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Oxygen plasma as a pretreatment for environmentally friendly low temperature dyeing of wool natural fiber

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Abstract

The aim of the study was to improve the dyeability of wool fiber with acid dye using a simple environmentally friendly pretreatment with oxygen plasma. Low pressure oxygen plasma treated wool yarns were characterized in terms of morphological, chemical and physical properties by SEM, FTIR and tensile strength measurements. The plasma treated samples were then dyed with an acid dye at different temperatures to compare the results with the untreated yarn. Color measurements were performed to compare the color strength of dyed samples. Fastness properties to washing and light were determined and compared. The plasma treated sample showed better dyeability at lower temperature with good fastness properties.

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Introduction

Wool is a natural protein fiber from the fleece of the domesticated sheep. Wool fiber has a typical core–shell structure consisting of an inner protein core, cortex and surface shell, cuticle. The cuticle consists of several layers. The upper layer, epicuticle, contains lipoproteins. The surface morphology of wool plays an important role in wool processing, since the hydrophobic nature of the cuticle and the high cross-linking density in the outermost fiber surface creates a barrier, which influences sorption properties. Unwanted effects such as felting and the barrier of diffusion are most probably due to the presence of wool scales. So sometimes it is essential to modify the wool surface to improve hydrophilicity, dyeability and anti felting of the resulting goods (Shahidi *et al.* 2013; Kan *et al.* 1998a).

Previously, the modifications of wool surface morphology were conducted by two different methods including chemical degradation of the wool scales and deposition of a polymer layer on the scales. Both processes consume a large amount of chemicals and generate polluted effluents. Due to the increasing awareness of ecological and economical restrictions, the development of environmentally friendly and economical processes is of great demand in research and industry. As a result, low-temperature plasma method was developed rapidly over the last decade and was introduced to the textile industry to replace the conventional processes (Kan *et al.* 1998b, a).

Numerous investigations have indicated that low-temperature plasma treatment can introduce chemical and physical changes on wool surface, and improve processing and performance characteristics of wool fibers. The type of the changes depends on treatment time, pressure and nature of the plasma gas. Kan *et al.* studied the effect of low-temperature plasma (LTP) treatment on the wettability and dyebath exhaustion rate during chrome dyeing of wool. They showed that LTP treatments can alter the dyeing properties to various degrees and the nature of the LTP gases plays an important role in affecting the behavior of chrome dyeing (Kan *et al.* 1998a).

In another study, Wool fabrics were treated with atmospheric pressure helium glow discharge plasma in to improve felting and dyeing behavior with cold brand reactive dyes using cold pad-batch method at neutral pH. The color strength of the plasma treated dyed wool was found to be nearly double of the color strength of dyed untreated wool fabric (Panda *et al.* 2012).

In a previous study, we studied the effect of atmospheric plasma treatment on dyeing of wool with a natural cationic dye, berberine. Plasma treatment improved the absorption of the cationic dye to wool fiber. Compared to untreated samples, plasma treated wool showed better dyeability with good fastness properties against washing, rubbing and light (Haji and Shoushtari 2011).

Gawish *et al* studied the effects of low-pressure pseudo-discharge oxygen plasma and chitosan treatment on the dyeing properties of wool fabrics. Their results have revealed that oxygenated plasma improved the wettability, hydrophilicity and dyeability of the virgin and chitosan-treated wool fabrics, compared with untreated fabrics. Also, it was observed that washing fastness and color fastness to light were improved due to plasma treatment and that the presence of chitosan before or after the plasma exposure has no effect on these properties (Gawish *et al.* 2011).

Wool fibers can be dyed by exhaustion method using acid, mordant, metal-complex and less frequent reactive dyes, respectively. Acid dyes are water-soluble anionic dyes, with a relatively low molecular weight, that are attracted to cationic groups within the wool, primarily the $-NH3^{+}$ groups in acidic pH .

The present work focused on the possibility to improve wool dyeing with acid dye at temperatures lower than those used in conventional processes

(98°C) and without dyeing auxiliaries, by plasma pretreatment.

Experimental

Materials

Wool yarn with linear density of 200 Tex was scoured with 1% non-ionic detergent at 50 °C for 30 min, then dried at ambient temperature. Acid dye (Ariacid Rocceline NF) was obtained from Alvan sabet Co., Iran. All chemicals used in this study were of analytical grade and purchased from Merck. Distilled water was used throughout the work.

Methods

Plasma Treatment: The woolen yarns were pretreated using radio frequency (13.56 MHz) low pressure plasma equipment (model: Junior plasma, Europlasma, Belgium) with oxygen gas. The sample chamber was evacuated to 100 mTor and maintained at this pressure during process. Then, oxygen was introduced with a flow rate of 20 sccm (Standard Cubic Centimeters per Minute). Plasma was generated at 100 W for five minutes. After that, air was introduced into the chamber and the plasma treated sample was removed.

Dyeing: Dyeing of the samples was performed using 1% owf of acid dye in the presence of 5% owf glauber's salt (L:G= 40:1, pH=3). The dyeing was started at 40°C and the temperature was raised to final temperature (70 or 100 °C) at the rate of 2°C per minute. Then the samples remained in that condition for 1 hour, and then rinsed and air dried. All dyeing processes were carried out using a laboratory dyeing machine made by Rissanj co.-Iran.

The exhaustion of the dye was measured at durations of 0, 10, 20, 30, 45, 55, 70, 80 and 90 min, respectively, using Eq. (1) in which the absorbance was measured with a Jenway 6305 UV-Vis spectrophotometer at 530 nm.

$$\%E= [(A_o-A_t)/A_t]*100 \quad (1)$$

Where A_o is the absorbance of dye solution at 0 min, and A_t is the absorbance of dye solution at t min.

Color strength measurements: the reflectance of dyed samples were measured on a Color-eye 7000A spectrophotometer using illuminant D65 and 10° standard observer. Color strength (K/S) of each dyed sample was calculated using kubelka-munk equation:

$$K/S= (1-R)^2/2R \quad (2)$$

Where R is the observed reflectance, K is the absorption coefficient and S is the light scattering coefficient.

Color fastness tests: Color fastness to washing and light was measured according to: ISO 105-C01: 1989(E) and ISO 105-B02: 1994(E) respectively.

ATR-FTIR analysis: Fourier Transform-infrared (FTIR) measurements were carried out using a IRAffinity-1 instrument (Shimadzu, Japan) with a resolution of 4 cm^{-1}. An average of 45 scans was recorded.

Scanning electron microscopy: Scanning electron micrographs were taken on a SIGMA VP field emission scanning electron microscope (FESEM) (ZEISS, Germany) to study the effect of plasma treatment on the surface structure of wool fibers.

Results and discussion

FTIR Studies

FTIR studies were carried out on raw and plasma treated samples to investigate the chemical surface changes after plasma treatment. Fig. 1 shows the FTIR spectra of raw and O_2 plasma treated wool samples. An increase in the intensity of the peaks at 1650 and 1520 cm^{-1} was observed which is related to an increase in -C=O groups after plasma treatment. These functional groups were produced on the surface of the wool fibers as a result of oxidation effect of active species induced by plasma in the gas phase on the wool surface atoms. The increase in oxygen

containing surface groups is responsible for improved wetting and dye absorption of plasma treated wool fibers.

Oxygen plasma can produce bunte salt and cysteic acid residues on wool surface as a result of the cleavage of the disulphide linkage. The formation of bunte salt and cysteic acid on the polypeptide chain provide a polar surface for the wool fibers which can improve its wettability. On the other hand the cleavage of disulphide linkages helps to remove the surface barrier of the fibers. Also cystine monoxide and cystine dioxide residues form on the surface which will generate a more reactive substrate providing more suitable sites for introducing agents like dyes (Kan *et al.* 1999; Fakin *et al.* 2009).

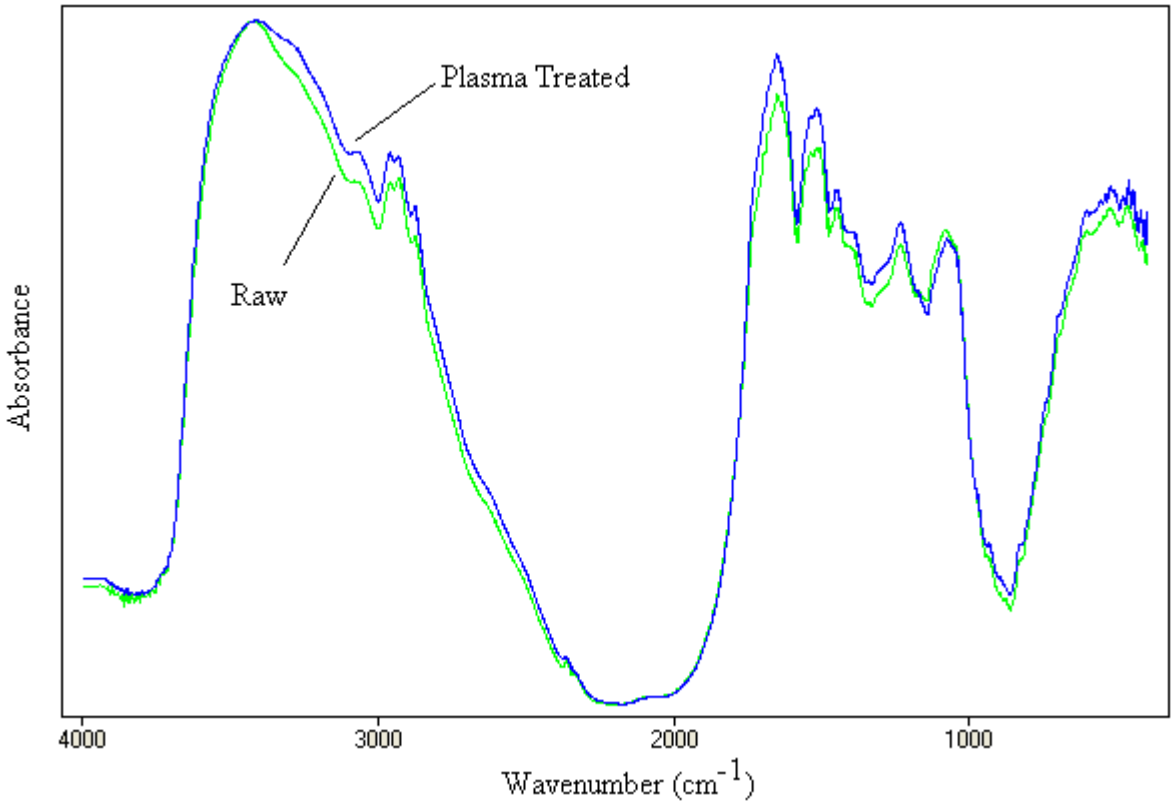


Fig. 1. FTIR spectra of raw and O_2 plasma treated wool samples.

SEM images

Fig. 2 shows the surface morphology of wool fibers before and after plasma treatment. The surface of untreated wool fiber is covered with compact and intact scales, which makes the wool fiber surface difficult to get wet and diffused by dye molecules. However, after plasma treatment, it can be seen that the surface scales of the wool fibers have been destroyed due to the etching effect caused plasma leading to better dyeing of the fibers (Haji and Shoushtari 2011).

Dyeing Properties

The untreated wool fiber had a poor wetting property because of the surface hydrophobic layer and so the dyeability of the untreated wool specimen is worse than that of the plasma treated specimen. As shown in fig. 3, the slope of the curve for the plasma treated sample at the initial stage of the dyeing was steeper than that for the untreated one, implying that the initial rate of dyeing was greater the plasma treated sample. This is due to the dye molecules diffusing relatively faster into the plasma treated fibers as a result of surface chemical and morphological modifications mentioned earlier. The time to reach the final exhaustion was shorter for the plasma

treated sample. The alteration of the fiber surface might provide a pathway for dyestuffs to diffuse into the fiber easily, and hence the dyeing rate of the fabric was improved (Kan *et al.* 1998b).

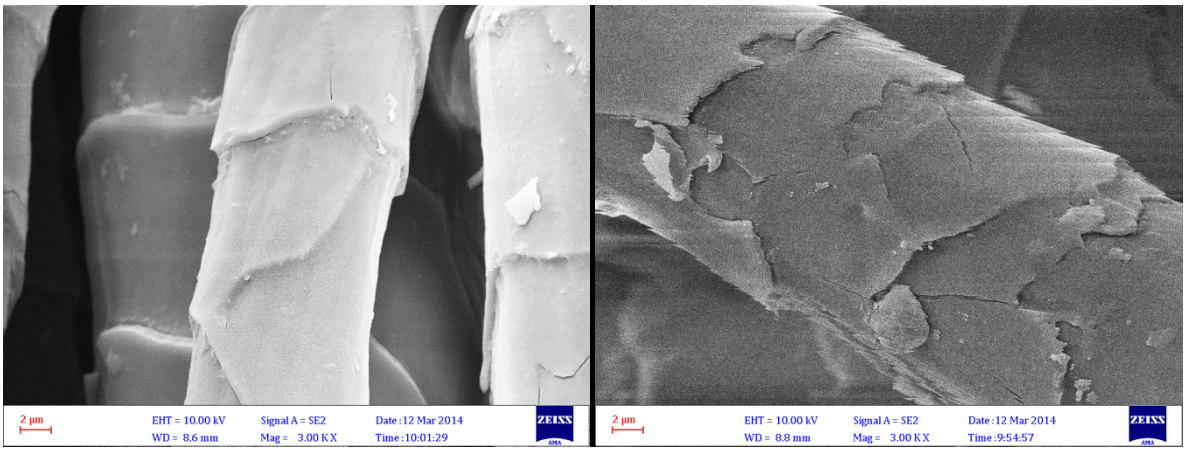


Fig. 2. FESEM images of raw (left) and plasma treated (right) wool fibers.

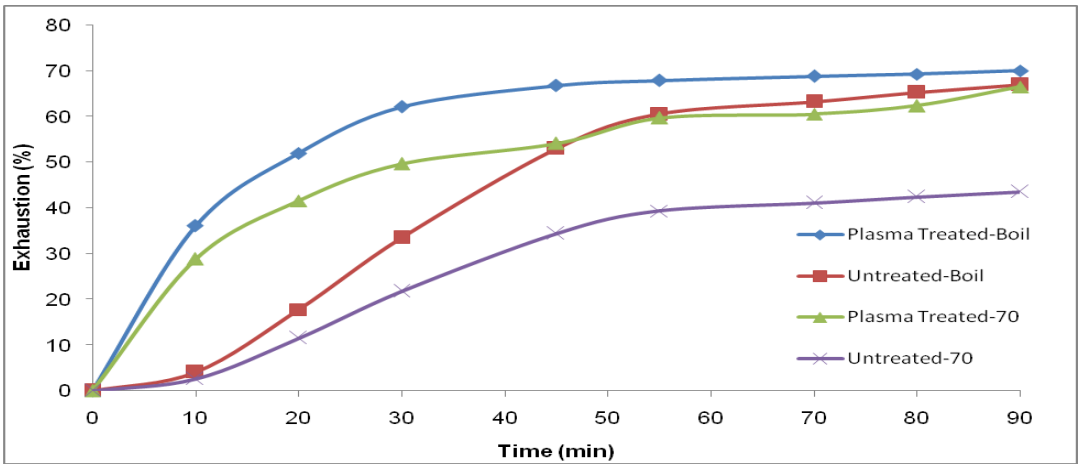
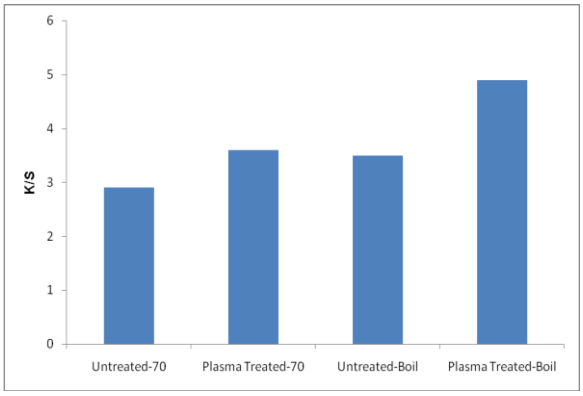


Fig. 3. Exhaustion rates of untreated and plasma treated wool samples

The results show that the exhaustion increased for both samples with increasing the dyeing temperature from 70 ºC to boil. In both temperatures, plasma treated sample absorbed more amount of dye at a faster rate. After 60 min, plasma treated sample dyed at 70 ºC reached the same exhaustion % as untreated sample dyed at boil for 90 min. it means that plasma treatment has lowered the temperature and time needed to reach a similar dye exhaustion compared with untreated fibers.

This can be seen in fig. 4 too. The color strength of the plasma treated sample dyed at 70 ºC is the same as the untreated sample dyed at boil.



Fastness properties

Table 1 shows the fastness properties of dyed samples. All samples have acceptable fastness properties and plasma treatment had no adverse

effect on the fastness properties of samples. The sample dyed at 70 °C after plasma treatment showed better wash fastness compared to untreated sample dyed at the same temperature due to better penetration of the dye into the fibers.

Sample	Wash Fastness	Light Fastness
Untreated-dyed at boil	4	6-7
Plasma treated-dyed at boil	4	6-7
Untreated-dyed at 70 °C	3-4	6-7
Plasma treated-dyed at 70 °C	4	6-7

Conclusion

Low pressure oxygen plasma was applied on wool fibers prior to dyeing with acid dye. The surface changes were studied by FTIR and FESEM. FTIR spectra confirmed the chemical changes of the fibers, while FESEM images showed the etching of the surface scales.

The plasma treated samples showed better dyeability at lower temperature and needed less time to reach the equilibrium. The fastness properties of dyed samples were good. The results showed that plasma treatment can be considered as an easy pretreatment to enhance the dyeability of wool fibers with acid dyes besides economizing in time and energy.

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