Effect of plasma and alkaline combination treatment on dyeing properties of PET Fabric and Mechanism Discussion

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ABSTRACT

Investigation of plasma and alkaline combination treatment of polyethylene terephthalate (PET) fabric and its application in dyeing with less energy consuming, less time and pollution, also the dyeing mechanism. It firstly explored the alkaline treatment of PET and textile properties at different conditions of treatments. This paper, studied a low alkaline treatment temperature and concentration were chosen to combine together with plasma treatment at different process. The topographical changes and chemical modification of the fabric surface were analyzed, using X-ray Photoelectron Spectroscopy (XPS). The XPS results showed that the plasma pre-treatment had introduced oxygen containing groups (O–C=O, C–O, and COOH) onto the fabric surface. Dyeability of the modified fabric was investigated by dyeing using disperse blue dye. The obtained results were compared with the high temperature dyeability of the untreated fabric. The result showed that a significant increase of K/S value (8.87) and % dye uptake (71.71) at dyeing temperature of 100 ° C with a combination of plasma and alkaline treatments compared with untreated, only plasma treated and only alkaline treated fabrics at dyeing temperature of 130 ° C. Thus, the oxygen/argon plasma-alkaline combination treatment of PET fabric can get better dyeing properties at low dyeing temperature of 100 ° C than that at 130 ° C, which means a lot of energy saving. Overall, plasma treatment has a great improvement effect with alkaline treatment to improve the dyeing properties of PET fabrics at low temperature.

Keywords: O_2/Ar plasma, surface modification, dyeing, Disperse Dye, PET fabric, XPS.

INTRODUCTION

Polyethylene terephthalate (PET) is the most important synthetic polymer in the world due to its excellent physical and chemical properties, such as high strength, good chemical resistance and thermal stability (Maria, et al., 2015). It is widely used in the textile industry for outdoor, sports and active wear, as well as protective clothing, it is also used for the production of medical textiles, automotive parts, and other numerous technical applications (Zhenhua, et al., 2014; Nasser, et al., 2012; Thomas, et al., 2014). Its main disadvantage is the hydrophobic character that weakens the physiological properties of fabrics and causes difficulties in finishing. Polymer materials with good surface wettability find application in many fields, such as printing, adhesion, dyeing and functional finishing (Nasser, et al., 2012).

Significant work has been done, for development and implementation of new techniques, to improve the wetting, surface roughness and adhesion behavior of PET and other synthetic fibers (Radhia, et al., 2012). For this purpose, many studies have been carried out including: chemical methods (solvent pre-treatment, grafting of different monomers treatment with different reagents, micro-encapsulation techniques and application of supercritical carbon dioxide) (Choon and Hyun 2015; Dan, et al., 2015; Laijiu, et al., 2015). Alkaline treatment of PET is a well-known finishing process for fabrics. It leads to soft handing, silky-luster and improves, wettability (Nabil, et al., 2003; Yeuk, Antonio And Thompson 1996), but alkaline etching at high temperature and high concentrations would cause a significant weight loss in the fiber. The main concern with these treatments are high energy consumption, high cost and environmental issues. Extensive research has been done on the use of alkaline hydrolysis to improve the water absorption properties and softness of the PET fabric. But PET has weak resistance to strong alkaline solution, as can easily break it down into its monomers,

terephthalic acid and ethylene glycol, through a hydrolysis reaction (Satyendra and Goje 2003).

Moreover, physical methods (corona discharge, microwave, ozone-gas, gamma, UV, laser and plasma functionalization) have also been used (Majid, et al., 2013; Bessem, et al., 2012; Rino, et al., 2007; Antonino, et al., 2006). However, some of these methods often damage the otherwise excellent mechanical and unaffected the bulk properties of fibers. In the textile industry, plasma treatment have many advantages over conventional wet chemical treatments. Mainly because it does not involve large quantities of chemicals and water, thus is dry and ecofriendly green process. When a polymer surface is contacted with cold plasma, different concurrent processes may occur at the plasma polymer interface, depending on the chemical and physical characteristics of the plasma (Zeynep and Dilek 2012). It changes the surface geometry, by introducing surface roughness (Qufu, et al., 2007), but lowers the good soft handing and silky-luster. The efficiency of the plasma treatment depends on device parameters and treatment conditions such as; time, pressure, power, frequency, flow rate and gases. The atmospheric pressure plasma treatment may be employed to tailor the surface properties of polymers for specific applications (Akishev, et al., 2013). Therefore, treatment parameters must be optimized to achieve the desirable surface modifications without causing physical degradation and in order to achieve a small ageing effect (Canal, et al., 2004; Takke, et al., 2009).

The main objective of this paper is to combine the plasma pre-treatment and alkaline together to achieve low temperature dyeing of PET through displaying coordination peeling and drilling effects of these two treatments. The study, investigate the effect of O_2/Ar plasma pre-treatment of PET fabrics followed by 4% alkaline etching at 85 °C, to create functional groups. The mechanism of dyeing will be discussed and the surface morphology and chemical structure of PET fabrics will be evaluated by: X-ray photoelectron spectroscopy (XPS) analysis. UV–visible spectrometer and Color measurement

spectrophotometer for determination of dye uptake and K/S value were carried out respectively. The washing and light fastness test were also carried out.

METERIALS AND METHODS

Materials:

The polyethylene terephthalate (PET) textile material was nominated to be used for this study with typical 1/1 plain woven fabric and weight 86.1 g/m^2. The fabrics were boiled in water twice to remove contaminations, the cleaned samples were chopped into the dimension of 6 x 6 cm^2 and then weighted. Oxygen and argon (purity 99.99%) were obtained from Shanghai Cheng Gong Gas Industry Co., Ltd.

Alkaline (sodium hydroxide) 99% from (Xin Jiang Tian Cheng Corporation Limited, China). At optimum conditions were realized using concentration of alkaline solution 4% w/w for 5 min. The liquor to fabric ratio was 50:1. At the end of the designated time the fabrics were washed for 15 min and followed by drying at 100 °C for 30 min.

Therefore, C.I. Disperse Blue 183, with λ_{max} 530 nm from Zhejiang Longsheng Group Co., Ltd, (Lonsen), was used as received and its structure is shown in Figure 1. All the experiments were carried out using distilled water.

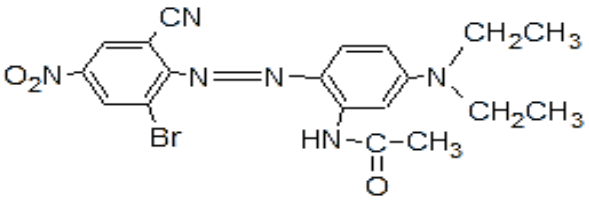

Figure 1. Structure of the Blue dye (C.I. Disperse Blue 183).

Methods:

A dielectric barrier discharge (DBD) plasma system used for the surface treatment in this study is shown in Figure 2 (a) where the discharge is held between two parallel copper electrodes with

diameter of 60 mm and covered with a very thin Al_2O_3 ceramics plate and the electrode gap is 3 mm. Also it is shown in Figure 2 (b) are discharge voltage and current waveforms, in which the discharge current displays the glow-like regular multiple pulse discharge peaks. The generated discharge characterization AC power in the frequency of about 20 kHz is applied between the upper and grounded electrode. The flow rate ratio of O_2/Ar was 10:1. Pumping the chamber and continue filling of the mixed gases until to the atmospheric pressure. The discharge started and treated the fabrics for a certain duration at treatment time for 3 min.

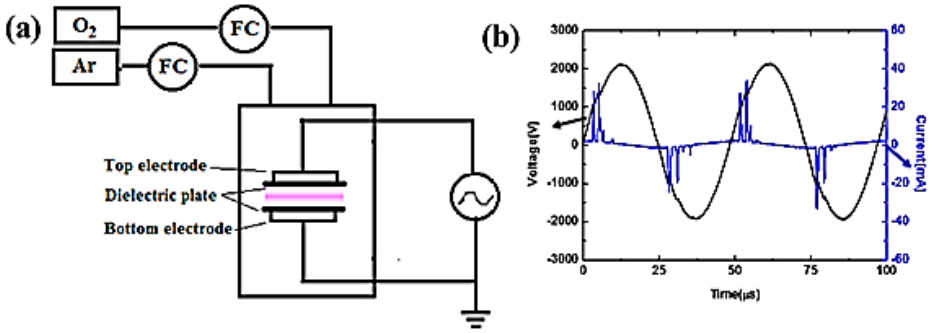

Figure 2. Experimental setup of (a) the DBD discharge plasma system; (b) I-V curve of a typical discharge voltage and current waveforms at 20 kHz.

Dyeing procedure:

Dyeing procedure was carried out in a dye bath containing 2% o.w.f shade in distilled water without any auxiliaries and the liquor ratio of the disperse dye was 1:50. To decide the period of dyeing, fabrics were immersed in the dye bath at room temperature and the temperature was increased up from the room temperature to 90 °C and 100 °C with a gradient of 2 °C/min which was followed by increasing of the temperature up and held constant to 100 °C and 130 °C respectively, with a gradient of 1 °C/min for 60 min, and cooled down to 60 °C with a gradient of 4 °C/min. The dyeing profile is shown in

Figure 3. At the end of the dyeing process, the dyed samples were rinsed with water, fully washed and dried in air.

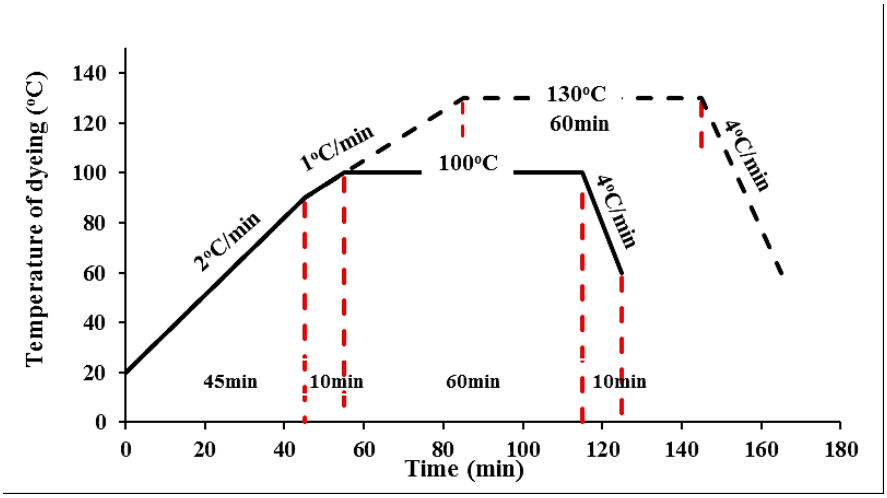

Figure 3. Profile of dyeing

Fabric surface characterization:

Surface chemical composition analysis (XPS):

The surface chemical structures are analyzed by X-ray photoelectron spectroscopy (XPS) with a Kratos Axis Ultra spectrometer (Kratos Analytical-A Shimadzu group company) using a monochromatic X-ray source power of 75~150W for acquiring a survey spectrum and narrow scan spectra. The base pressure in the sample chamber was controlled at less than 5×10^{-9} Torr. Photo emitted electrons were collected at a take-off angle of 90°.The BE scale was calibrated according to the C1s peak (284.8 eV) of adventitious carbon on the analyzed sample surface. De-convolution analysis of C1s peaks was carried out using an XPS Peak analysis software.

Dyeability analysis:

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 measurements of the average of four reflectance values, taken at

different positions on the dyed fabric, from (360-700 nm). The reflectance at the wavelength of maximum absorption λ_{max} was used to calculate the color yield of the dyed fabric by applying the Kubelka-Munk equation (2) (Dan, et al., 2015).

Determination of dye uptake:

To determine the amount of dye uptake, the fabric samples were removed from the dye bath; the dye solution was put into a 50 ml flask and 5 ml was used for every time measurement, the wavelength at the maximum absorbance (530 nm) for the dye (C.I. Disperse Blue 183) was applied to measure the absorbance of the dye solution.

Color fastness properties:

Color fastness was evaluated according to the respective international standards ISO 105-F10 for color fastness to washing, using a standard SDC DW multifiber fabric as the adjacent fabric (worsted wool, acrylic, spun polyester, spun polyamide, bleached cotton, and acetate) which was stitched to dyed PET fabric (all fabric pieces had the same diameter) and then washed using 5 g/L of the nonionic detergent and 2 g/L of sodium carbonate anhydrous at 60 °C for 30 min. Also color fastness to light was evaluated according to the respective international standards ISO 105-B02. Exposure time 40 h was applied on samples until remarkable color degradation was observed by using the Blue Wool standards.

RESULTS AND DISCUSSION

Composition changes by XPS analysis:

C1s and O1s deconvolution analysis was performed as shown in Figure 4 (a) and (b). The carbon content decreases and oxygen content increases after plasma, alkaline and plasma-alkaline combination treatments, in which combination treatment has the most obvious effect in increasing oxygen content. The ratio of oxygen content over the carbon content increases from 21.93% of untreated to 30.13% of O_2/Ar plasma pre-treatment followed by alkaline etching of PET surface. C1s deconvolution analysis was performed as shown in

Figure 4 (a_1–a_4). The C1s spectra of the untreated and treated PET structure have three kinds of sub-peaks: the peak at 284.7 eV (aromatic ring), which corresponds to C-C/C-H groups, while the peaks at 286.1 eV (methylene carbons singly bonded to oxygen atom) and 288.7 eV (carbon atoms in ester group) represent the C–O (and/or C–OH) and O–C=O (and/or COOH) groups respectively (KUBELKA, 1954).

In addition, the shake-up peak resulting from the π - π* shake–satellite peak was fitted at binding energy of (290.9 eV), attributed to the opening of the benzene ring in PET surface at low concentrations (1.54%) of treated sample for 5 min alkaline and (1.94%) of treated sample for 3 min O_2/Ar plasma + 5 min alkaline etching (Lin and Chen, 2006).

Figure 4 (b), (b_1-b_4) shows the XPS around the O1s energy region. The two O1s peaks can both be fitted with two main peaks at energies of 531.5 eV and 533.3 eV. The peak at 531.5 eV corresponds to oxygen atoms doubly attached to carbon in the form of COO groups, while the peak at 533.3 eV corresponds to the oxygen atoms singly attached to carbon. The increased intensity ratio between the peak at 533.3 eV and at 531.5 eV showed the increase of the oxygen atoms singly attached to carbon such as C-OH. The C1s and O1s spectra both demonstrate that the fiber surface is oxidized and many COOH and COH groups are generated. Treatment of the surface and creating the chain scission by the bombardment of the active species has led to decrease of C–C/C–H signal. However the C-O and O-C=O bonds had remarkable increase. This means that the treatment provides active species with sufficient energy to cause chain scission, and active oxygen components provide oxidation of C–C/C–H groups rather than ester groups. The increase of these polar C-O and O-C=O groups are responsible for the high hydrophilicity of PET fabrics after treatment (Kale and Desai, 2011). The polar groups generated on the fiber surface are the main cause of the improvement of hydrophilicity and enhance the dyeability of the PET fabrics as will be displayed in the surface wettability (Water Contact Angle) and the effect of pre-treatment on dyeing properties.

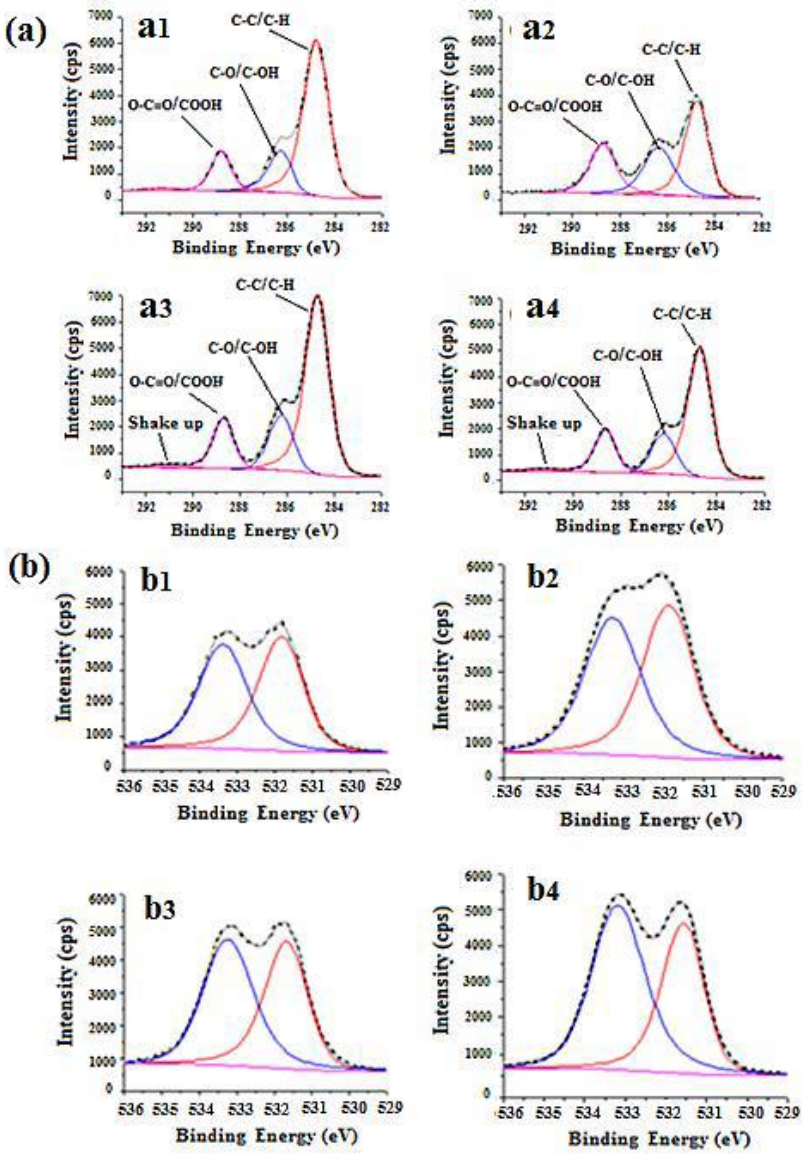

Figure 4. Deconvoluted XPS (a) C1s core level spectra, (b) O1s core level spectra, original (dotted line) and fitted (solid line) of PET fabrics: (a_1, b_1) untreated; (a_2, b_2) O_2/Ar plasma treated for 3 min; (a_3, b_3) alkaline treated for 5 min; (a_4, b_4) 3 min O_2/Ar plasma + 5 min alkaline etching

Effect and mechanism of plasma and alkaline combination treatment on dyeing properties:

As displayed in Figure 5 is an explanation about the effect and mechanism of plasma, alkaline and plasma-alkaline combination treatment on the dyeing of PET fabric. From the step of a_1 to b, oxygen/argon plasma treatment leads to the polymer chain scissions of the ester bonds by high-energy electrons, ions and photons, especially on touch with the electron avalanche in the fine filament channel. Besides creating a rough surface like Figure 3 the SEM (a_2) and/or Figure 4 the AFM (a_2), (Amel and Zhang, 2018), 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^+ radical and creates some oxidized polar anionic groups like -C=O, -COOH, -C-OH (Szili, et al., 2011).

From the step of b to c as in Figure 5 when etching 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 fabric surfaces. More ester chain scission of PET occurs, resulting in a considerable weight loss and the formation of hydroxyl and carboxylate end groups as in Figure 5 (d_1), Figure 3 (a_4) and/or Figure 4 (a_4), (Amel and Zhang, 2018). The rough and hydrophilic surface with anionic groups becomes attractive to the cationic dye molecule micelles covered by surface active agent molecules with hydrophilic end groups outward and dispersed in water. According to this, the absorption and diffusion of the dyestuff micelles along the fiber surface are all improved. More dyestuff molecules get into the rough surface holes having carboxyl or hydroxyl groups and form hydrogen bonds and retain on the fiber surface. Compared with step a_2 to $d_2$in Figure 5, the rough surface after plasma pre-treated followed by alkaline has much more micro/nano holes and a lot of -COOH and -C-OH anionic groups and easily attracts more cationic disperse dyes. Deep coloration effects are obtained.

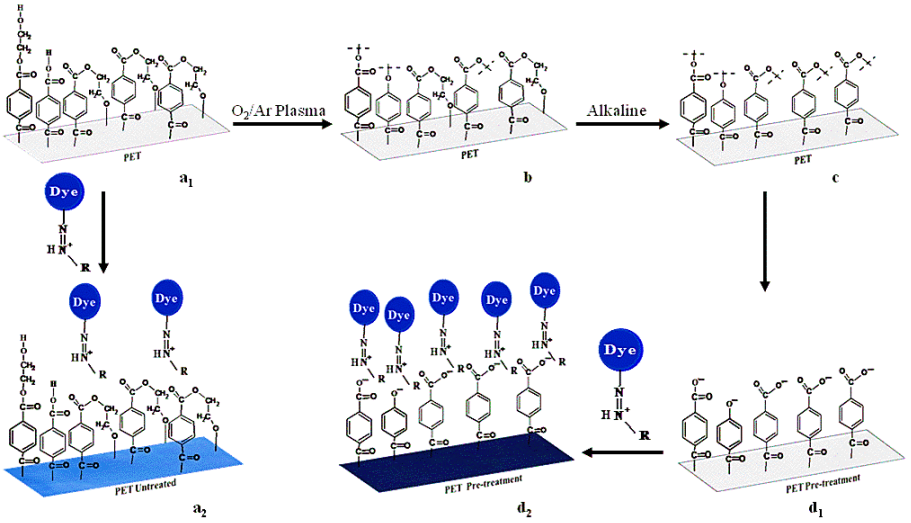

Figure 5. The mechanism of (a_1) untreated (b) treated O_2/Ar plasma for 3 min (c) treated alkaline for 5 min (d_1) plasma pre-treatment for 3 min followed by a 5 min alkaline etching (d_2) pre-treatment after dye (a_2) untreated after dye with blue disperse dye.

Figure 6 shows the K/S value of the PET samples of untreated, treated sample exposed to O_2/Ar plasma for 3 min, 4% alkaline etching for 5 min at 85 °C and plasma pre-treatment for 3 min followed by a 5 min 4% alkaline etching at 85 °C respectively. The dyeing temperature is 100 °C which is lower than the usual disperse dye bath and 130 °C. As we know through the results of XPS, the polar groups generated on the fiber surface are the main cause of the improvement of hydrophilicity and enhance the dyeability of the PET fabrics, as displayed in the paragraphs in the surface wettability water contact angle figure 5 as shown in (Amel and Zhang, 2018), can improve the transport of dye molecules into the structure of PET fabrics which eventually improves dyeability.

As already shown in (Amel and Zhang, 2018). The 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 fabric at 5 min shows a few rises to pits and craters and through plasma pre-treatment at 3 min and 4% alkaline etching for 5 min at 85 ^{o} C shows the broad and rough 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 effect of the dyeing temperature on the dyeability was investigated, as shown in Figure 6. It is obvious that K/S values increased with the increase in the dyeing temperature for both untreated and oxygen/argon plasma treated or plasma pre-treated followed by alkaline etching fabrics, with a more pronounced increase in the oxygen/argon plasma treated or plasma pre-treated followed by alkaline etching fabrics than in the untreated fabrics. The adsorption and diffusion of disperse dye on PET are greatly influenced by temperature as an increase in temperature increases the mobility of the polymer chains in the amorphous regions of the fabric. However, after plasma pre-treated followed by alkaline etching, higher K/S value can be obtained than that of untreated, only plasma treatment or only alkaline treatment. That means diffusion of the dyestuff (and therefore the increased rate of dyeing) has been improved because of the surface roughness and hydrophilicity improvement by reducing cohesion between polymer chains and increasing the kinetic energy of the dye molecules for treated fabrics.

Thus, the oxygen/argon plasma treatment and pre-treatment of PET fabrics can obtain the same or better dyeing properties at a lower dyeing temperature of 100 °C than those of untreated fabric at the dyeing temperature of 130 °C, which means a lot of energy saving. For better K/S values of the fabrics, the 100 °C dyeing temperature was selected for subsequent experiments in this study.

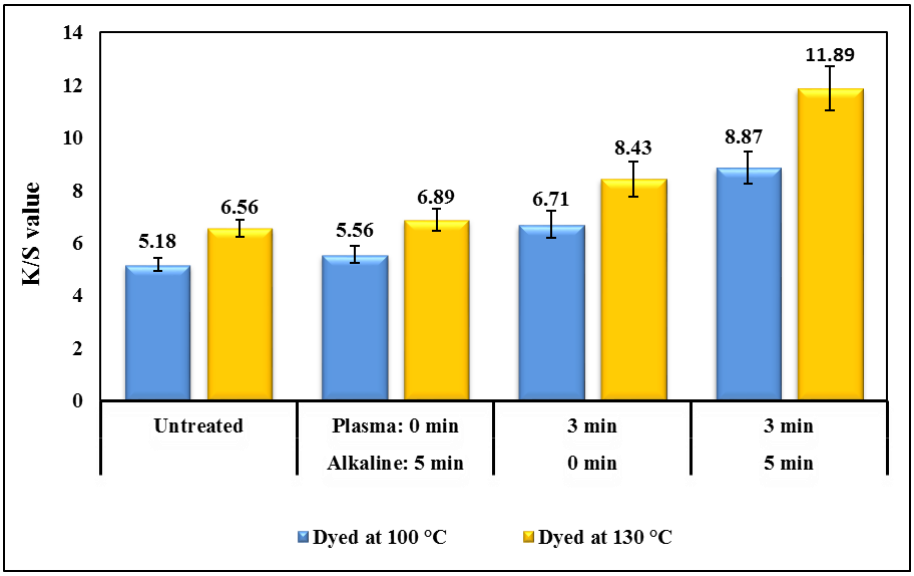

Figure 6. K/S values of different treatment and dyeing processes

UV–visible absorption spectrum for the dye uptake values of the untreated, treated samples exposed to O_2/Ar plasma for 3 min, 4% alkaline etching for 5 min at 85 °C and plasma pre-treatment for 3 min followed by 4% alkaline etching for 5 min at 85 °C respectively, are shown in Figure 7. 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 fabric. These changes in dyeing behavior reflect the changes in the fabric structure that have been affected by O_2/Ar plasma pre-treatment on alkaline etching at lower dyeing temperature of 100 °C. The dyeability and the dyeing properties of PET fabrics are shown in Figure 8.

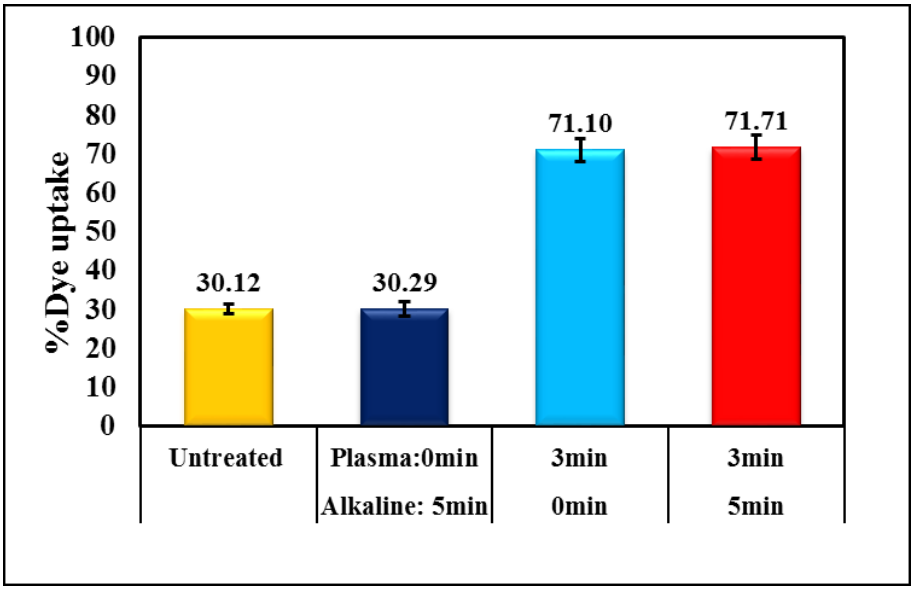

Figure 7. % Dye uptake of PET untreated and treated samples with alkaline for 5 min, O_2/Ar plasma 3 min and 3 min plasma pre-treatment + 5 min alkaline etching dyed at 100 °C for 60 min.

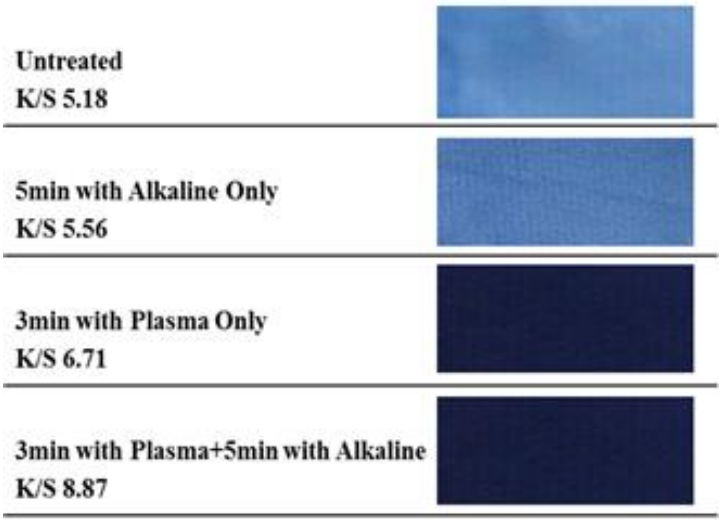

Figure 8. Photographs of dyed PET fabric samples.

Fastness properties of dyeing PET with blue disperse dye:

Table 1 shows a summary of the washing and light fastness of dyed PET fabrics through standard measurement methods. It is shown that the untreated, treated and pre-treated dyed fabrics have almost the same levels of light fastness and washing fastness although some of the O_2/Ar plasma treated for 3 min and plasma pre-treatment for 3 min on 5 min alkaline etching fabrics showed a little bit higher level of light fastness. This indicates that plasma pre-treatment has no obvious effects on the color fastness for its surface effects.

Table 1. Color fastness of PET fabrics with different treatment and dyeing processes.

Treatment time	Washing fastness						Light fastness
	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	
Untreated	5	4 - 5	4	4 - 5	4	4 - 5	2 - 3
Treated 3min O_2 /Ar Plasma	5	4 - 5	4	5	4 - 5	4 - 5	> 5
Treated 5min Alkaline	4 - 5	5	5	5	5	5	4
Treated 3min O_2/Ar Plasma + 5minAlkaline	4	5	4 - 5	4 - 5	5	5	> 5

Table 1: illustrate the Washing fastness: a staining on (wool), (acrylic), (polyester), (nylon), (cotton), (acetate): 5-Excellent, 4-Very good, 3-Good, 2-Fair, 1-Poor.

Light fastness: 8-Maximum, 7-Excellent, 6-Very good, 5-Good, 4-Fairly Good, 3-Moderate, 2-Slight, 1-Poor.

CONCLUSION

This paper investigated the modification and dyeing of PET fabrics by atmospheric oxygen/argon plasma pre-treatment followed by 4% alkaline etching at 85 °C. XPS analysis revealed the introduction of polar oxygen groups and the ratio of oxygen and carbon content is the highest on the PET surface after combination treatment.

Based on the above findings and the references in the literature, oxygen polar groups with rough surfaces after combination treatment over the PET surface impart wettability to the fabric. 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.

The effect and mechanism of the plasma and alkaline combination treatment is discussed. It is reasonably believed that the rough and hydrophilic surface with anionic groups after combination treatment becomes attractive to the cationic dye molecule micelles covered by surface active agent molecules with hydrophilic end groups outward and dispersed in water. More dyestuff molecules get into the rough surface holes having carboxyl or hydroxyl groups and form hydrogen bonds and retain on the fiber surface. Thus, better K/S values could be obtained at a lower dyeing temperature of 100 °C in the combination treatment of fabric than at high dyeing temperatures of 130 °C for the untreated or only plasma or only alkaline treated fabric. This means that the oxygen/argon plasma pre-treated fabric followed by alkaline etching can reduce the dyeing temperature from 130 °C to 100 °C, with better dyeing properties and have great potential for energy saving in PET fabric dyeing process.

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