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Improving the PET Fabrics Surface using O_2/Ar Plasma and Alkaline Treatments at Low Temperature

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ABSTRACT

Low energy consumption and resource saving have been increasingly adopted in industries to combat resource depletion and to control pollution in the recently. In textile industries the wet chemical processing of textile materials through is adoption of plasma etching. This paper, studied the effect of plasma pre-treatment on alkaline etching on polyethylene terephthalate (PET) fabrics, based on the weight loss after plasma treatment using three different gases argon, oxygen, and oxygen/argon mixture (Ar, O_2, and O_2/Ar). It is found that O_2/Ar plasma treatment and low temperature alkaline treatment gave optimal surface roughness properties of PET fabric. The topographical changes and chemical modification of the fiber surface were analysed using Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM). Then a low alkaline treatment temperature at 85 ° C and 4% concentration was chosen to combine together with plasma treatment at different processes. Also the surface wetting properties were calculated depending on contact angle (cos θ). Water drop spread rapidly and the contact angle was zero on the PET fabric surface by combination treatment and the surface tension between solid-liquid interfacial tensions were 72.72 mJ/m^2 which is equal to the liquid surface tension. It is recommended to study the dyeing properties after improving the surface of PET fabric using plasma/alkaline treatment.

Keywords: O_2/Ar plasma, surface modification, alkaline treatment, PET fabric.

INTRODUCTION

Alkaline hydrolysis is a popular method applied in textile industry to improve various physical and chemical properties of synthetic fibers, especially for Polyethylene Terephthalate (PET) fabric such as hydrophilicity, hand feeling, moisture regain etc. The alkaline hydrolysis of PET fabrics and fibers are usually carried out with an aqueous alkaline solution, such as sodium hydroxide. In the alkaline hydrolysis process, PET undergoes a nucleophilic substitution. Chain scission of PET occurs, resulting in a considerable weight loss and the formation of hydroxyl and carboxyl end groups, which improve the handling, moisture absorption of the fabric with enhanced softness **(Nabil A I et al, 2003) and (Yeuk-Lam H et al,1996).**

Alkaline hydrolysis, however, are being accompanied by weight loss and decreases the tensile strength of the PET fabric. Increase in concentration, duration of exposure and temperature increase the rate of hydrolysis of PET. The higher treatment temperature deteriorates the effect of alkali on PET fabric surface. **(Satyendra M and A S Goje, 2003)**. (**Robin Ng et al, 2009)** reported that treating PET fabric with an aqueous solution of sodium hydroxide decreases the weight of the fabric and its tensile strength. Increase in concentration of sodium hydroxide, and

temperature increases the weight loss of the treated fabric. The big problem caused by alkaline hydrolysis is that the small hydrolysis fragment from the polymer chain cannot be bio-degraded for a long time and can cause great environmental pollution.

Several researchers have studied the kinetics of alkaline hydrolysis of polyester fabric. It was found that hydrolysis took place only on the surface of the fabric as peeling off the outer layer of the fiber and the rate controlling slow step was the attack of water molecules in the ionized form of the ester linkage. **(Nikola. K. et al, 1990)** showed that alkaline hydrolysis of PET at 100 °C is a first order reaction with respect to each of the surface and hydroxyl ions. **(Natalia. P et al, 2014)** hypothesized that weak surface hydrolysis of PET material generate additional hydroxyl and carboxyl groups on the surface. They have reported that treatment of PET in 0.0125–1.5 mol/l, alkaline solution at the temperature for 15-20 minutes is the optimum condition for obtaining free functional groups.

Based on the discharge mode of atmospheric glow-like plasma with very fine filament discharge channel as shown by **(Amel. E. et al, 2016)**, a rough surface with nano and micro etching pits was formed. These fine filaments discharge behave more like a drilling head with high energy species. In this study, the surface morphology was discussed of PET fabrics and have been evaluated by: scanning electron microscopy (SEM), and atomic force microscopy (AFM), weight loss measurement, contact angle measurements and surface tension measurements.

METERIALS AND METHODS

Materials:

The polyethylene terephthalate (PET) textile material was used for this study with typical 1/1 plain woven with the fabric density 86.1 g/m^2, thickness 0.51 mm and fiber diameter 11.7 µm The fabrics were boiled in water twice to remove contaminations. The cleaned samples were chopped into the dimension of 6 cm x 6 cm and then weighed. Used argon, oxygen, and oxygen/argon mixture (purity 99.99%) were bought from Shanghai Cheng Gong Gas Industry Co.,Ltd.

Alkaline (sodium hydroxide) purity is 99% from (Xin Jiang Tian Cheng Corporation Limited, China). The samples were treated with 4% w/w alkaline solution. All the experiments were carried out using distilled water.

Methods:

The samples were treated by a dielectric barrier discharge (DBD) for 3 min, which is the optimum time used for the surface treatment in this study as shown by **(Amel. E. et al, 2016)** Figure 1 (a). The DBD plasma is generated inside a reaction chamber, where the discharge is held between two parallel copper electrodes with a diameter of 60 mm and the dielectric very thin Al_2O_3 was placed between the two electrodes with a thickness of 1 mm ceramic plate and the dielectric gap is 3 mm. Treatment of the fabric surfaces was determined and the samples were treated under these plasma conditions. The AC power in the frequency of about 20 kHz was used at a power of 75 w and at a pressure of 10^2 KPa. The gas flow rate was (O_2 = 50 ml/min, Ar = 0.5 l/min). The chamber was first pumped and then filled with the mixed gases up to the atmospheric pressure. After that, the discharge was started and the fabrics were treated for a certain duration of treatment time.

Alkaline Etching of PET Fabric at Low Temperature:

The concentration of alkaline solution used was 4% w/w. The liquor to fabric ratio was 50:1. The process of alkaline etching was as follows: the solution of alkaline was first heated from room

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temperature to 40 °C with a gradient of 1.5 °C/min, which followed by increasing of the temperature up and held constant to 85 °C for 5 min and cooled down to 40 °C with a gradient of 1 °C/min, the alkaline profile is shown in **Figure 1**.

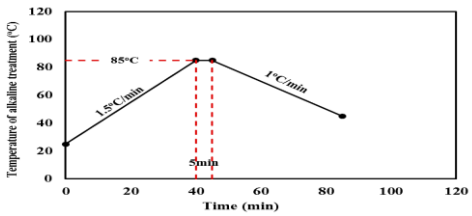

Figure 1: Profile of alkaline treatment

At the end of the designated time, the fabrics were removed and rinsed using distilled water for several times, then washed for 15 min and followed by drying process in oven at temperature of 100 °C for 30 min. Each of the treated fabric was measured on the weight in order to determine if significant damage occurred or not during treatment.

Fabric Surface Characterization

Surface Morphology Analysis:

The surface morphology of PET samples was observed by JSM-5600LV (JEOL Ltd.) scanning electron microscope (SEM).The samples were coated with a thin gold layer of less than 100 A° to prevent charging on the surface of the fabrics for SEM observation directly after plasma treatment.

Atomic Force Microscopy:

Atomic force microscopy (AFM) (Multimode NanoscopeIIIa, Digital Instrument, USA) was used to examine the surface morphology of the PET sample before and after the plasma pre-treatment. The scanning mode used was a tapping mode, the scanning range was set at a size of 2.0 µm×2.0 µm of scan rate 1.001 Hz and data scale 200.0 nm. The roughness of PET sample were analyzed using a computer. All the samples were scanned at room temperature in the atmosphere. Two parameters, such as the mean square root of roughness (RMS) and the average roughness (Ra) were calculated from the following equations (1) and (2) **(Lin Y. S and C. L Chen 2006):**

$$\mathbf{RMS} = \sqrt{\frac{\sum_{n=1}^{N}(Z_n - \dot{Z})}{N-1}} \qquad (1)$$

$$\mathbf{Ra} = \sum_{n=1}^{N} \frac{|Z_n - \dot{Z}|}{N} \qquad (2)$$

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Where RMS represents the standard deviation of the roughness from the mean height, and Ra represents the mean deviation of the roughness from the mean height. N is the number of data points in the image, n and ` are the pixel locations on the AFM image, Zn is the height value of n locations, Z` is the height value.

Weight Loss:

The weight percentage changes were calculated from the weight change in the fabrics after the treatment and the weight loss (WL) was calculated using the following formula **(Afra. H, et al 2010)**:

$$\%\mathbf{WL} = \frac{\mathbf{W_1} - \mathbf{W_2}}{\mathbf{W_1}} \times \mathbf{100} \qquad (\mathbf{3})$$

Where: $\mathbf{W_1}$ and $\mathbf{W_2}$ are the weight of the samples before and after treatments respectively.

Contact Angle:

The contact angle measurements of individual PET fabrics were performed using 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. A 25 mm × 25 mm PET specimens were cut. The measurement of water drop was taken immediately after placing the water drop on the surface.

Surface Tension and Contact Angle – Young's Equation:

Consider a liquid drop resting on a flat, horizontal solid surface (PET) in Figure 5. The contact angle is defined as the angle formed by the intersection of the liquid-solid interface and the liquid-vapor interface (geometrically acquired by applying a tangent line from the contact point along the liquid-vapor interface in the droplet profile). The interface where solid, liquid, and vapor co-exist is referred to as the "three phase contact line".

Ideally, the shape of a liquid droplet is determined by the surface tension of the liquid. In a pure liquid, each molecule in the bulk is pulled equally in every direction by neighboring liquid molecules, resulting in a net force of zero. However, the molecules exposed at the surface do not have neighboring molecules in all directions to provide a balanced net force. Creating an internal pressure. As a result, the liquid voluntarily contracts its surface area to maintain the lowest surface free.

Surface tension is caused by the unbalanced forces of liquid molecules at the surface energy. From everyday life, it is know that small droplets are spherical, which give the minimum surface area for a fixed volume. The spherical shape of the liquid drop is changed to spread across the surface of a solid and wet the surface. Work is needed in order to change the drop shape and also reshape it. This intermolecular force to contract the surface or the work per unit area is called surface tension (γ). Since it is not possible to measure the surface tension of a solid directly, the contact angle is used as an indirect approach for this purpose. The contact angle (θ) between a drop of liquid and the surface of a solid corresponds to the surface tensions of the three interfaces as the Young's equation. Surface tension is usually expressed as dynes/cm or it is given in SI units of mN/m or mJ/m^2 **(Jan K. S and A. W. Neumann, 1987).** In practice, external forces such as gravity deform the droplet; consequently, the contact angle is determined by a combination of surface tension and external forces. Theoretically, the contact angle is expected to be a characteristic of a given solid-liquid system in a specific environment. As first described

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by Thomas Young **(Jacco H. Snoeijer1 and Bruno Andreotti, 2008)**. in 1805, the contact angle of a liquid drop on an ideal solid surface is defined by the mechanical equilibrium of the drop under the action of three interfacial tensions is calculated from the following equations:

$$\gamma_{lv} \cos\theta = \gamma_{sv} - \gamma_{sl} \qquad (4)$$

Where γ_{lv} , γ_{sv} , and γ_{sl} represent the liquid-vapor, solid-vapor, and solid-liquid interfacial tensions, respectively and θ is the contact angle.
The calculation of solid surface tension, γ_{sv} , from the contact angle, θ of a liquid of surface tension γ_{lv} , starts with the Young's equation:

$$\gamma_{sl} = \gamma_{sv} - \gamma_{lv} \cos\theta \qquad (5)$$

Thus, in order to determine γsv, further information is necessary. Consequently, one obvious approach is to seek one more relation between the parameters in the equation **(6)**, such as an equation of state.

$$\gamma_{sl} = f(\gamma_{lv}, \gamma_{sv}) \quad u \qquad (6)$$

The simultaneous solution of equations 6 and 7 would solve the problem. Note that if the commonly used assumption of negligible liquid vapor adsorption is applied, then equations (6) and (7) may be written in terms of γ_l and γ_s, rather than of γ_{lv}. An old equation of state of solid-liquid interfacial tensions is due to Rayleigh and later **(Robert. J. G, 1977).**

$$\gamma_{sl} = \gamma_s + \gamma_l - 2(\gamma_s \gamma_l)^{1/2} \qquad (7)$$

Whereγ_s is the solid surface tension (equal to γ_{sv} if adsorption is neglected) and γ_l (or equivalently γ_{lv}) is the surface tension of the liquid. Combining equations (7) with (5) to give (8) **(Jan K. S and A. W. Neumann, 1987)**.

$$\gamma_s = \frac{1}{4}\gamma_l(1 + \cos\theta)^2 \qquad (8)$$

RESULTS AND DISCUSSIONS

Weight Loss:

Figure 2 shows the weight loss of PET fabrics treated with plasma, initiated in various gases of Ar, O_2, and O_2/Ar mixture plasma pre-treatment on 4% alkaline etching at 85 °C, that can be directly weighed ensures that the weight loss measurement is correct before and after treatment. 5 min alkaline treatment showed the smallest weight loss value, which means less degradation for short treatment time and less roughening of the surface. However, the weight loss of PET fabrics treated with three different gases (Ar, O_2, and O_2/Ar mixture) plasma treatment for 3 min was found to be higher compared to 5 min alkaline treatment. The weight loss of PET fabric treated with plasma pre-treatment on alkaline etching treatment was more than the total amount of the fabrics by the two treatments separately, which shows the synergistic effect of the surface.

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According to the statistical evaluations the best results are obtained by O_2/Ar gases with respect to other gas types.

With respect to weight loss, O_2/Ar gas treatment was selected for all the analyzed samples.

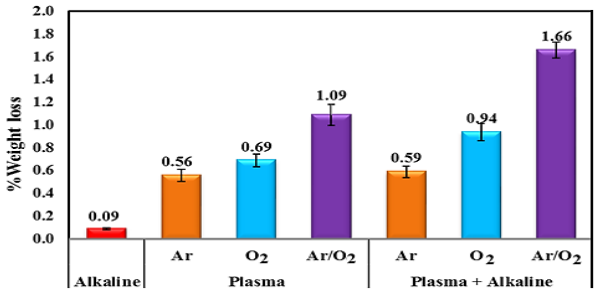

Figure 2: Weight loss (%) of PET fabrics with 5 min alkaline only, 3 min plasma take-up versus gases and 3 min Plasma pre-treatment on 4% alkaline etching for 5 min at 85 °C.

Scanning Electron Microscope:

Figure 3 shows the typical SEM images of the surface morphology of the PET fabrics. As shown in Figure 3 (a $_1$), the untreated fabric has smooth fiber surfaces occasionally distributed with micro size dust particles. However, 3 min O_2/Ar plasma treated PET fabric has rough fiber surfaces uniformly distributed with micro size protrusion dots and depressed pits as shown in Figure 3 (a $_2$). These results are due to the physical and chemical etching effect to the fabric surface molecules caused by active particles such as ions and radicals in the oxygen/argon plasma onto the fabric surfaces.

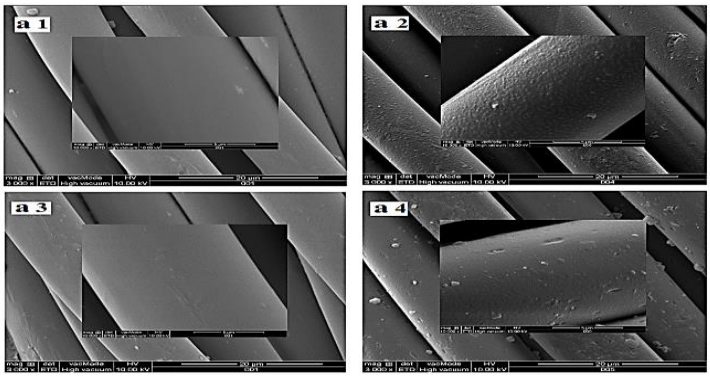

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Figure 3: SEM images of PET fabrics (a $_1$) untreated; (a $_2$) treated for 3 min O_2/Ar plasma; (a $_3$) treated for 5 min by 4% alkaline at 85 °C; (a $_4$) 3 min O_2/Ar plasma pre-treated and 4% alkaline etching for 5 min at 85 °C

PET fabric surface in Figure 3 (a $_3$) with 4% alkaline treatment for 5 min at 85 °C did not cause notable changes in the surface topography of the PET fabric for a short treatment time. In Figure 3 (a $_4$) O_2/Ar plasma pre-treatment for 3 min followed by 4% alkaline etching for 5 min at 85 °C shows that the pre-treatment was sufficient to change the PET surface to be more hydrophilic and significant roughness was created on the fiber surfaces by some etching on the fiber surfaces. The protrusion dots become larger and more obvious because of the removing of the adhering broken PET fragments during alkaline treatment, and the sample appears soft.

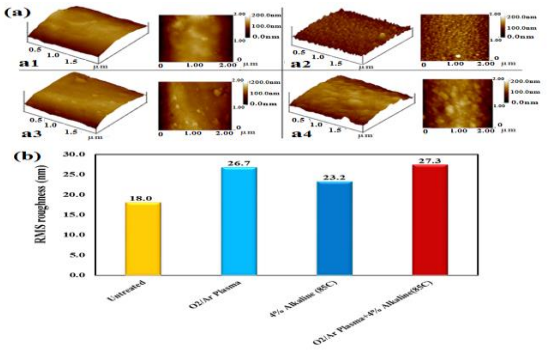

Figure 4: (a) AFM images of PET (a_1) untreated; (a_2) treated for 3 min O_2/Ar plasma; (a_3) 5 min alkaline; (a_4) 3 min plasma pre-treated and 5 min alkaline; (b) RMS roughness

Atomic Force Microscopy:

AFM analysis gave similar information with SEM results on the plasma induced surface chemical and topographical modifications.

Figure 4 (a) shows AFM images of PET fiber surface before and after O_2/Ar plasma and alkaline treatments. As shown in Figure 4 (a_1) and (a_2), the untreated sample shows relatively flat surface and the O_2/Ar plasma treatment for 3 min shows small edges of the hill, uniformly distributed on the fabric surface respectively. Figure 4 (a_3) shows PET fiber treated with an alkaline solution for 5 min displaying a few rise to the pits and craters. While the sample in Figure 4 (a_4) after 3 min O_2/Ar plasma pre-treatment followed by 5 min 4% alkaline etching at 85 °C, gave a broad and rough wavy surface due to alkali and plasma combination treatment. The number of ridges on the surfaces is influenced by O_2/Ar plasma pre-treatment and alkaline etching.

Moreover, Figure 4 (b) shows the root mean square roughness (RMS) of the untreated and treated PET fabrics. RMS was increased in the treated PET fabric for 3 min O_2/Ar plasma

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treatment when compared with the 5 min alkaline only.RMS by 3 min O_2/Ar plasma pre-treatment followed by 5 min alkaline etching was the most **(Navaneetha K. P, 2009) and (Ruggero. B, 2005)**.

Contact Angle:

Figure 5 shows the surface wettability (Water Contact Angle). Measurement of the contact angle was difficult because the water droplets spread immediately after being deposited on the treated surface of the PET fabric. However it was done by putting the dye solution drops on different areas of each sample as described by **(Amel. E et al, 2016)**.

To compare the wettability of plasma and alkaline treated PET fabrics, their respective contact angles were measured. Figure 5 shows the contact angle as a function of the treatment process. In the untreated sample water droplet does not entirely spread and showed a static water contact angle (< 90°). When a water droplet was placed on the pre-treated or alkaline samples, it spread almost completely, indicating that the fabrics is wetted by water easily after plasma or alkaline treatment. When a water droplet was placed on a combination of plasma pre-treatment followed by alkaline etching, it spreads completely, indicating very good hydrophilicity. The contact angle of treated PET fabrics increased with plasma pre-treatment for 3 min followed by 4% alkaline etching for 5 min at 85 °C increased more than a summary of contact angles treated separately by the two methods, which show coordination effects and this is attributed to an increase in the surface free energy **(Clark. A et al 2002)**.

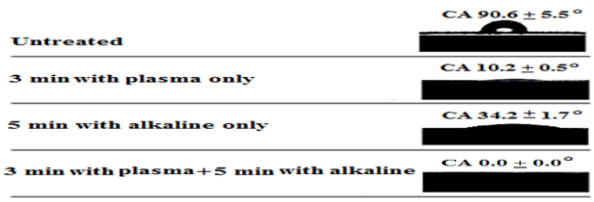

Figure 5: Contact angle (CA) photographs of PET fabrics

Figure 5: Contact angle (CA) photography of PET fabrics.

Surface Tension:

The surface tension of the liquid (water) γ_l = 72. 8 mJ/m^2 at 20 ^{O}C for the calculation (data from Spelt and Neumann, 1987). As shown in **Figure 6**, through equations (7) and (8) the surface tension (γ_{sl}) of untreated PET surfaces is around 18.00 mJ/m^2 at 20 ^{O}C. PET fabric with a surface tension of less than 37 mJ/m^2, is not capable of absorbance and consequently there is a problem with spreading of liquids upon the surface **(Souheng WU. J, 1974)**. Fox and Zisman created the concept of critical surface energy theory in 1950 **(Hejda. F et al. 2010)**. They introduced a linear relationship between the surface tension of a series of liquids and cosine of the advancing contact angle. In the Zisman theory, the surface energy is determined using critical surface tension (γ_c), which differs from the quantity of (γ). The concept of the Zisman theory is based upon wetting the surface with a liquid with a surface tension less than or equal to its surface tension. In the experiment after combining the plasma pre-treatment for 3 min followed

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by 4% alkaline etching for 5 min at 85 °C, the surface tension between solid-liquid interfacial tension obtained is 72.72 mJ/m^2 which is equal the liquid surface tension.
A critical surface tension which is the surface tension of the liquid needed to completely wet the solid (contact angle between the solid and liquid is zero). This critical surface tension value differs from the surface free energy of the solid, and is not divided into dispersive and polar components. In practice, critical surface tension is defined by measuring the contact angle between several different probe liquids and the studied surface. The results are then plotted by having cos θ to calculate the surface tension in y axis and the different treatment in x-axis. The plasma pre-treatedfor 3 min followed by 4% alkaline etching for 5 min at 85 °C shows the measurement points and extrapolated to point cos θ=1 which will give the critical surface tension value of the surface.

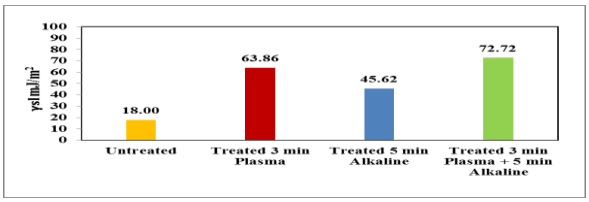

Figure 6: Variation of surface tension of polyethylene terephthalate fabrics for different treatment processes.

CONCLUSION

Plasma pre-treatment followed by alkaline etching was used as a proposed technique. This study shows that differently prepared PET fabrics have different influences on their surfaces. The change in surface morphology shown by SEM and AFM imaging indicates that the pre-treatment using both oxygen and argon gases could increase the roughness of the PET surfaces compared with plasma, alkaline treatment only. It is indicated that plasma pre-treatment has great synergistic effect with alkaline treatment. Therefore, the rough surface of fabric with hydrophilic polar groups leads to enhancement of the absorbability of PET fabrics.

الخلاصة

الطاقة القليلة المستهلكة وتوفير مواردها تتزايد صورها في الصناعات في الأونة الاخيرة وذلك للتقليل من استنزاف الموارد والتحكم فى التلوث. تستخدم العمليات الكيميائية التى استبدلت باستخدام تحفيز البلازما فى صناعة النسيج . تناولت هذه الورقه دراسة تأثير معالجة أقمشة البولستر (PET) بتحفيز البلازما والمعالجة بالقلوى وذلك اعتماداً على فقد الوزن بعد المعالجة بالبلازما باستخدام ثلاثة انواع من الغازات الارجون و الاكسجين و خليط اكسجين /ارجون. حيث وجد أن العلاج بتحفيز البلازما باستخدام خليط من الارجون والاكسجين بالاضافة للعلاج بالقلوي فى درجة حرارة منخفضة قد أعطى خصائص سطح خشن لقماش البولستر (PET). ومن ثم تحليل التغيير وتحسين السطح للشعيرات باستخدام جهاز مجهر المسح الالكترونى (SEM)، ومجهر القوى الذري (AFM). وبعد ذلك تم اختيار العلاج بالقلوي فى درجة حرارة منخفضة 85 درجة مئوية و تركيز 4% على ان يتم الجمع بين العلاج

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بالقلوى مع تحفيز البلازما جنبا إلى جنب في عمليات مختلفة. وايضاً تم حساب خصائص تبلل السطح اعتمادا على زاوية التماس ($\cos\theta$)، حيث انتشرت قطرة الماء بسرعة وكانت زاوية الاتصال صفر على سطح القماش بعد الجمع بين طريقتى المعالجة

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