed sample are higher than the untreated sample at the maximum wavelength. Although the enhancement of the dyestuff leads to an increase in the color strength, the pre-treatment reactions resulted in the formation of additional C=O and O–C=O groups. Similarly, an increase in the surface energy and the surface roughness has been observed, which correlates with the increase in the exposure treatment time consequently leading to an enhancement in hydrophilicity.

Acknowledgments

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