Surface Modification and Dyeing of Ultra-High Molecular Weight Polyethylene Fibers by Plasma Discharge Mode

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ABSTRACT

In this study Ultra-high molecular weight polyethylene (UHMWPE) fibers were subjected to oxygen/argon plasma treatment in order to improve the wettability without changing bulk properties. The results showed that surface roughnesshave been modified by plasma treatment, indicating that the plasma treatment favored the interaction with dyeing UHMWPE fibers. After the treatment the treated fibers were characterized by using scanning electron microscopy (SEM),Fourier transforms infrared spectroscopy analysis (FT-IR), X-ray Diffraction (XRD), atomic force microscopy (AFM). Dyeability of the modified fibers were investigated by dyeing using disperse blue dye and also fibers were printed with pigment red colour.The obtained results were compared with the dyeability of the untreated fiber. The result showed that a significant increase of K/S value after dyeingandafter printing at dyeing temperature of 100 ° C. Thus, the oxygen/argon plasma treatment of UHMWPE fiber can obtain good dyeing properties overall, plasma treatment has a great improvementof theroughnesssurface and dyeing property of UHMWPE fiber.

Keywords: UHMWPE, surface modification, Disperse dye, pigment, K/S value.

تحسين السطح بوضع تفريغ البلازما وصباغة ألياف البولي إيثيلين عالية الوزن الجزيئي

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الملخص

تعرضت ألياف البولي إيثيلين عالية الوزن الجزيئي للعلاج بتحفيز البلازما باستخدام خليط من الارجون والاكسجين من أجل تحسين قابلية الامتصاص دون تغيير خصائص الالياف. أظهرت النتائج أن خشونة السطح قد تم تحسينها عن طريق معالجة البلازما، مما يشير إلى أن معالجة البلازما تحسن التفاعل مع صباغة ألياف البولي إيثيلين عالية الوزن الجزيئي. اختبرت الألياف المعالجة عن طريق جهاز مجهر المسح الإلكتروني (SEM)، حيث يقوم فوراً بالتحليل الطيفي بالأشعة تحت الحمراء (FTIR)، انحراف الأشعة السينية (XRD)، الفحص بمجهر القوى الذرى (AFM). تم فحص صباغة الألياف المعدلة عن طريق الصباغة باستخدام الصبغة الناشرة الزرقاء وطباعة الياف أخرى بالوان البجمنت الحمراء. تمت مقارنة النتائج التي تم الحصول عليها بعد الصباغة مع الألياف غير المعالجة. أظهرت النتيجة ان هنالك زيادة كبيرة في قيمة K / S بعد الصباغة والطباعة عند الصباغة في درجة حرارة 100 درجة مئوية. وبالتالي، فإن معالجة ألياف البولي إيثيلين عالية الوزن الجزيئي بتحفيز البلازما باستخدام خليط من الارجون والاكسجين يمكن أن تحصل على خصائص صباغة جيدة بشكل عام، والمعالجة بتحفيز البلازما يعمل على تحسن كبير في خشونة السطح وخاصية الصباغة لالياف البولي إيثيلين عالية الوزن الجزيئي.

INTRODUCTION

Plasma is the fourth state of matter and composed highly excited, ionic, atomic, molecular and radical species. Plasma gasses are excited in to these energetic levels by microwave, radio frequency, corona and dielectric barrier **(Khelifa. F, et al., 2016)**. Much functionality will arise near the surface while the plasma applied to the polymer with proper density and treatment time. In activation process, firstly hydrogen atoms are abstracted from the polymer chains by breaking the bond with the plasma energy. So radicals are created at the midpoint of the polymer chains and these radicals then built up new bonds with each other by cross linking and with species that are activated by plasma gas **(Ellinas. K, et al., 2016;Noeske. M, et al., 2004)**. Type of the applied plasma gas is one of the most important parameter to obtain required surface activation.

Plasma surface treatment of the polymers is one of the most effective and economical technique to modify the surface properties of the polymeric materials without changing bulk material **(Ellinas. K, et al., 2016)**. It is possible to selectively modify the surfaces to enhance wide variety of surface properties such as surface wettability, functionality, hydrophilicity, hydrophobicity surface roughness, scratch resistance, wear resistance, physical, chemical and mechanical properties by applying proper plasma method and gasses to the polymer surfaces **(Bormashenko. E, et al., 2015;Barbarash. L.S, et al., 2016;Khelifa. F, et al., 2016)**. Therefore, plasma surface modification has become increasingly popular method to improve the functionally required

properties of biomaterials instead of developing new materials with expensive and time consuming processes **(Noeske. M, et al., 2004)**. Noeske et al. Studied effect of atmospheric plasma jet on adhesion property of a series of polymers included High-Density Polyethylene (HD-PE). They reported that adhesion property of the plasma treated polymers increased by oxygen functionality of the surfaces. In previous study **(Huang. C.Y, etal., 2013)**, argon plasma surface modification was applied to surface of UHMWPE textile. Reported that the peel strength of the UHMWPE increased and contact angle decreased from 80^{o} to 28^{o} **(Huang. C.Y, et al., 2013)**.

Today the use of polymeric materials has become very popular in engineering applications. As a result of a continuous improvement in fibers, development of innovative fabrication technologies, advanced polymeric offers possibilities for major leaps in design, manufacturing, energy conservation, product utility and diversity. Since its development in the early 1950s, ultra-high molecular weight polyethylene (UHMWPE) has gained popularity owing to its bio-compatibility. **(Liu. Y and Sinha. S.K 2013; Chmutin. I, et al., 2017; Panjwani, B, et al., 2011; Grinev. V, et al., 2018)**, self-lubricating properties. **(Minn, M. and Sinha, S.K 2008)**, chemical stability **(Wang. H.G, et al., 2016)**, wear and impact resistance, thereby making it an excellent choice for a range of engineering and biomedical applications.

Ultra-high molecular weight polyethylene (UHMWPE) is a semi-crystalline engineering thermoplastic with a high level of performance, exceptional wear resistance, low coefficient of friction, high impact strength, and chemical inertness, all of which are suitable for tribological applications to overcome wear and friction issues. A major application of (UHMWPE) since the Sixties has been as replacement parts for human joints damaged by acute arthritis or injuries. However, one yet unsolved issue is the production of (UHMWPE) wear debris after a certain period of use **(Wang. A, et al., 1998)**. UHMWPE fiber, which is one of super fibers, because it has very high strength 25~45 g/d, stronger than para-aramid fibers and is already being used in various industrial fields that need strong tensile properties.

In addition, it has low specific gravity less than 1.0 and excellent chemical resistance make it possible to expand use of the fiber in many extreme conditions and possesses a unique complex of properties including a low coefficient of friction, anti-adhesive properties, and the highest wear resistance among polymers, due to which it is widely used in various friction units in machines and mechanisms, for the fabrication of implante joints, etc However, it has been generally considered that the UHMWPE fiber was impossible to dye at any dyeing systems because of the extreme hydrophobicity and high level of crystallinity **(Goerg. H. M, et al., 2010)**.

In order to dye polyolefin fibers such as UHMWPE and polypropylene fibers, two kinds of coloration methods are being considered. First method is adding pigment at the stage of fiber formation, and the second is chemical modification of the fibers. The first method is possible to dye the fibers, but the application is relatively restricted since the color is determined at the stage of fiber formation. The second method is also possible to dye the fibers, but there are disadvantages such as the loss of the typical properties of the fibers by chemical modification **(Jin-seok. B, et al., 2014)** and low color fastness. For dyes to have affinity toward the extremely hydrophobic fibers like UHMWPE, they also need to have extreme hydrophobicity. The previous studies showed that the dyeing of UHMWPE fibers without any physical and chemical modifications is possible at the established process by super hydrophobic dyes having long alkyl substituents which impart extreme hydrophobicity to the dyes **(Taekyeong. K. 2014)**.

In this study, it is aimed to define effect of oxygen + argon gas mixture plasma on surface properties of UHMWPE fibers. The surface morphology of UHMWPE fibers were discussed and

evaluated by: scanning electron microscopy (SEM), Fourier transforms infrared spectroscopy analysis (FTIR), X-ray Diffraction (XRD), atomic force microscopy (AFM) and K/S value was carried out to evaluatethe color of dyeing and printing of UHMWPE fibers.

MATERIALS AND METHODS

Materials:

The Ultra-high molecular weight polyethylene (UHMWPE) fibers were used in this study with molecular weight 35 x 10^5 (supplied by Dacheng Advanced Material Co., Ltd., China). The density and the diameter of fiber were 0.97 g/$cm^3$and about 40 um respectively, and the length was used 5 cm. Therefore, Pigment and C.I. Disperse Blue 183, with λmax 530 nm from Zhejiang Longsheng Group Co., Ltd, (Lonsen), 1 g/L of red pigment dyeing solutions were prepared by mixing 1 g of Pigment and 30 g of Binder in 1 L of deionized water. The pigment and binder were supplied by Clariant. Pigment dyeing was carried out by padding cotton fabric with a horizontal padder until a pick-up percentage of 70% was achieved. The pigmented samples were dried at 90 ° C for 10 min and cured at 145 ° C for 5 min by a curing machine was used as received. All the experiments were carried out using distilled water

Plasma treatment:

The plasma treatment was carried out using the dielectric barrier discharge (DBD) plasma devices are built of two electrodes, the upper electrode is connected to ahigh frequency power source and the lower electrode is connected to the ground. The frequency of high voltage can be adjusted and it can be 20 kHz. The electrodes are usually round, but also the high voltage can be applied between the spiralwound stainless steel electrodes fitted inside a water-cooledquartz tube which is ground. The fibers are placed between these electrodes. The pressure in the reactor chamber is 10^5 Pa.The gas flow rate was (O_2 = 50 ml/min, Ar = 0.5 l/min). The chamber was first pumped and then filled withthe mixed gases up to the atmospheric pressure. After that, the discharge was started and the fibers were treated for a certain duration of treatment time for 3 minand followed by drying at 100 °C for 30 min.

Fiber Surface Characterization

Scanning Electron Microscope (SEM):

The surface morphology of UHMW-PE samples were 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 fibers for SEM observation directly after plasma treatment.

Fourier transforms infrared spectroscopy analysis (FTIR):

The FTIR spectra were recorded by using NICOLET 6700 analytical FT-IR Spectrometer, Nicolet Company, USA. The total reflectance of spectra was taken with a resolution of 4 cm^{-1} and accumulation of 32 scan times in the reflectance mode for each UHMW-PE samples with a range of 400–4000 cm^{-1}. The spectrum peaks were analyzed using spectrum v5.02 software.

X-ray Diffraction (XRD)

The untreated and treated samples were characterized for its change in crystalline behavior by wide-angle X-ray diffraction technique. The experiment was carried outusing a Nicolet 6700 diffract meter (Bruker Corporation, Germany). Cu Kα radiation (λ=1.5406 A) was used at 40 mA and 40 kV. The operating parameter of 2θ ranged from 10 °to 50 °, and the scan rate was 0.2 seconds/step.

Atomic Force Microscopy:

Atomic force microscopy (AFM) (Multimode Nanoscope IIIa, Digital Instrument, USA) was used to examine the surface morphology of the UHMWPE samples before and after the plasma 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 UHMWPE samples were analyzed using a computer. All the samples were tested 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 Chen. C. L 2006)**.

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

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

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.

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

$$\frac{K}{S} = \frac{(1-R)2}{2R}$$

Where: R is the decimal fraction of the reflectance of the dyed fiber, K is the absorption coefficient of the substrate, and S is the scattering coefficient of the substrate. The K/S value is commonly used to demonstrate how deep the color is for a given textile fibers.

RESULTS AND DISCUSSION

Fiber Surface Morphology Before and After Modification:

The surface morphology of the UHMWPE fibers was studied using a JSM 5600LV SEM. As shown in **Figure 1**, Figure 1 (a) the surface of the original UHMWPE fiber was smooth. However, after modification, the surface of the fibers became rough as a result of O_2/Ar plasma treated fibers surface uniformly distributed with micro size protrusion dots and depressed pits as shown in Figure 1 (b). These results are due to the physical and chemical etching effect to the fibers surface molecules caused by active particles such as ions and radicals in the oxygen/argon plasma onto the fibers surface. There was an increase in the surface roughness in Figure 1 (c) after dyeing more than printing surface as shown in Figure 1 (d).

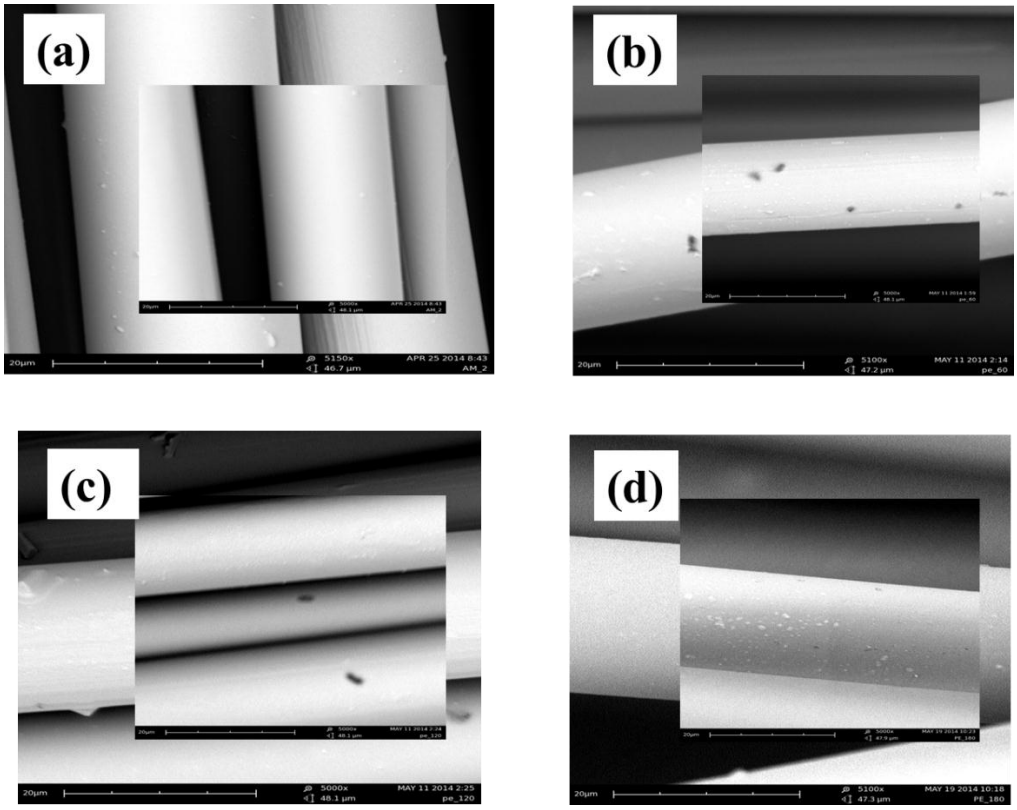

Figure 1: SEM images of UHMWPE fibers, (a) untreated; (b) treated for 3 min O_2/Ar plasma; (c) treated for 3 min O_2/Ar plasma and dyed; (d1) treated for 3 min O_2/Ar plasma and printing.

ATR-FTIR test was used to evaluate the surface change andto determine the different functional groups existingin the UHMWPE fibers. **Figure 2 (a, b, c and d)** shows the FTIR spectra of the UHMWPE fiber before and after surface treatment. FTIR characteristic peaks of UHMWPE fibers appear at wavelengths of 2952-2942 cm^{-1} could be attributed to the asymmetric stretching of the CH_3-CH_2- chain and the symmetrical stretching of the –CH_2-CH_2- chain, respectively. Besides that, a new bands are observed in treated UHMWPE fibers spectrum, compared with as-received UHMWPE fibers spectrum in Figure 1. They are a broad band around3676 cm^{-1} corresponds to the hydroxyl groups (-OH) and a band around 1535 cm^{-1}corresponds to the oxygen-based groups increased after O_2/Ar plasma treatments. Broad absorption corresponding to the hydroxyl group is seen after O_2/Ar plasma treatment. Absorbed water and dye was detected after the O_2/Ar plasma treatments. However, optimization of the treatment conditions was not attempted using FTIR, as it is a qualitative characterization.

The decrease in intensity of these peaks after dye and printing reflect the physicochemical interaction of the polymer with inorganic particles between the contact surfaces. Further this decrease in intensity can also be attributed to the decrease in the number of free UHMWPE chains.

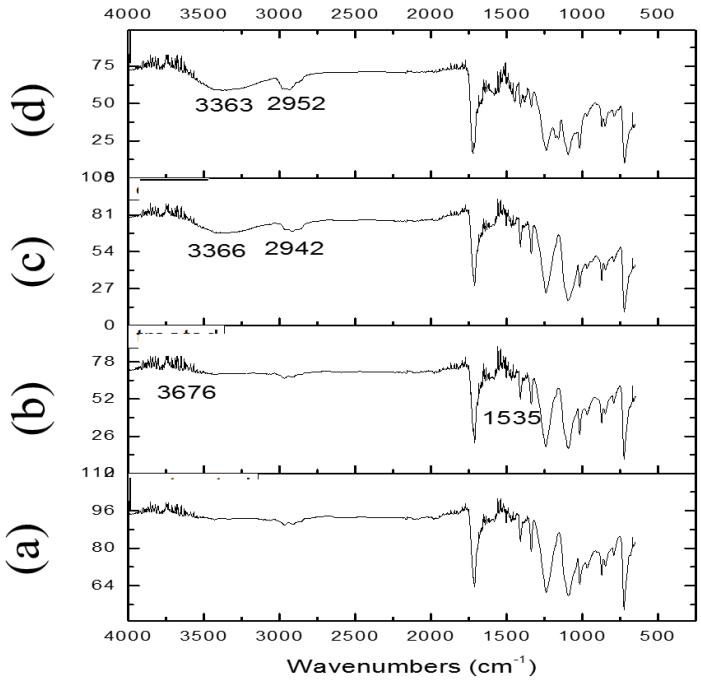

Figure 2. ATR - FTIR spectra of UHMWPE fibers, (a) untreated; (b) treated for 3 min O_2/Ar plasma; (c) treated for 3 min O_2/Ar plasma and dyed; (d) treated for 3 min O_2/Ar plasma and printing respectively.

X-ray diffraction (XRD) is a crystal structure analysis method using the atomic arrays within the crystals as a three dimensional grating to diffract a monochromatic beam of X-rays. The angles at which the beam is diffracted are used to calculate the inter-planer atomic spacing (d-spacing) giving information about how the atoms are arranged within the crystalline compounds. X-ray diffraction is also used to measure the nature of polymer and extent of crystallinity present in the polymer sample. The results of XRD analysis are reported in **Figure 3**. The data of this analysis showedthat no noticeable changes in the value of (size of crystals) of the UHMWPE fibers, but the O_2/Ar plasma treatment slightly increased the total crystallinity. This results indicates that the treatment has not changed the arrangement or decreased the strength of the fibers.

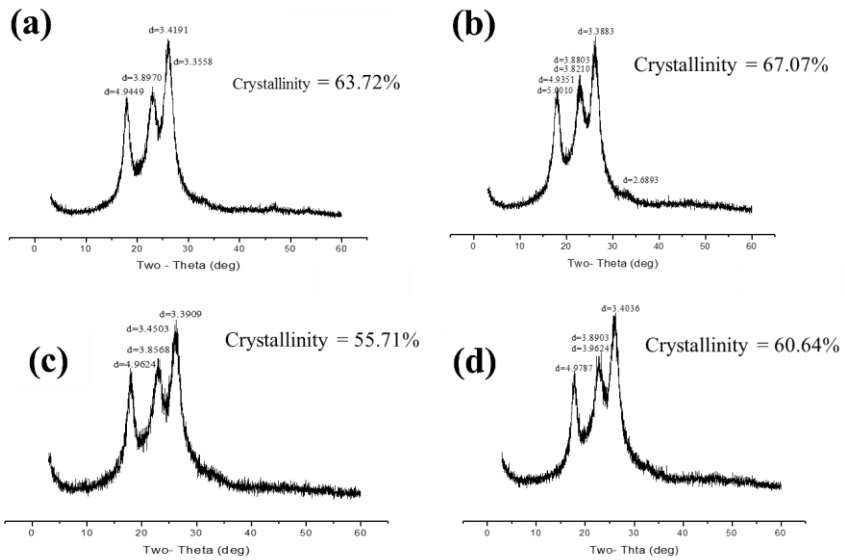

Figure 3. XRD results of UHMWPE fibers, (a) untreated; (b) treated for 3 min O_2/Ar plasma; (c) treated for 3 min O_2/Ar plasma and dyed; (d) treated for 3 min O_2/Ar plasma and printing.

Moreover, AFM analysis gave further information on the plasma induced surface chemical and topographical modifications. **Figure 4** shows AFM images of UHMWPE fibers surface with different process. Figure 4 (a) untreated fiber show a relatively flat surface and Figure 4 (b) the O_2/Ar plasma treated for 3 min, show a small extent of surface roughness and the edges of the hill, uniformly distributed on fiber surface, Figure 4 (c and d) after O_2/Ar plasma treated for 3 min and dyed and printing fibers showed a rather broad and rough wavy surface. The number of ridges on the surfaces is influenced by plasma treatment and dyed. The root mean square roughness (RMS) of the treated UHMWPE fibers was increased in the treated for 3 min O_2/Ar plasma treatment and RMS by 3 min O_2/Ar plasma treatment followed by dyed samples was the most. The surface morphology is also researched using the acquired surface roughness (Rq) values for the different conditions. Significant plasma-induced effect on the surface roughness is found for all examined conditions, as the root mean square values for the treated samples are in the same order for the untreated UHMWPE as it has already been reported that O_2/Ar plasma enhanced radical reactions and restrains electron and ion etching effects.

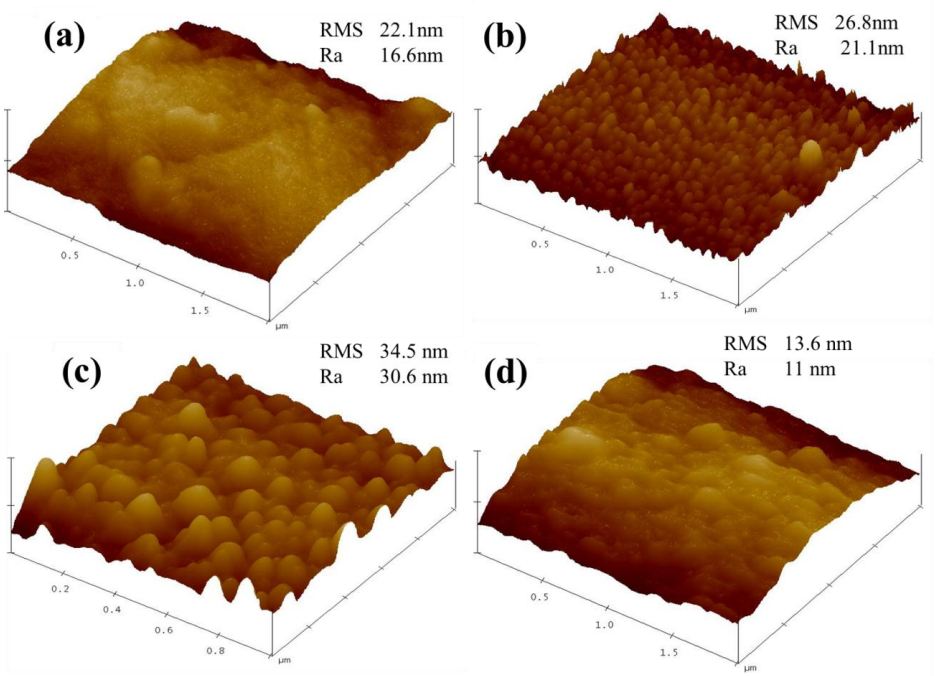

Figure 4. AFM images of UHHMWPE fiber (a) untreated; (b) treated for 3 min O_2/Ar plasma; (c) treated for 3 min O_2/Ar plasma and dyed; (d) treated for 3 min O_2/Ar plasma and printing; and RMS roughness

The untreated and O_2/Ar plasma treated of UHMWPE fibers are put into the dye bath and dyed. The highest temperature is 100 °C, which is much lower than the usual disperse dye bath. The color strength rate was shown in **Table 1**. It is displayed that the color strength rate of the fibers with the disperse dyes increased with the plasma treatment time and then reached the highest after dyed more thanafter print with pigment.

Table 1. K/S values of treatment fibers and after dyeing and printing processes

Time treatment(Min)	Dyeing with disperse		Print with pigment	
	K/S value	% Color strength	K/S value	% Color strength
Untreated	8.0029	0.00	2.9490	0.00
Plasma treated	11.2190	28.67	6.0124	50.95

Effect of using cold plasma on dyeing properties of UHMWPE fiber

The dyeability of hydrophobic fibers, such as the UHMWPE fiber is very poor. It is known that introducing hydrophilic sites on the hydrophobic fibers can improve the dyeability of these fibers. Plasma modifications resulting in unsaturated bonds and/ or free radicals on the surface of the fibers have a significant influence on the overall surface changes and consequently on dyeability. From **Figure 5** it is shown that the low temperature dyeing and printing properties of UHMWPE fiber to disperse Blue 183 have been improved a lot after O_2/Ar plasma treatment for 3 min.

Application of different plasma gasses may result different surface chemical structure so different surface mechanical properties. Different functional groups such as C–O, C=O, O=C-O can be obtained on the polymer surface by oxygen plasma. Addition of inert gas to active gas based plasma provides an increase in oxygen functionality by enhancing radical formation and more effective surface activation.

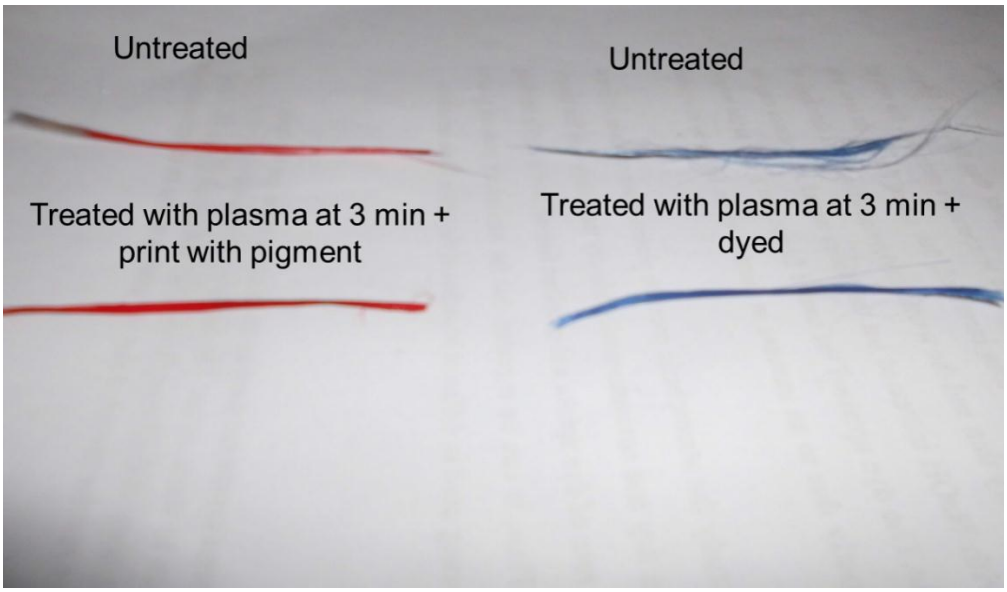

Figure 5: Photographs of dyed and printing properties of UHHMWPE fibers: untreated and O_2/Ar plasma treatment for 3 min.

CONCLUSION

From this study, plasma treatments of textiles look very promising. Can be used in substitution of conventional processes and for the production of innovative textile materials with properties that cannot be achieved via wet processing. In general, no significant alteration of bulk properties is produced. Fast and extremely gentle, as well as environmentally friendly, being dry processes characterized by low consumption of energy.

Studied on argon and oxygen plasma surface modification of UHMWPE fibers they concluded that both plasma surface treatment applications improved the wettability, also if gas mixture is used,

the ratio of gasses in this mixture is important as well. Plasma induced activation of polymer surface, without changing bulk properties generally divided in two groups according to the nature of plasma gasses, as reactive or inert. Oxygen (O_2) is most common active gas for plasma treatment. Besides, carbon dioxide (CO_2) and ammonia (NH_3) are generally used as active plasma gasses. Argon (Ar) is the inert gas that used for plasma treatment.

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