Improving the Hydrophilic Properties of Wool Fabrics via Corona Discharge and Hydrogen Peroxide Treatment

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Received 5 April 2008; accepted 20 October 2008
DOI 10.1002/app.29573

ABSTRACT: Corona discharge has been widely applied to modify the surfaces of polymers. In this study, corona discharge was combined with a hydrogen peroxide treatment to improve the hydrophilic properties of wool fabric. Scanning electron microscopy photographs showed that the tip of wool scales was etched after corona discharge and that parts of the scales were peeled off after the hydrogen peroxide treatment. The surface hydrophilic properties of the wool fabric were improved greatly by corona discharge. Increases in the discharge voltage and the number of treatment passages enhanced the hydrophilic properties dramatically, but the improved properties deteriorated with increases in the number of washing cycles and storage time. The hydrogen peroxide treatment could improve the hydrophilic properties and especially the wicking properties of the wool fabric. The fabric became weaker and flexible with an average weight loss of 3% after the hydrogen peroxide treatment. A combination of corona discharge treatment and the hydrogen peroxide treatment made the wool fabric absolutely hydrophilic; the water penetration time of the treated fabric was less than 1 s even when the fabric was washed for several cycles or stored for 6 months. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 112: 1959–1966, 2009

Key words: corona; hydrophilic polymers; modification

INTRODUCTION

As one of the most widely used textile materials, wool is mainly applied in cold-weather clothing because of its excellent insulation qualities, resilience, and felting properties. To widen the application of wool fiber, wool fiber has been considered for close-fitting clothes. Wool fiber can absorb large amounts of water vapor; the moisture regain of wool is about 13.6% under standard conditions. However, water cannot be absorbed by wool fiber because of the water repellence of the surface layer of the wool scales (cuticles).[1] Besides, the scratching effects from the coarse fibers that stick out of the yarn make it difficult to use for close-fitting clothes. Wool fabric, with its fiber cuticle scale layer removed, has good water penetration properties.[2] Meanwhile, knitted fabric made from wool fiber and other fibers such as cotton and polyester can absorb and transfer water and thus can be used for summer clothes.[2] However, to obtain water-absorbing properties by the removal of the scales of wool fiber is not worthwhile because of the high expense of further finishing and the loss of the original properties of wool. Techniques to produce pure wool fabric with good water-absorbent properties are still unavailable.

Corona discharge has been widely used to modify the surface of wool fibers because of its etching effects on the top of scales. It has been applied to improve the shrink and dyeing properties of wool fabrics[3–6] and to pretreat wool fiber for grafting.[7,8] Corona discharge is achieved by the application of a high-frequency voltage between an electrode and an earthed dielectric roll.[6] It is simple and versatile, and no vacuum is needed; thus, it can be commercialized easily. The wettability of wool fiber after corona discharge has been observed, but no further investigation has been performed.[6,9] Previously, we combined corona discharge with an enzyme and resin treatment to change the shrink properties of wool fabrics.[9]

On the other hand, hydrogen peroxide is widely used for the bleaching of wool fabrics.[10,11] During the oxidizing reaction of hydrogen peroxide, hydrogen peroxide converts into the perhydroxyl species (HO_2^-), which attacks the amino acids in the keratins. Cystine is converted into cystine acid, and oxidative bleach agents rupture the disulfide chains.[12,13] After the treatment, wool fiber is bleached to a white color, and its surface is moderately damaged; this may change its hydrophilic properties.

Both corona discharge and hydrogen peroxide modify the wool fiber surface and thus improve the wettability of wool to some extent. However, wool fabric with good hydrophilic properties is still

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Journal of Applied Polymer Science, Vol. 112, 1959–1966 (2009)

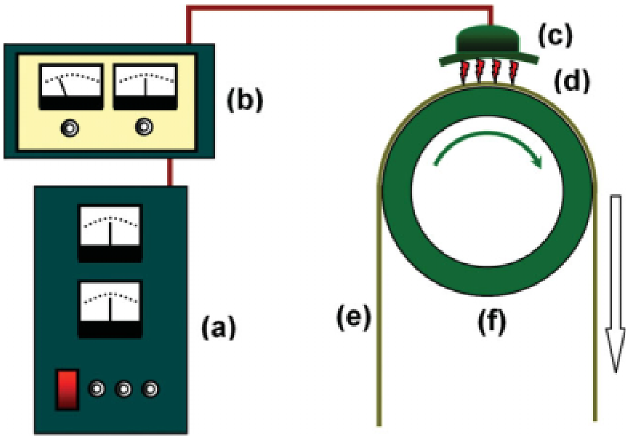

Figure 1 Schematic diagram of the corona discharge treatment: (a) high-voltage generator, (b) controlling system, (c) wire electrode, (d) discharge field, (e) fabric, and (f) poly(vinyl chloride) electrode. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

challenging from both scientific and industrial points of view. In this study, the hydrophilic properties of wool fabrics treated with corona discharge and hydrogen peroxide were investigated in detail. A combination of these two treatments was used to improve the hydrophilic properties of wool fabrics further. This is preparation for exploring close-fitting clothes such as sportswear, shorts, and T-shirts.

EXPERIMENTAL

Materials

Wool twill fabrics (serge, 270 g/m^2) were provided by the Third Wool Factory (Lanzhou, China). Samples for treatment were prepared with dimensions of 35 cm × 15 cm. All the samples were washed in deionized water and then dried and conditioned under conventional conditions (temperature = 20°C, relative humidity = 60%).

Hydrogen peroxide (30%), sodium carbonate, and sodium silicate, all chemical-grade, were acquired from Shanghai Chemical Reagents Co., Ltd. (Shanghai, China).

Corona discharge treatment

The corona discharge treatment was conducted with a 6-kW glow discharge generator (SDCD16-2-10, Dalian Number 9 Electronic, Inc., Dalian, China) with a treatment speed of 2 m/min in the presence of air. As shown in Figure 1, a high voltage was applied between the wire electrode and poly(vinyl chloride) electrode (the gap between the electrodes was 8 mm). A violet light was observed with brushlike discharges treated on the fabric during the treatment. The discharge voltage and the number of treatment passages could be changed on the controlling system. For a conventional treatment, the discharge voltage was set at 13 kV, and the number of passages was three. In the experiments, one parameter was changed while others were kept stable to investigate the effect of the parameter on the hydrophilic properties of treated wool fabric. After the corona discharge treatment, samples were stored in air for different numbers of months to determine the effect of the resident time on the hydrophilic properties of the treated samples.

Hydrogen peroxide treatment

Wool fabrics were treated in an aqueous bath with a liquid-to-fabric ratio of 25 : 1. The hydrogen peroxide concentration was set at 5%, and the solution contained 0.2% (w/w) sodium carbonate and 0.7% (w/w) sodium silicate. The bath temperature was controlled at 50°C, and the treatment time was 1 h. In the experiments, the hydrogen peroxide concentration was changed while other parameters were kept stable to investigate its effect on the hydrophilic properties of the treated wool fabric.

Combination of the corona discharge and hydrogen peroxide treatments

Wool fabrics were pretreated with corona discharge and then treated with hydrogen peroxide. Other wool fabrics were pretreated with hydrogen peroxide and then treated with corona discharge. Procedures for the hydrogen peroxide and corona discharge treatments were set according to the foregoing parameters with a discharge voltage of 13 kV, three passages (corona discharge), and a hydrogen peroxide concentration of 5%.

Measurements and characterizations

Scanning electron microscopy (SEM) analysis was carried out on a field emission scanning electron microscope (Sirion TMP, FEI Co., Hillsboro, OR) at an acceleration voltage of 15 kV. Wool fiber was picked out from the yarn of treated and untreated fabrics and stuck onto the SEM sample holder; all the samples were sputter-coated with a 10–15-nm layer of gold before testing.

After the treatment, the fabrics were placed on a table to test their wettability to water; 0.7 mL of water was dropped with a syringe from 2 cm above the sample. The absorbing time was then recorded by a stop watch when there was no obvious mirror reflection on the wet mark. Every fabric was tested five times, and the results were averaged. The samples were also washed according to the American Association of Textile Chemists and Colorists washing standard[14] for 1, 5, and 10 cycles, and then the wettability was tested.

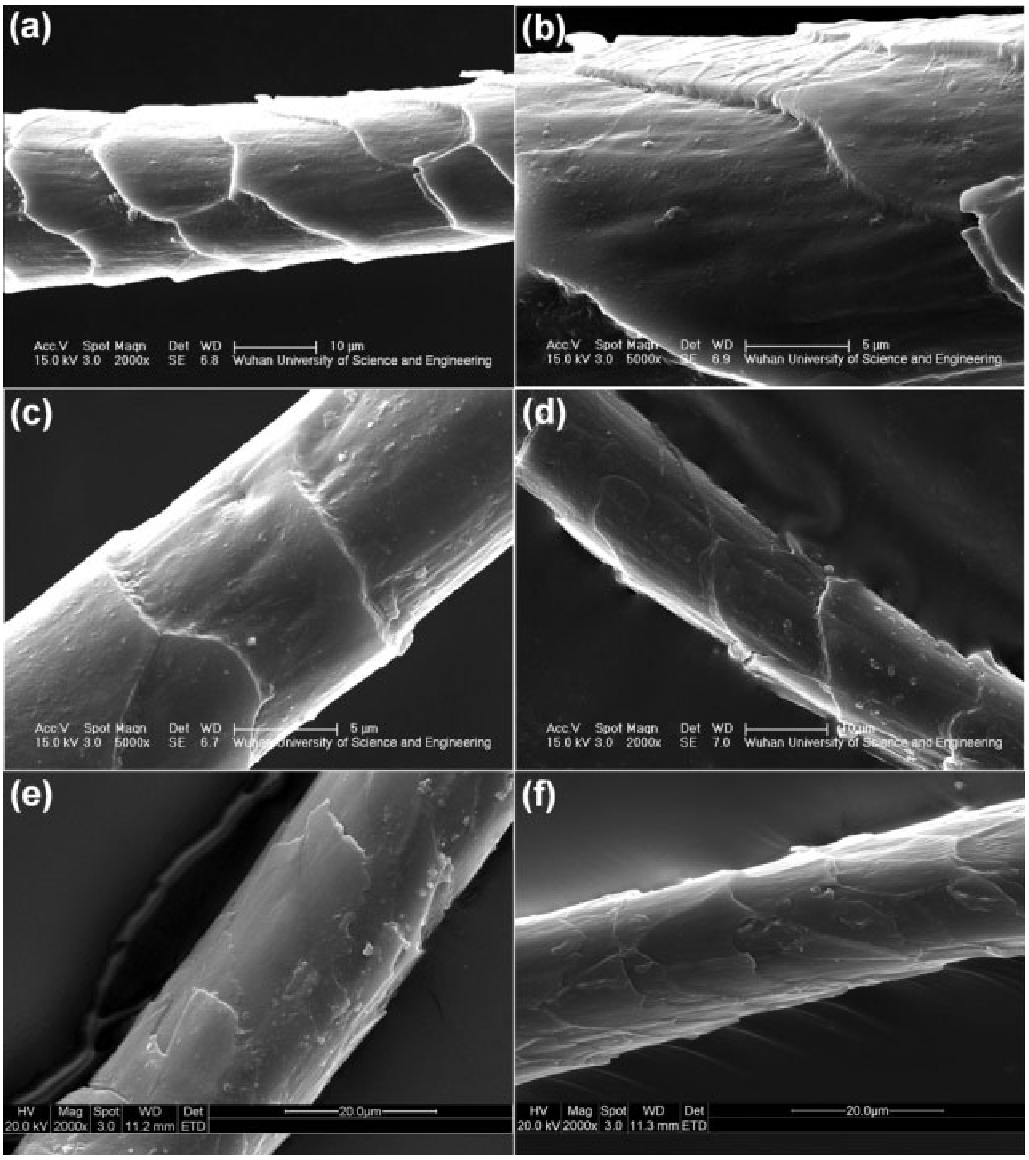

Figure 2 SEM photographs of (a,b) untreated wool fiber, (c) wool fiber treated with corona discharge, (d) wool fiber treated with hydrogen peroxide, (e) wool fiber treated with a combination of corona discharge and hydrogen peroxide, and (f) wool fiber treated with a combination of hydrogen peroxide and corona discharge (corona discharge: 13 kV × three passages; 5% peroxide concentration).

The wicking properties were tested by the vertical suspension of the fabric above a reservoir of distilled water with its lower edge immersed, and the wicking height was measured every minute.[15]

The dry weights of the fabric before and after the hydrogen peroxide treatment were tested, and the weight-loss percentage was calculated as follows:

$$\text{Weight-loss percentage} = \frac{W_0 - W_1}{W_0} \times 100\% \quad (1)$$

where W_0 is the dry weight of the sample before the treatment and W_1 is the dry weight of the sample after the treatment.

The mechanical properties of the treated samples were tested on an Instron 5566 universal testing machine (Datapoint Labs, Ithaca, NY) at a gauge length of 30 mm and a strain rate of 50 mm/min. Samples were obtained from the warp direction of the treated fabrics, the width of the samples was 20 mm, each sample was tested five times, and the results were averaged.

The whiteness of all the samples was tested on a whiteness meter (WSB-II, Wenzhou Instruments and Apparatus Co., Ltd., Wenzhou, China).

RESULTS AND DISCUSSION

SEM observation

Figure 2 shows SEM photographs of untreated wool fiber [Fig. 2(a,b)] and wool fiber treated with corona discharge [Fig. 2(c)], hydrogen peroxide [Fig. 2(d)], and a combination of the methods [Fig. 2(e,f)]. The scale shape of the wool fiber can be observed in Figure 2(a), with the tips of the scales extruding from the surface, as shown in Figure 2(b). As shown in Figure 2(c), the tips of some scales were etched severely without any part extruding from the stem of the fiber. This suggests that corona discharge just slightly modifies the surface of wool fiber with an evident etching effect on the tips of the scales. As shown in Figure 2(d), some scales were peeled off, and others were severely damaged. Obviously, the hydrogen peroxide treatment produces more evident modification of the wool surface in comparison with corona discharge.

With the combination treatments, most of the scales were severely etched and peeled off, as shown in Figure 2(e,f). When the wool fiber was pretreated with corona discharge, most wool scales were etched, and thus the scales were much easier to peel off after the fiber was subjected to the hydrogen peroxide treatment. As shown in Figure 2(e), no evident integral scale could be observed on the fiber. On the other hand, some scales were peeled off and others remained stable when the wool fiber was treated with hydrogen peroxide, as shown in Figure 2(d). After this pretreated fiber was subjected to the corona discharge treatment, the top of the existing scales was easily etched, and the geography of the scales could be observed, as shown in Figure 2(f).

Corona discharge

Fabrics were treated for three passages under different discharge voltages (8, 10, 12, and 14 kV); the water penetration time was tested. After that, the fabrics were washed for different cycles, and the water penetration time was tested accordingly after the washed fabrics were dried. Figure 3 illustrates the hydrophilic properties of wool fabrics treated with corona discharge with different discharge voltages and washing cycles. The water penetration time obviously decreased with the increase in the discharge voltage. This was more evident when the number of washing cycles increased as the water penetration time dropped dramatically (Fig. 3).

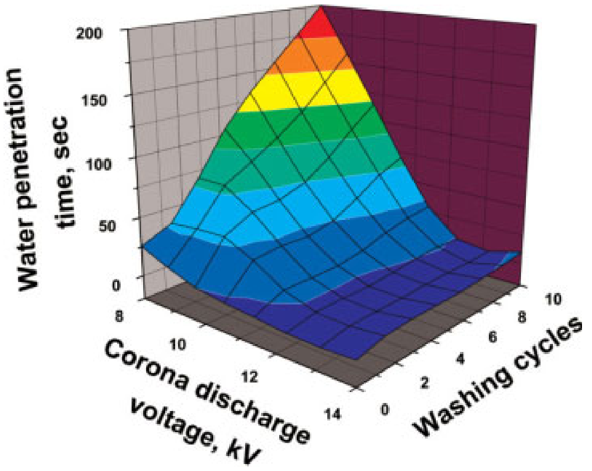

Figure 3 Effect of the discharge voltage on the hydrophilic properties of the treated fabrics with different washing cycles. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Because the water penetration time for untreated wool fabric is more than 1800 s,[9] corona-discharge-treated wool fabric has excellent wettability to water. Three possible reasons can explain this phenomenon. First, the corona discharge modifies the surface structure of wool fibers, the oxidation of which incorporates oxygen atoms into the polymer chains of the wool fibers and thus grafts hydrophilic groups to them. X-ray photoelectron spectroscopy analysis of corona-discharge-treated wool fibers in previous works[6,16] has shown that the content of oxygen increases, and this supports the point. Second, there is a hydrophobic layer on the wool scale (cuticle),[1] which contributes most to the hydrophobic properties of wool fiber. X-ray photoelectron spectroscopy analysis has also shown that the S2p peaks are weaker after corona discharge; this means a decrease in —S—S for the removal of cuticular material from wool fiber.[16] Corona discharge is thought to etch this layer and thus increase the wettability. Third, the etching effects on the tip of the wool scale (as shown in Fig. 2) make water accessible to the stem of the wool fiber, which is hydrophilic because of the large number of polar groups such as OH and CONH. As the discharge voltage increases, more evident surface modification occurs on the surface of the wool fiber, and this enhances the hydrophilic properties of the wool fabric further. However, an excessively higher discharge voltage is not applicable because this would make the treatment too severe and thus some burning marks on the surface of the fabric would be observed.[9]

On the other hand, an increase in the number of washing cycles makes the hydrophilic properties of corona-discharge-treated wool fabrics worse. However, this is not so obvious when the discharge voltage is 14 kV. After 10 washing cycles, the fabric showed a rough and hairy surface, and many fibers extruded outside the yarns. For one thing, the extruded fibers in the surface of the fabrics held the water droplets and repelled the water from penetrating further. Moreover, the fabric was subjected to aqueous processing and deformation during the process, and this might have modified the surface of the fabric and thus weakened the corona discharge effects, as the results show in Figure 3.

The appropriate discharge voltage was determined to be 13 kV, and the number of treatment passages

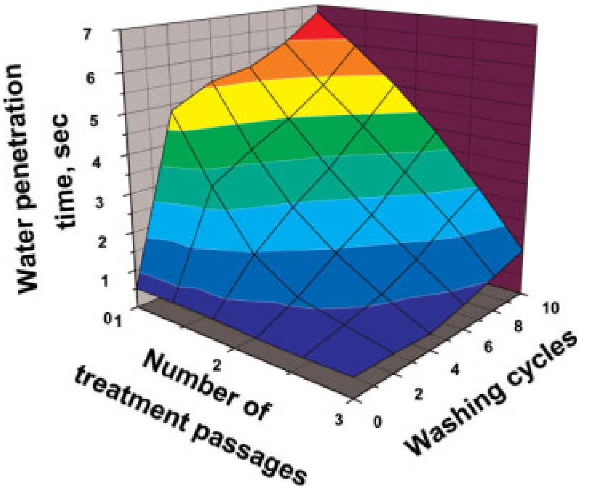

Figure 4 Effect of the corona discharge treatment passage (discharge voltage = 13 kV) on the hydrophilic properties of the treated fabrics with different washing cycles. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

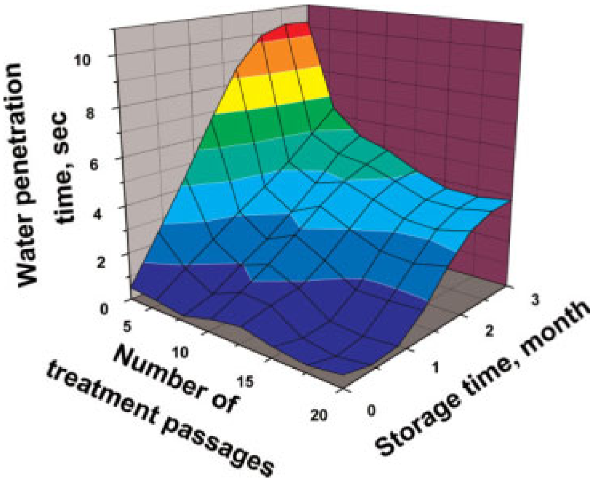

Figure 5 Effect of the storage time on the hydrophilic properties of the treated fabric (discharge voltage = 13 kV; without washing). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

was changed to show the effects on the hydrophilic properties of wool fabric. The hydrophilic properties of the treated fabrics are shown in Figure 4. As the number of treatment passages increased, the hydrophilic properties of the treated fabric improved evidently; the properties changed a little after the fabric was washed. In the process of corona discharge, a brushlike discharge was sprayed onto the surface of the wool fabric and might have caused uneven effects on the surface. As the number of treatment passages increased, to some extent the repeated treatments improved the uniformity of corona discharge irradiation and strengthened the etching effect and the surface modification. Thus, the hydrophilic properties of the fabric were enhanced a little bit.

The hydrophilic properties of corona-discharge-treated wool fabric weakened dramatically when the fabric was stored in air for several months, as shown diagrammatically in Figure 5. The aging problem was observed even though the fabric was treated with 20 passages. When the fabrics were treated with fewer than five passages, the water penetration time increased from less than 1 s to more than 10 s. Because the diffusion of unoxidized low-molecular-weight material species has been reported to be the reason for the recovery of hydrophobic properties of corona-discharge polymers,[17] the diffusion of the unoxidized components on the surface of wool fibers is suggested to be the reason for the deterioration of the hydrophilic properties of the treated samples. Besides, surface reconstruction and reorientation of the hydrophilic groups[18] are possible reasons for the recovery of the hydrophobic properties. The weakening of the properties is not so severe because 10 s is very quick in comparison with the 1800-s penetration time for untreated wool fabric.

TABLE I
Effect of the Hydrogen Peroxide Concentration on the Water Penetration Time, Weight Loss, and Mechanical Properties of Wool Fabrics

Concentration (%)	Water penetration time (s)	Weight loss (%)	Mechanical properties			
			Breaking elongation (mm)	Breaking stress (N)	Breaking energy (J)	Modulus (N/mm)
0	>1800	0	22.39	146.8	2.370	23.64
1	2.03	1.99	30.00	142.4	2.759	16.61
2	1.43	3.70	30.00	139.7	2.641	15.31
3	2.24	0.85	27.67	141.1	2.580	16.19
4	3.05	2.27	27.86	142.0	2.653	16.40
5	3.96	0.56	28.50	143.8	2.623	17.33
6	5.74	2.21	28.17	142.9	2.646	17.44
7	1.12	0.58	30.67	145.6	2.882	15.33
8	1.27	2.98	28.17	144.1	2.530	15.34
9	2.83	0.76	30.00	143.3	2.702	15.29
10	3.29	3.04	29.50	142.9	2.724	16.20

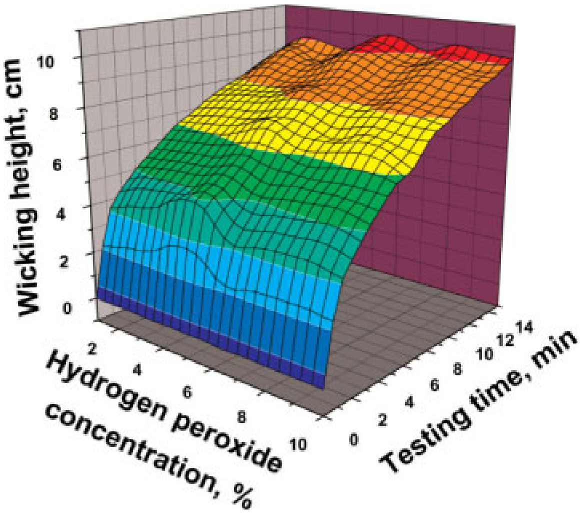

Figure 6 Wicking height of the treated fabric. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Hydrogen peroxide treatment

Wool fabrics were treated with hydrogen peroxide of different concentrations. The hydrophilic properties of the treated fabrics are shown in Table I. The treated fabrics obviously absorbed water much more quickly than the untreated fabric. The time for the treated fabrics to absorb a 0.7-mL water droplet was less than 5 s, whereas the untreated one needed more than 1800 s to absorb the same amount of water. Hydrogen peroxide had oxidative effects on the surface of the wool scale, and the weight of the fabric decreased after the treatment, as shown in Table I. The weight loss fluctuated with increases in the concentration of hydrogen peroxide. Most of the values were less than 3%, and this means that the treatment destroyed just small amounts of the fiber. During the oxidation, the disulfide linkage of cystine and peptide was attacked, the linkage broke somewhat, and the fringe and other parts of the scale were destroyed, as shown in the SEM photograph in Figure 2. The surface layer of the scale was badly destroyed, and this made water accessible to the stem of the fiber and thus enhanced the hydrophilic properties of the fabric.

At the same time, the mechanical properties of the fabrics after treatment are shown in Table I. The values of the mechanical properties fluctuated slightly with the increase in the hydrogen peroxide concentration. In comparison with the untreated sample, the breaking stress and modulus of the treated samples decreased evidently, and this means that the samples were weaker after the hydrogen peroxide treatment. Meanwhile, the elongation at break and breaking energy increased to some extent, and this indicated that the toughness and flexibility of the fabrics increased slightly. As the surface of the wool fiber was modified greatly, much water was easily incorporated into the stem of the fiber. Water might have associated with the —OH-containing hydrophilic amino acid in the keratins of the wool fiber, and more free volume was introduced into the polymer chains, which made the wool fiber more flexible.

Besides, the fabrics treated with hydrogen peroxide showed perfect wicking properties. The wicking height of the treated fabric versus the time is shown diagrammatically in Figure 6; the trends of the curves for all treated fabrics were almost the same with small fluctuations. The wicking height of the untreated fabric increased slowly to about 0.5 cm after 15 min. In comparison, the hydrogen peroxide treated wool fabrics showed excellent wicking properties, and this also suggested the improvement of the hydrophilic properties of the fabrics.

Combination of the corona discharge and hydrogen peroxide treatments

Both the corona discharge and hydrogen peroxide treatments can improve the hydrophilic properties of wool fabrics. However, for the corona discharge treatment, the aging problem and the inability to withstand washing will greatly confine its applications in improving the hydrophilic properties of wool fabrics. As for hydrogen peroxide, the water penetration time is more than 1 s, which is not good

TABLE II
Water Penetration Times of the Treated Fabrics (5% Hydrogen Peroxide, 13-kV Corona Discharge with Three Passages, and a Combination of These Two Treatments) with Different Washing Cycles

	Washing cycles			
Sample	0	1	5	10
Peroxide-treated	<1	2.3	2.8	1.3
Corona-treated and peroxide-treated	<1	<1	<1	<1
Peroxide-treated and corona-treated	<1	<1	<1	<1
Corona-treated	<1	<1	1.1	1.0

TABLE III
Water Penetration Times of the Treated Fabrics (5% Hydrogen Peroxide, 13-kV Corona Discharge with Three Passages, and a Combination of These Two Treatments) After Different Storage Times

	Storage times (months)			
Sample	0	1	2	6
Peroxide-treated	<1	1.2	2.1	1.1
Corona-treated and peroxide-treated	<1	<1	<1	<1
Peroxide-treated and corona-treated	<1	1.1	1.2	1.2
Corona-treated	<1	3.1	5.5	10.7

TABLE IV
Mechanical Properties of Wool Fabrics with Different Treatments

Sample	Mechanical properties			
	Breaking elongation (mm)	Breaking stress (N)	Breaking energy (J)	Modulus (N/mm)
Untreated	22.39	146.8	2.370	23.64
Corona-treated	23.33	145.7	2.294	22.12
Peroxide-treated	28.50	143.8	2.623	17.33
Corona-treated and peroxide-treated	28.50	144.0	2.626	16.67
Peroxide-treated and corona-treated	29.00	141.3	2.656	16.73

enough to explore close-fitting clothes such as sportswear. For this reason, a combined method containing both of these two treatments was tested to show its effect on the hydrophilic properties of wool fabrics.

Two ways were used to treat the fabric: a hydrogen peroxide pretreatment plus a corona discharge treatment and a corona discharge pretreatment plus a hydrogen peroxide treatment. The hydrophilic properties of the fabrics treated with the different methods with different numbers of washing cycles are shown in Table II. It is obvious that the water penetration time of both combined methods was less than 1 s, whereas that of the hydrogen peroxide treated and corona-discharge-treated fabrics was more than 1 s.

Table III shows the effects of the storage time on the hydrophilic properties of wool fabrics treated with the different methods used in this study. After 6 months, just wool fabric treated with the corona discharge pretreatment plus hydrogen peroxide showed a water penetration time of less than 1 s. It seems that the oxidation of the corona discharge treatment might enhance the hydrogen peroxide treatment.

The mechanical properties of wool fabric treated with different methods are listed in Table IV. Obviously, the mechanical properties of the wool fabric did not show much change after the corona discharge treatment. Like the mechanical properties of the wool fabric treated with hydrogen peroxide, the breaking elongation and breaking energy of the wool fabric treated with the combination methods increased greatly, and the breaking stress and modulus decreased slightly.

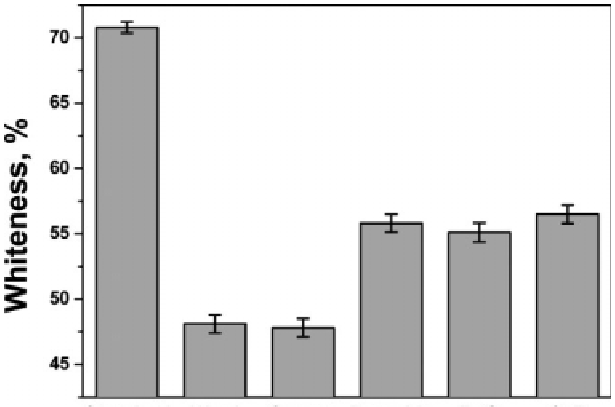

Figure 7 Whiteness of the wool fabric with different treatments (P + C = peroxide + corona; C + P = corona + peroxide).

The whiteness of all the samples is shown in Figure 7. The corona discharge treatment had no effect on the whiteness of the wool fabric; however, the hydrogen peroxide treatment greatly improved the whiteness of the wool fabric because of its bleaching effect. The whiteness of the wool fabric treated with the combined methods showed little difference versus that of the fabric treated with hydrogen peroxide.

CONCLUSIONS

The hydrophilic properties of wool fabric were greatly improved by the combination of the corona discharge and hydrogen peroxide treatments. SEM photographs showed that the tips of the wool scales were etched after corona discharge and that parts of the scales were peeled off after the hydrogen peroxide treatment; this indicated surface modifications of the surfaces of the wool fibers treated with these two methods. The water penetration time of wool fabric decreased to less than 1 s after corona discharge. Increases in the discharge voltage and the number of treatment passages enhanced the hydrophilic properties, but the improved properties deteriorated after the treated fabrics were washed for different cycles or stored for different times. The water penetration time of hydrogen peroxide treated wool fabrics was less than 5 s. The fabrics became weaker and flexible with an average weight loss of 3%. The wicking properties of wool fabrics were dramatically improved after the hydrogen peroxide treatment. A combination of corona discharge and hydrogen peroxide treatments made the wool fabric absolutely hydrophilic; the water penetration time of the treated fabric was less than 1 s even when the fabric was washed for several cycles or stored for

6 months. The fabric subjected to corona discharge pretreatment plus hydrogen peroxide showed the best hydrophilic properties in comparison with the fabrics treated with the other methods in this study. In the future, wool clothing in the summer can be explored on the basis of the excellent hydrophilic properties of wool with this technique.

The authors are grateful to Xiaolin Shen and Fengjuan Li for providing technical assistance.

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