

# IMPROVEMENT SURFACE PROPERTIES OF POLYPROPYLENE AND POLYESTER FABRICS BY GLOW DISCHARGE PLASMA SYSTEM UNDER ATMOSPHERIC CONDITION

## ATMOSFERİK GLOW DEŞARJ PLAZMA SİSTEMİ İLE POLİPROPİLEN VE POLİESTER KUMAŞLARIN YÜZEY ÖZELLİKLERİNİN İYİLEŞTİRİLMESİ

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### ABSTRACT

In order to raise wettability and to improve soil release of polyester (PES) and polypropylene (PP) woven fabrics, atmospheric pressure plasma were applied. In this study, argon and air plasma were used for modification of PES and PP fabrics. The capillary method and contact angle measurements of plasma treated and untreated textile materials were applied to evaluate the improvement in water uptake of PES and PP fabrics. Chemical changes in the fabric surface after atmospheric plasma treatments were determine by Fourier Transform Infrared (FTIR) and XPS. Their surface morphologies were evaluated by SEM.

**Key Words:** Atmospheric plasma, Polyester, Polypropylene, Fiber, Hydrophilicity

### ÖZET

Atmosferik plazma işlemi uygulanmış poliester (PES) ve polipropilen (PP) dokuma kumaşların kir iticilik özelliği ve ıslanabilirliği incelenmiştir. Bu çalışmada, argon ve hava plazma PES ve PP kumaşların modifikasyonu için kullanılmıştır. PES ve PP kumaşların hidrofilliklerini değerlendirmek için, işlem görmemiş ve plazma işlemi görmüş materyallere kapilarite ve temas açısı ölçümleri uygulanmıştır. Plazma işlemi sonunda kumaş yüzeyinde meydana gelen kimyasal değişim Fourier Transform İnfrared (FTIR) and XPS ile belirlendi. Yüzey morfolojisi SEM ile değerlendirilmiştir.

**Anahtar Kelimeler:** Atmosferik plazma, Poliester, Polipropilen, Lif, Hidrofillik

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### 1. INTRODUCTION

The concern in the textile industry over reduction of water consumption and waste-water treatment in all areas of fiber, yarn and fabric preparation has caused a continuous search for new non-aqueous treatment. The plasma treatment of textile materials has been utilized as a substitution for current wet treatment and pre-treatment processes. Plasma treatments have been proven effective for increasing particular surface properties of textile materials. The plasma can be produced when a gas at low pressure

or atmospheric pressure is submitted to an electric field (1, 2).

In plasma area, many phenomena can occur derived from the plasma/surface interacion of the material. One the main effects of the interaction between active chemical species due to the plasma colliding with a textile materials is the tearing away of molecules chains, the formation of new functional groups and morphological alterations, like the formation microporosity (3). The treatment gives rise to the formation of free radicals at the surface and further down in the textile

materials which react with the excited species in the plasmas. The surface treatments also lead to a crosslinked skin propagated by recombination of polymer radicals. Chain scission takes place simultaneously with cross-linking and oxidation. Small molecule degradation products are constantly removed by evaporation and sputtering, while polymeric scission products remain intermeshed with undergraded polymers and crosslinked networks. The modified depth of polymeric materials thanks to plasma is between 50-500Å (4).

Many studies have already been carried out with low pressure plasma, but atmospheric pressure plasma treatments would be ideal for the continuous processing of textile materials. It was previously believed that plasmas generated at atmospheric pressure are not uniform or stable; however a research on atmospheric plasma has shown their uniformity, stability and applicability to processing and treatment of textiles. Atmospheric plasma devices and treatments could be utilized for use in high-speed continuous processing operation, and when optimized, could replace or increase many current wet-chemical finishing processes (5, 6).

In this study, PES and PP woven fabrics were modified by atmospheric pressure plasma treatment utilizing air and argon gases and their chemical and morphologic modifications were investigated.

## 2. EXPERIMENTAL SET-UP

### 2.1. Materials

In this study, 67 g/m<sup>2</sup> PES and 145 g/m<sup>2</sup> PP plain woven fabrics were used. These fabrics extracted with acetone for 4 hours, thoroughly dried in air. Argon (purity of >99.99) and air (20.9% oxygen, 79.1% nitrogen and relative humidity < 3 ppm) as the process gases were purchased from BOS. Acetone, potassium bichromate, carbon black, liquid paraffin and CCl<sub>4</sub> employed in this work were purchased from Merck Chemical Company.

### 2.2. Atmospheric Pressure Plasma Treatment

For plasma treatment a laboratory scale atmospheric plasma reactor was utilized. The discharge is produced between four electrode (diameter = 17 mm) couples. One of the electrodes in the couple is covered dielectric material, and the inter-electrode distance is set 2 mm (7). PES and PP fabrics were treated with a power 50, 100 or 130 W and at different time intervals.

### 2.3. Material Characterization

#### 2.3.1. Hydrophilicity

In order to evaluate hydrophilicity of the original and treated PP and PES fabrics, capillary rise and contact angle measurements of each material were carried out. Capillary rise measurements were made the method which was

described in the Ref. 2. Capillary rise measurement was repeated eight times, and arithmetical mean was taken. Contact angle were measured by using a goniometric system (Tampere University, in Finland); measurement was repeated ten times, and the arithmetical mean was taken.

#### 2.3.2. Soil Resistance

Carbon black has been generally used for soil preparations because of their light reflectance properties to simulate soil in a fabric artificially. In our study, the soiling behaviour of the fabrics was investigated according to the procedure described by Seventekin and Oktem *et.al.* (6). To present to soiling data,  $\Delta E$  values of the fabrics were collected from the datacolor Instrument measurement results (Minolta 3600d model spectrophotometer, Turkey).

#### 2.3.3 Chemical Characterisation

**FTIR / ATR Analysis:** (Fourier Transform Infrared Spectroscopy with Attenuated Total Internal Reflectance

*mode measurement*) The Infrared (IR) spectra of fabrics were determined by means of a Perkin Elmer 100 FTIR spectrometer in ATR reflection mode using a Diamond/zinc selenide crystal. To ensure reproducible contact between the crystal faces and the fabric, a pressure of 50 kPa was applied to the crystal holder by means of a calibrated torque screw driver. An average of 15 scans using a resolution 4 cm<sup>-1</sup>.

**XPS Analysis:** X-ray photoelectron spectroscopy was used to monitor the modifications produced in the outermost (5-10 nm) polyester and polypropylene fibres surfaces. In this study, the measurement depth was 7 nm. The fabric samples were analysed using SPCS XPS system with a Mg K $\alpha$  X-ray source operated at 10 kV and 200 W. The pressure inside analysis chamber was 10<sup>-8</sup> to 10<sup>-9</sup> torr. Survey scans were taken with a pass energy of 48 eV. The peak positions were corrected for charging relative to the C-C bond which was assigned a binding energy of 284.5 eV.

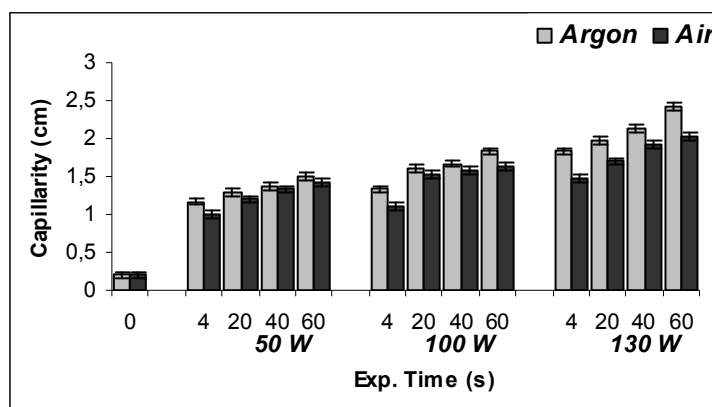


Figure 1. Capillary rise on PP fabric after plasma treatment at various power and times

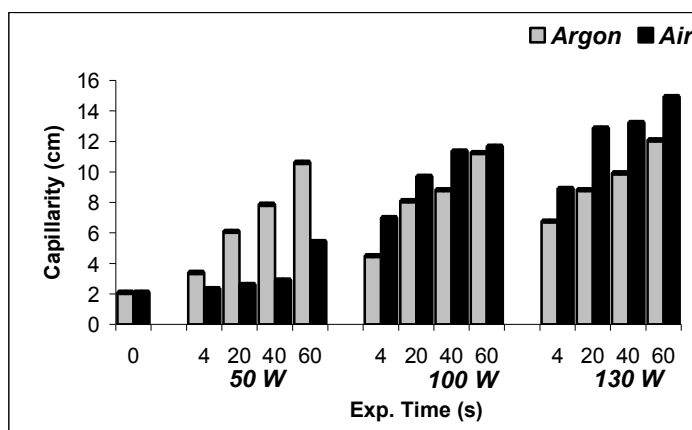


Figure 2. Capillary rise of PES fabric after plasma treatment at various power and times

**Table 1.** Contact angle values of PP and PES fabrics (C.A: Contact Angle, S.D. Std. Dev.)

Power (W)	Time (sec)	PP Fabrics				PES Fabrics			
		Argon Pls.		Air Pls.		Argon Pls.		Air Pls.	
		C.A (°)	S. D.	C.A (°)	S. D.	C.A (°)	S. D.	C.A (°)	S. D.
0	0	105.7	2.47	105.7	2.47	90.22	3.06	90.22	3.06
50	4	96.24	2.31	98.98	2.54	74.95	3.26	75.66	3.41
	20	91.02	2.3	91.32	2.45	71.67	3.2	73.94	3.12
	40	87.7	1.97	91.13	2.43	70.54	3.35	73.14	3.27
	60	85.97	1.5	88.38	2.26	54.98	2.95	71.01	3.12
100	4	94.68	2.45	96.48	2.35	72.88	3.17	65.7	3.21
	20	87.69	2.01	89.56	2.15	56.94	3.05	52.98	2.97
	40	83.28	1.72	88.28	2.2	52.56	2.76	48.01	2.28
	60	82.41	1.47	85.2	1.95	51.9	2.46	47.86	1.92
130	4	93.94	2.25	94.04	2.26	70.43	2.85	55.02	3.03
	20	83.69	1.96	88.74	2.13	49.91	2.5	47.34	2.65
	40	82.08	1.5	84.24	1.96	48.56	2.56	41.12	2.15
	60	81.44	1.44	83.01	1.4	45.17	1.81	39.23	1.6

### 2.3.4. Morphology

The microstructure of PP and PES woven fabric before and after plasma were characterized by using SEM a Philips XL30S FEG microscope with digital microscopy imaging.

## 3. RESULTS

### 3.1. Evaluation of Hydrophilicity

In this study, to improve the surface properties of PES and PP fabrics, argon and air plasma were carried out at atmospheric pressure. The surface properties were evaluated by measuring capillary rise and contact angle as a function of plasma treatment time, power and gases. The treatment time and power in both air and argon range between 4-60 s and 50-130 W, respectively. Fig. 1 and 2 show the warp direction capillary rise results as a function of exposure time and plasma power for PP and PES fabric, respectively. Table 1 shows contact angle results of PP and PES fabric after treatment in air and argon plasma.

As shown in Table 1, the contact angle for the PP fabrics was found to change from 105.73° for the untreated sample to the lowest value 81.44° after treatment in both discharges, and capillary rise height increased, while contact angles decreased. Figures 2 and Table 1 show that the plasma exposure led to similar changes in the PES fabric surfaces with respect to the contact angle: the contact angle decreases from 90, 22° for the untreated sample to 39, 23° after

treatment in plasmas, and capillary rise height increased. Similar increases in wickability/capillarity were observed by Borcia et. al.(8).

It is shown that the wettability is strongly enhanced by plasma treatment. When air and argon are used in the plasma state for a treatment time of 1–60 second, it incorporates hydrophilic groups such as —OH, C=O, C—O—O—, O—C=O etc., predominantly making the surface more hydrophilic. Capillary rise height increased unlike contact angles, but a little difference between two plasmas can be seen more clearly from capillarity and contact angle results.

It is shown that air plasma is more efficient in implanting oxygen functionalities than argon plasma. In air plasma, most of the created radicals at the polymer surface will quickly react with an oxygen particle, resulting in an efficient implantation of oxygen functionalities. In contrast, plasma treatment in argon leads to a cross-linking reaction in combination with surface oxidation. This crosslinking reaction inhibits the surface oxidation, resulting in a slower incorporation of oxygen functionalities.

When PP and PES are treated in oxygen-containing argon plasma, the cross-linking and oxidation process occurs in different steps, resulting eventually in an oxidized three-dimensional cross-linked structure (9). However, the exact mechanism leading to this oxidized cross-linked structure is not yet described in detail.

### 3.2. Evaluation of Ageing

The most serious problem for plasma treatments is the durability of the treatment. The most probable reason for this has been considered to be “overthrough” or migration of the created polar groups into the textile materials (4). The lack of durability has been attributed largely to the surface rearrangement of macromolecular segments which is truly physical but not due to the loss of chemical groups introduced by the surface treatment. Furthermore the aging seems to depend on the chemical nature of the substrate. To determine ageing properties of synthetic textile fabrics after plasma treatment, capillary rise measurement were fulfilled after seven, thirty and one hundred eighty days for samples that were obtained maximum capillarity values (130 W-60 s argon and air plasma). PP and PES fabric results are given Figure 3 and 4, respectively.

As it is shown in the figures, although argon and air plasmas made the surfaces of the PES and PP fabrics hydrophilic, the hydrophilicity decreased with the ageing time, due to the rearrangement of hydrophilic functional groups formed by plasma treatments (10). Ageing for the air treated samples were higher than argon treated samples. When inert gases like argon were used for the treatment of polymer surface, ions and UV photons, present in the discharge, would break C—C or C—H bonds, leading to the production of carbon radicals. Many more radicals were formed or induced on the PES and PP

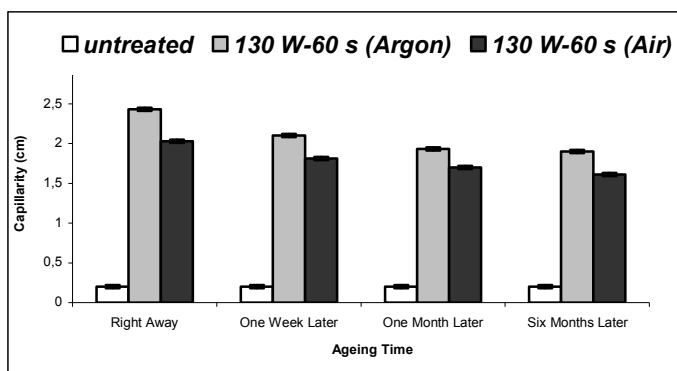


Figure 3. Ageing of plasma treated PP fabrics

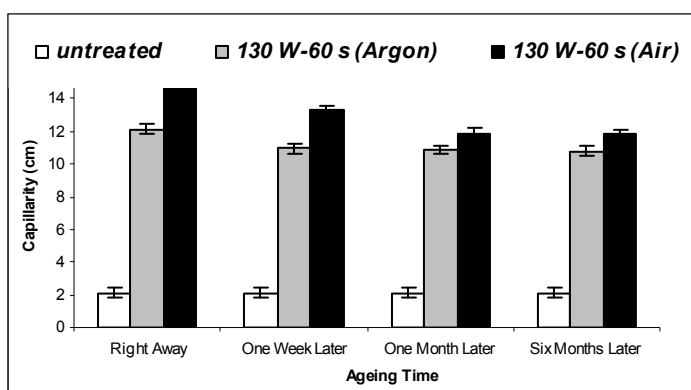


Figure 4. Ageing of the plasma treated of PES fabrics

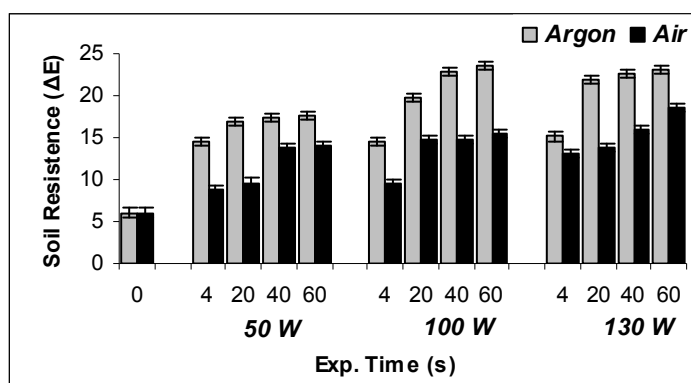


Figure 5. Soiling behaviour PP fabrics after atmospheric pressure plasma

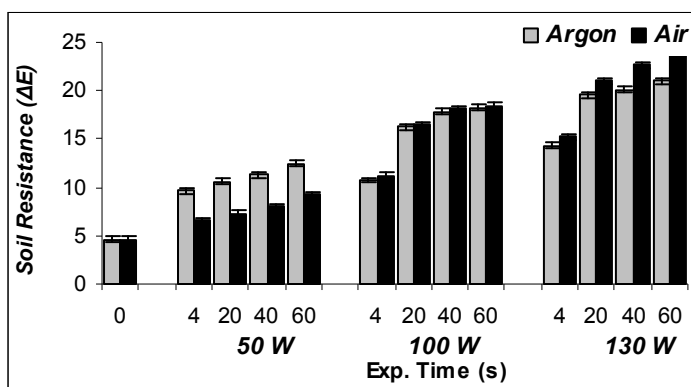


Figure 6. Soiling behaviour PES fabrics after atmospheric pressure plasma treatment

surface by argon and these radicals reacted with each other to form crosslinked layers, which would be expected to prevent the rearrangement of hydrophilic functional groups formed by plasma treatment (11).

### 3.3. Evaluation of Soil Resistance

Soil release characteristics are especially important for the manufacturers and the end-users of the fabrics. Hydrophobic synthetic fibers attract soil to a greater extent than natural fibers because of the development of electrostatic charges on the surface (6).

The  $\Delta E$  values for the PP and PES fabrics modified by atmospheric argon and air plasmas are given Fig. 5 and 6, respectively. Soil release usually correlates with the hydrophilicity of the fabrics. Activating surfaces are to change their polarity. Polar surfaces are made more non-polar to render them repellent to liquids and reduce adhesion of soil particle, non-polar surfaces will be made more polar to improve water wetting and soil release properties (11).

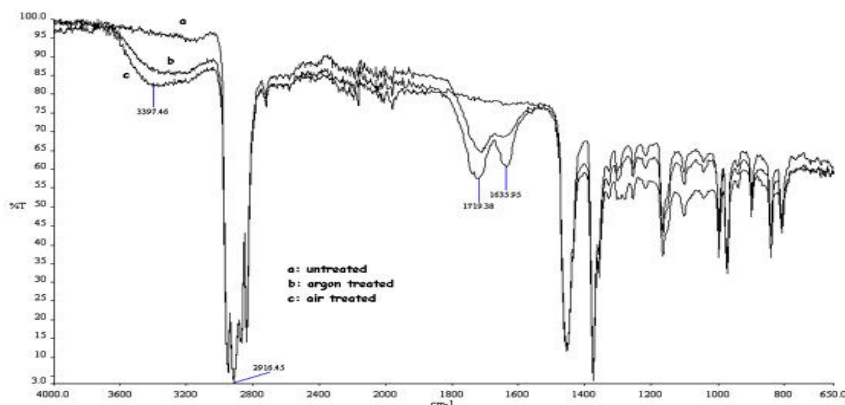
The relative color intensities of both PES and PP fabrics were increased thanks to air and argon plasma treatment with increasing treatment time and power.

### 3.4. Evaluation of Chemical Characterization

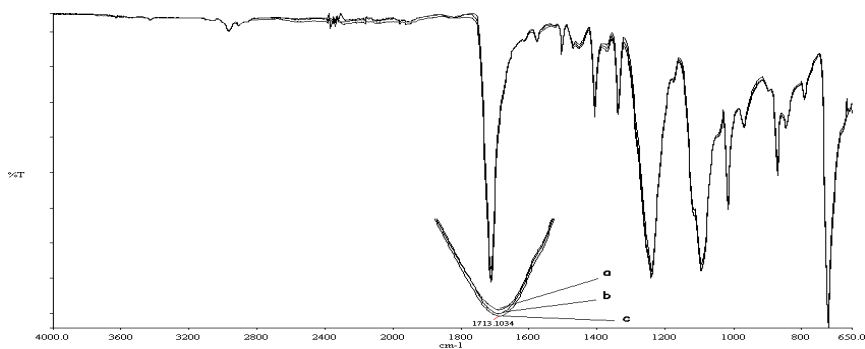
#### 3.4.1 FTIR/ATR Analysis

Treated PP and PET fibres are measured by FTIR spectrometer in order to investigate what chemical changing happens at the surface of polymer. The infrared absorption spectras of untreated and plasma-treated PP and PES fabrics are shown in Figure 7 and 8, respectively.

In Figure 7, for the untreated polypropylen fibers, there are two strong absorption peaks. Peaks are assigned as follows: C-H vibration peak in the range of  $1460-1470\text{ cm}^{-1}$ , anti-symmetric/symmetric  $\text{CH}_2$  ve  $\text{CH}_3$  peaks in the range of  $2820-2950\text{ cm}^{-1}$ . After the plasma treatments, some new absorption peaks were appeared in Figure 7: -OH stretching vibration  $3500-3200\text{ cm}^{-1}$ , -COOH (carboxylic acid) absorption peak in the range of  $1700-1750\text{ cm}^{-1}$ , C=C non-saturated double bond conjugated with  $\text{COO}^-$  absorb peaks in the range  $1630-1645\text{ cm}^{-1}$  were appeared (12, 13). Functional groups could be generated



**Figure 7.** FTIR results of treated and untreated PP fabrics. a) Untreated, b) argon plasma treated c) air plasma treated



**Figure 8.** FTIR results of treated and untreated PES fabrics. a) Untreated, b) argon plasma treated c) air plasma treated

**Table 2.** Chemical compositions of 130 W-60 s argon and 130 W-60 s air plasma treated polyester and polypropylene fabrics

	Elemental composition (%)			O/C ratio	N/C ratio
	C	O	N		
Polypropylene Sample					
Untreated	93.8	6.0	0.2	0.064	0.002
Argon treated	84.0	15.2	0.7	0.181	0.008
Air treated	84.3	14.7	1.0	0.174	0.012
Polyester Sample					
Untreated	73.3	26.6	0.4	0.363	0.006
Argon treated	64.8	34.1	1.1	0.526	0.017
Air treated	63.3	35.2	1.5	0.556	0.024

of the fabric surface because intensity of the carbonyl and hydroxyl band increased (13). The functionalization of the surface of the fiber means a higher polarity leading to an increased hydrophilicity.

As can be seen from Fig. 8, contrary to the new absorption peak appearance of plasma-treated PP fibre, no new absorption peak come up in the infrared absorption spectra of plasma treated PES compared to the untreated one. The peaks at the wave

numbers, 1714, 1250, 1120, 1046 and 726  $\text{cm}^{-1}$  are specific for PES. The three peaks between 2970 and 2846  $\text{cm}^{-1}$  are the C-H  $\text{sp}^3$  vibration (14). The peak height at 1713  $\text{cm}^{-1}$  due to C=O absorption shows a slight increase on plasma treatment.

### 3.4.2 XPS Analysis

The XPS show C (1s) and O (1s) peaks, which characterise the carbon and oxygen atomic composition in PES and PP fabrics. For more clarity,

the chemical composition in terms of C (1s), O (1s) and N (1s) is given in Table 2 and Figure 9 and 10 as function of gases. From this it is clear that the plasma treatment has a significant influence on the PES and PP surface.

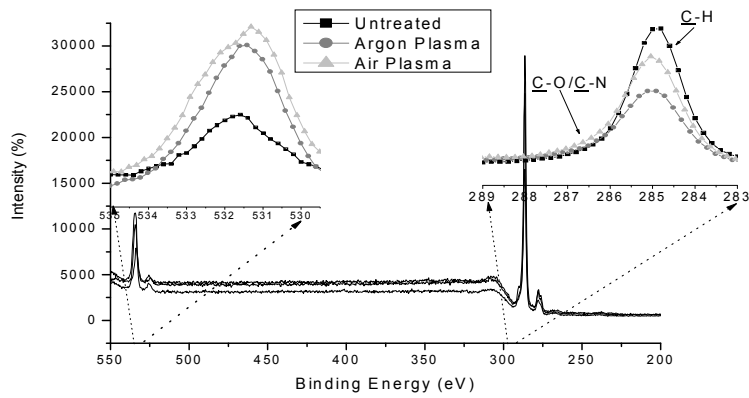
When PES fabric was treated in air plasma for 60 s, a decrease in the C (1s) peak was observed (from 73.3 to 63.3%). At the same time the O (1s) peak increased (from 26.6 to 35.2%). When PES fabric was treated in argon plasma for 60 s, a decrease in the C (1s) peak was observed (from 73.3 to 64.8%). At the same time the O (1s) peak increased (from 26.6 to 34.1%). The O/C ratio of the untreated, argon and air plasma treated PES increases as follows, respectively:  $0.363 < 0.526 < 0.556$ . Increasing O/C atomic ratio means that oxygen-containing functional group amount increases.

For the PP fabric, the O/C and N/C ratio of the untreated sample are 6.4 and 0.2 %, respectively. This means that the untreated PP fabric appears to contain some contamination or more probably low-level surface oxidation. After plasma treatment in air and argon, the O/C atomic ratio increases to 18.1 and 17.4 % for saturated samples, respectively. These increases in O/C ratio suggest that new oxygen-containing groups are formed on the fabric surface after treatment with an air or argon plasma.

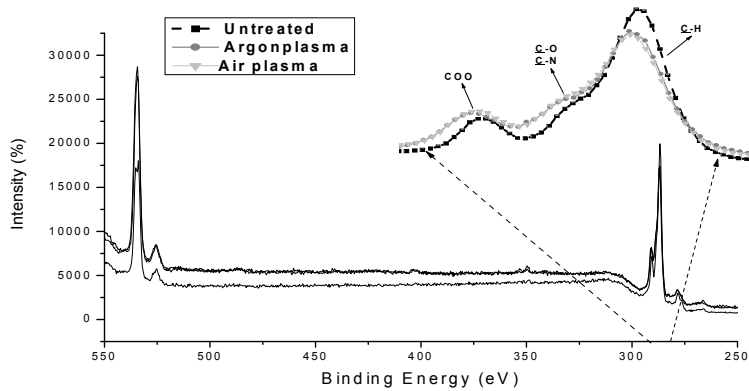
Furthermore, after plasma treatment, N/C atomic ratio increases a little because of nitrogen-containing functional groups or contamination.

### 3.5. Evaluation of Surface Morphology

It is known that plasma affects the polymer surface, especially the morphology. SEM images of the untreated, argon and air plasma treated PP and PES fibers are given Fig. 11 and 12, respectively. As seen in figures, surface morphology changes significantly after plasma treatment due to etching on the surfaces of the fiber.



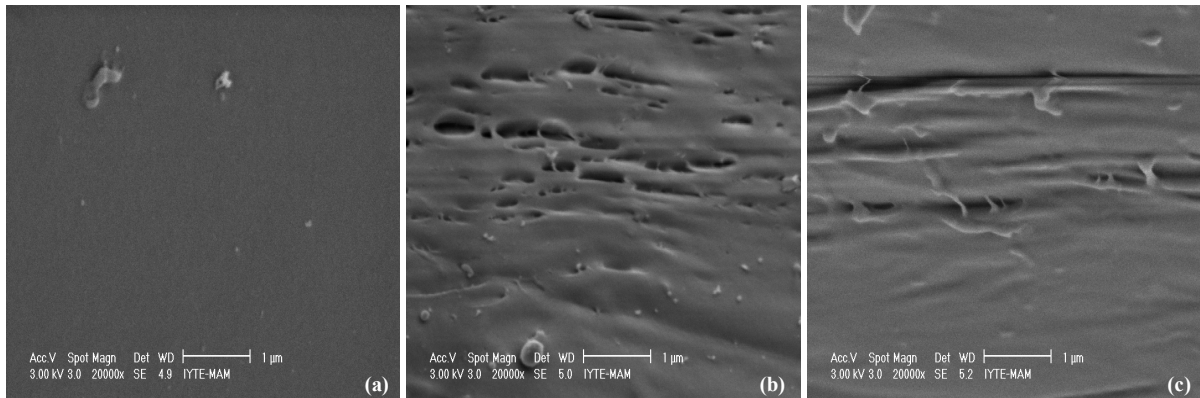
**Figure 9.** XPS results of untreated and treated PP fabrics



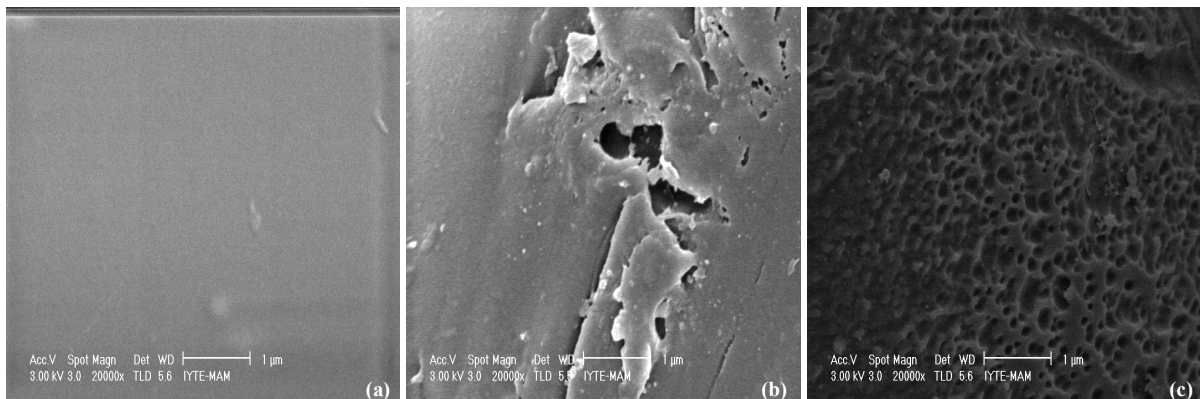
**Figure 10.** XPS results of untreated and treated PES fabrics

Scanning electron microscopy (SEM) image was observed to comprehend the alteration of surface morphology of treated fabrics. The PP and PES fiber surfaces after argon and air plasma treatment are shown in Figures 11–12 with a magnification of 20,000. It can be seen that the surface-etching process occurs on the surface of argon and air plasma treated fiber.

Figure 11 (a) and 12 (a) demonstrates the SEM image of untreated PP and PES fiber, while Figures 11 (b), (c) and 12 (b), (c) display the SEM images of plasma treated PP and PES fiber with argon and air gases, respectively. Figure 11 (a) and 12 (a) clearly demonstrate that the untreated fibers surface are smooth and free from roughness, indicating that no damage occurs on the fibers surface. However, in Figures 11 (b), (c) and 12 (b), (c) illustrate a change in the fiber surface morphology. For the PP fiber, when compared with the air plasma treatment, the argon plasma treatment is more effective on the fiber surface. For the PES fiber, when compared with the argon plasma treatment, the air plasma treatment has a more severe damage on the fiber surface. This is probably due to the comparatively slow rate of physical etching introduced by



**Figure 11.** SEM images of PP Fabrics. a) Untreated b) Argon treated c) Air treated



**Figure 12.** SEM images of PES Fabrics. a) Untreated b) Argon Treated c) Air treated

the argon plasma gas (15). Granules, microcraters, and ripple-like structures of submicrometer size are observed on the air plasma-treated surface (Fig. 11 (c)) (16). In summary, plasma treatment with air gas is more efficient because air has oxygen molecules. It also signifies that a stronger porous surface effect is created on the air plasma-treated PES fibers.

#### 4. CONCLUSION

In this paper, PP and PES fabrics were plasma-modified using a dielectric barrier discharge in air and argon at atmospheric pressure. The results showed that all of contact angle of PP and PES surfaces decreased, and their capillary rise height increased with plasma treatment, which means the surface hydrophilicity of all material surfaces have been improved.

Air plasma treatment was more effective in reducing the contact angle due to the fast reaction between created radicals at the polymer surface and oxygen species. In contrast, in argon, cross-linking competes with surface oxidation, resulting in lower modification efficiency.

The modified surface was not stable with time, and undergoes rearrangement of the modified surface layers, leading to the incorporation of some of the functional groups within the surface layers. Therefore, to characterize and use these surfaces, their time-dependent nature has to be taken into account.

Soil release usually correlates with the hydrophilicity of the fabrics. It can be roughly estimated that the better is the soil release, the better is the hydrophilicity of the fabrics.

The XPS and ATR-FTIR results showed that the percentage of oxygen atom in these samples increases and it consisted with the contact angle measurement. Evaluating SEM results of PES and PP surface with their contact angle data, it was found that the increasing of surface roughness in micron scale can also improve hydrophilicity, besides the contribution of the rising up of percentage of the oxygen atom.

Therefore, it can be concluded that atmospheric pressure cold plasma could be effectively and safely used to improve hydrophilicity of the polymeric substrates without incurring high cost or creating environmental pollution.

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