

# Effect of Ozone and Ultrasound on the Fiber Properties of Angora Rabbit

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Received 20 July 2007; accepted 1 April 2008

DOI 10.1002/app.28779

Published online 10 February 2011 in Wiley Online Library (wileyonlinelibrary.com).

**ABSTRACT:** The aim of this study was to investigate the effects of novel treatment techniques (ozone and ultrasound) on the dyeability of Angora rabbit fiber. For this purpose, the effects of the ozonation time and fiber moisture during ozonation on the dyeing properties of Angora rabbit fiber were researched. Also, dyeing was performed conventionally and with the use of ultrasound techniques after ozonation; these were compared in terms

of the color yields. Consequently, it was found that the ozonation process and dyeing with power ultrasound improved the dyeability of Angora rabbit fiber significantly. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 3119–3125, 2011

**Key words:** dyes/pigments; electron microscopy; fibers; FT-IR; modification

## INTRODUCTION

Angora fiber refers to the downy coat and keratinous textile material produced by the long-haired Angora rabbit.<sup>1,2</sup> This fiber is one of the luxury fine fibers, which also include mohair, cashmere, and alpaca. These luxury wools represent only 3% of the world's clean wool production, but their price can be 10–30 times more than that of sheep wool because of their lightness, fineness, softness, image, and reputation.<sup>3,4,42</sup>

Angora fiber has widespread use in health products for arthritis patients and in thermal underwear for cold climates because of its warm nature. However, the bristle content of most Angora fleeces makes them unsuitable for the production of next-to-skin wear or high-quality suit materials. Furthermore, because of difficulties in weaving pure Angora wool caused by its smooth surface and low interfiber friction, Angora fiber is usually blended with another fiber such as wool.<sup>3,5,6,42</sup>

The fibers themselves are very clean, so they do not need scouring. However, in some cases, bleaching processes can be carried out. During processes such as washing, bleaching, and dyeing, these fibers must be processed cautiously to minimize fiber damage. Therefore, Angora wool can be treated at a low temperature and with low mechanical friction.<sup>7–9,42</sup>

On the other hand, new advanced techniques and technologies [treatments with ozone/ultrasound

(US)] for textile industries have been studied by some researchers because of increasingly stringent environmental legislation and much more competitive market demands.<sup>10–13,42</sup> The use of US is relatively novel, and the method is becoming a growing research field because of a wide range of emerging applications in chemical synthesis, therapeutics, environmental protection, electrochemistry, processing of solids and liquids, processing of food, and decolorization.<sup>14–16</sup> Ultrasonic energy is also used in textiles (e.g., dyeing) to speed and develop textile processes.<sup>17</sup> Several articles and studies have appeared in this field that report some savings in energy, chemicals, and processing time for wet textile processes with the use of US.<sup>10,18–25</sup>

The effectiveness of US is lower than that of ozone for bleaching when the frequency of US is taken into consideration.<sup>10</sup> The use of ozone in new advanced techniques is very popular because ozone reacts with organic compounds either directly under acidic/neutral conditions and/or indirectly by decomposition at a highly alkaline pH.<sup>26</sup> Ozone has the second strongest oxidizing power after fluorine (the relative oxidation potentials of ozone and fluorine are 2.07 and 3.06 eV, respectively), and it is advantageous because it can easily break down ingredients that cannot be decomposed with conventional oxidizers.<sup>27</sup> Therefore, it can be used for much faster bleaching of textiles in comparison with hydrogen peroxide and hypochlorite (e.g., its use in the bleaching of wood pulp).<sup>10,11,28</sup>

Dyeing Angora wool at a low temperature and with low mechanical friction is mostly preferred to minimize fiber damage. Therefore, the ozonation

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process, which requires a low temperature,<sup>10,11,29,42</sup> and power US, which improves the efficiency of dyeing, were investigated in this study in terms of the dyeability property of Angora rabbit fiber.

## EXPERIMENTAL

### Material

Angora rabbit fiber (fineness = 16.26  $\mu\text{m}$ , length = 38 mm) was supplied by the Association of Angora Rabbit Breeders in Turkey. The grade of the unfelted fiber was 2 according to Turkish Standard TS 12122. The whiteness degree of the raw Angora rabbit fiber was 49.2 (Stensby).

### Method

In our previous work the effect of ozone on the whiteness degrees of angora rabbit fiber was investigated moreover the effect of ozone on dyeing with milling type acid dyestuffs were searched too.<sup>42</sup> Different from this, here the effect of ozone on the dyeing with leveling type acid dyestuff was investigated. Meanwhile, since the effect of ozone on dyeing with leveling type acid dye is relatively limited compared with the one on dyeing with milling acid dye, additionally the effect of ultrasound during dyeing was investigated too.

First, the effect of the fiber water pickup value [WPV; eq. (1)] during ozonation was researched in terms of dyeability. To this end, fibers were soaked in water at pH 7; after a while, the WPV of the fibers was set at 0, 20, 40, 60, 80, and 100% with squeezing rollers. During these experiments, the ozonation time was set at 45 min. In light of previous studies, it can be said that ozone gas is susceptible to high temperatures. It can decompose in a short time at high-to-medium temperatures; on the other hand, under cold treatment conditions (10–12°C) and at room temperature (23–25°C), nearly the same results have been obtained. Because of this, it was decided to perform the treatments at room temperature.<sup>11,39</sup>

$$\text{WPV (\%)} = \frac{\text{Weight of impregnated fabric} - \text{Weight of greige fabric}}{\text{Weight of greige fabric}} \times 100 \quad (1)$$

After the optimization of the WPV, for investigating the effect of the ozonation time on dyeability, Angora fiber with 60% WPV was ozonated for five different ozonation times (5, 10, 15, 30, and 45 min). The samples were rinsed with water at 50°C for 5 min without the use of any chemicals after all the treatments. In all the experiments, soft mill water (permuted water) was used.<sup>42</sup>

Finally, ozonated and unozonated Angora fibers were dyed with acid dyestuff (C.I. Acid Blue 76). The dyeing was carried out conventionally and with the use of US techniques. These were compared in terms of the color yields. For dyeing, the material was soaked in the solution at 40°C for 10 min, and then the temperature was raised to 70°C in 30 min. At this temperature, the fibers were treated for 30 min. Then, the dye bath was cooled, and the Angora fibers were removed. Then, the dyed samples were rinsed in cold water (5 min), hot water (10 min at 50°C), and cold water (10 min) and dried at the ambient temperature.

The equipment used for the ozone bleaching had three components: the ozone generator, the applicator, and the ozone destroyer. The system was fully closed because of the harmful effects of ozone on health. The capacity of the ozone generator (Lundell Aquametrics, Inc.) was 0–5 L/min. Oxygen was supplied to the generator by an oxygen tube, and the generator supplied the required ozone–oxygen mixture to the applicator. The applicator was a cylindrical glass tube with a diffuser at the bottom. In these experiments, the flow rate of the ozone was adjusted to 1.5 L/min. The flow rate is a parameter depending on the treatment time. It was found that with higher flow rates, nearly the same results were achieved in a shorter treatment time as in a longer treatment time with lower flow rates.<sup>10</sup> Because five different ozonation times (5, 10, 15, 30, and 45 min) were investigated, the flow rate was set at a constant value. The Angora samples were placed in the applicator for the required time. The US treatment of the fibers was performed in an ultrasonic tank (AGS Group Ultrasonic, Ankara, Turkey) with a volume of 28 L. The frequency of the US waves generated by the sonicator plate was 42 kHz.

To evaluate the effects of ozonation and US on the dyeing properties of Angora fibers, samples were measured with a Minolta 3600d spectrophotometer (D65/10°) produced by Konica Minolta Sensing, Inc., and the  $K/S$  values of the color yields of dyed samples were calculated with the Kubelka–Munk equation:  $K/S = (1 - R)^2/2R$  (where  $R$ , is reflectance value in maximum absorption wave length (nm),  $K$ , is absorption coefficient, and  $S$ , is scattering coefficient). The, the  $K/S$  change [ $K/S_C$ ; eq. (2)] of the dyed samples was examined:

$$K/S_C = (K/S_{\text{Treated and dyed}} - K/S_{\text{Dyed}}) \times 100 / (K/S_{\text{Dyed}}) \quad (2)$$

Furthermore, Fourier transform infrared (FTIR) spectroscopy of the Angora fiber was carried out with a PerkinElmer Spectrum 100, and scanning electron microscopy (SEM) photographs of the treated

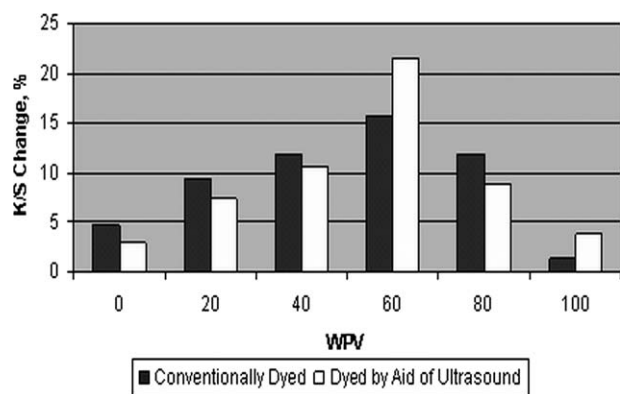


Figure 1 K/S<sub>C</sub> values of the dyed Angora rabbit fibers.

samples were taken to investigate the effects of ozonation and US with a Zeiss Supra 50VP.

Moreover, the moisture absorption/desorption properties of treated Angora rabbit fibers were investigated. For this purpose, fibers were set at 105°C from 30 to 240 min to investigate moisture desorption and at 20°C and 65% relative humidity from 30 min to 24 h to investigate moisture absorption. Weight changes of the fibers were calculated, and these results showed the moisture change [ $M_C$ ; eq. (3)] of the fibers:

$$M_C = \left( \text{Weight}_{\text{Treated}} - \text{Weight}_{\text{intreated}} \right) \times 100 / \text{Weight}_{\text{Untreated}} \quad (3)$$

## RESULTS AND DISCUSSION

### Effect of the fiber WPV on the dyeing properties of Angora rabbit fiber

Nowadays, some literature and research can be cited as evidence that the water content of a material is of great importance in terms of its ozonation efficiency.<sup>10-13</sup> That is why the fibers were impregnated with six different WPVs (0, 20, 40, 60, 80, and 100%) at this stage of experimentation. For investigating the effect of the water content on the ozonation efficiency and thus dyeability of Angora rabbit fiber, the treatment time, ambient temperature, and fiber pH were set to 45 min, 20–25°C, and 7, respectively.<sup>42</sup> To this end, Angora rabbit fibers were ozonated with different WPVs for 45 min, and untreated fibers were dyed conventionally and with US.

Figure 1 and Table I clearly show that performing ozonation process before dyeing caused significant increases in the color yields of Angora rabbit fibers. For example, when ozonation with 60% WPV was performed before dyeing, the K/S value of the conventionally dyed Angora rabbit fiber increased about 16%. This increase was eventually about 22% when dyeing was performed with power US.

It is well known that the WPV of fibers is of great importance during ozonation in terms of an increase in the whiteness degree.<sup>10-13</sup> However, the interesting point is that the WPV value used in ozonation has an important effect on K/S values too. The color efficiency of Angora rabbit fiber ozonated with 60% WPV before dyeing was the highest for both dyeing methods. Figure 1 shows that the K/S value of the ozonated Angora rabbit fiber increased with the rise in the WPV (until 60%). When the WPV increased more than 60%, the K/S value started to decrease gradually. It is essential to identify the WPV for the desired dyeing effect.

The other interesting point concerns the effect of power US on the dyeing properties of Angora rabbit fiber. Although conventional dyeing seems to be a more advantageous method than dyeing with power US in terms of color efficiency according to Figure 1, Table I prevents this misconception. Table I shows that US helps to improve the color efficiency of all Angora rabbit fibers. When conventional and power US dyeing methods were compared in terms of the untreated Angora fiber K/S values, it was seen that US supported the dye uptake of the fibers. The untreated Angora fiber K/S value increased from 3.5 to 4 with the aid of US. This was more explicit in the dyeing results of the ozonated Angora fiber with 60% WPV. The color efficiency of the ozonated Angora fiber with 60% WPV increased from 4 to 4.87 after dyeing with power US.

The intensification and acceleration of mass transport, particularly in the intrayarn pores, are the basis for the improvement of wet textile processes. Mass transport intensification using a conventional approach such as elevated temperatures is not always feasible because of undesired side effects such as fiber damage. The acceleration in dyeing rates observed by many

TABLE I  
K/S Values of the Dyed Angora Rabbit Fibers

Sample specification			
Type of treatment	WPV	Type of dyeing	K/S (630 nm)
Untreated			3.515
Ozonated	0	Conventionally dyed	3.6836
	20		3.849
	40		3.9324
	60		4.0699
	80		3.9309
	100		3.5627
Untreated			4.0105
Ozonated	0	Dyed by aid of US	4.1322
	20		4.3052
	40		4.4407
	60		4.8749
	80		4.3627
	100		4.1683

workers might be the cumulative effect of the aforementioned processes. Dye molecules have a tendency to form aggregates in solution, and this tendency increases with the increasing relative molecular mass of the dye molecule, but higher temperatures cause disaggregation. It is well known that ultrasonic energy causes the decomposition of dye aggregates in solutions, thereby keeping them in a monomolecular state. Cavitation and its physicochemical consequences enhance the dyeing rate and economize energy and time consumption.<sup>22</sup> The results of this part of the experimentation confirm that US enhances the penetration of dye molecules into the fibers.

In summary, US caused a 14–22% improvement in the color efficiency of Angora rabbit fibers. On the other hand, the whiteness of the fabrics increased after ozonation.<sup>10–13</sup> However, it also improved the dyeability of Angora rabbit fibers like US (Fig. 1). The increase in the  $K/S$  values ranged from 3.25 to 22% and was subject to the WPV after dyeing with the help of US. However, this improvement was less after conventional dyeing. The increase in the  $K/S$  values ranged from 2 to 16% and was subject to the WPV.

#### Effect of the ozonation time on the dyeing properties of Angora rabbit fiber

It is important to determine the treatment time for the desired bleaching effect and dyeing properties of ozonation, just like other finishing processes. Angora rabbit fibers were ozonated for 5, 10, 15, 30, and 45 min to investigate the effects of the ozonation time on the color efficiency. During ozonation, all samples were at 60% WPV and pH 7.

The whiteness degree and dyeing properties of Angora rabbit fiber increased with an increase in the ozonation time (Fig. 2). The increase in the whiteness degree was expected with the rise in the ozonation time, as mentioned in some research and in the literature.<sup>10–12</sup>

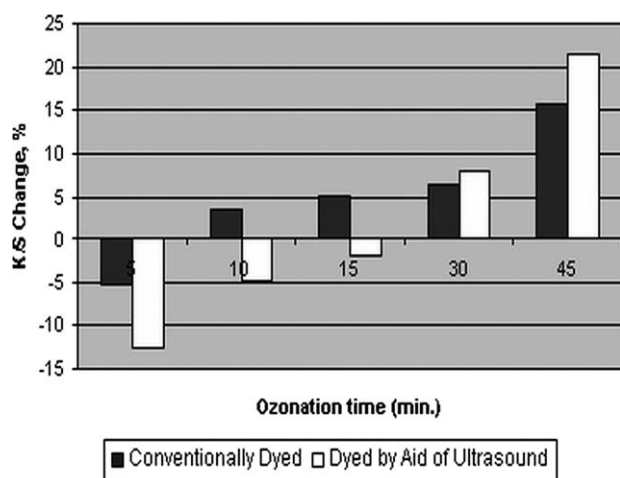


Figure 2  $K/S_C$  values of the dyed Angora rabbit fibers.

TABLE II  
 $K/S$  Values of the Dyed Angora Rabbit Fibers

Sample specification			
Type of treatment	Treatment time (min)	Type of dyeing	$K/S$ (630 nm)
Untreated			3.515
Ozonated	5	Conventionally dyed	3.3165
	10		3.6389
	15		3.6932
	30		3.7412
	45		4.0699
Untreated			4.0105
Ozonated	5	Dyed by aid of US	3.5085
	10		3.815
	15		3.9324
	30		4.3346
	45		4.8749

Figure 2 and Table II show that performing the ozonation process for more than 30 min before dyeing caused a significant increase in the color efficiency of the Angora rabbit fibers. For example, when the ozonation time was extended from 5 to 30 min, the  $K/S$  value of the conventionally dyed Angora rabbit fiber increased about 13%. This increase was eventually about 23.5% when dyeing was performed with power US.

Consequently, the maximum color efficiency was obtained for Angora rabbit fiber that was ozonated for 45 min with 60% WPV and then dyed with the help of US. The reason is thought to be that the cuticle of the Angora rabbit fibers was removed during ozonation. As a result, it is assumed that the rates of moisture absorption and desorption of the ozonated fibers, which are related to the comfort properties of fibers, had improved. Therefore, the moisture absorption/desorption properties of the fibers were investigated too.

In this stage of the experimentation, the behavior of the fibers in terms of moisture desorption at 105°C from 30 to 240 min and in terms of moisture

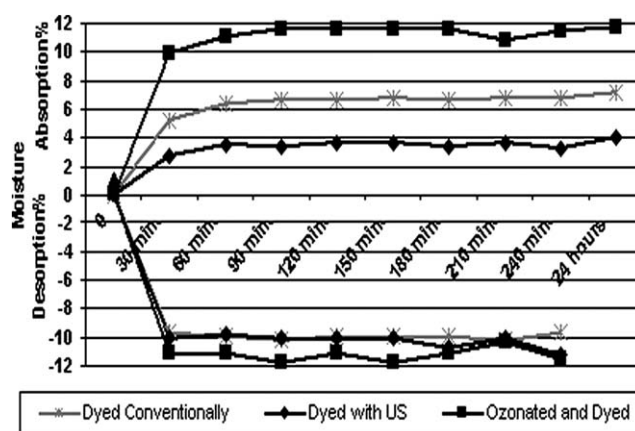
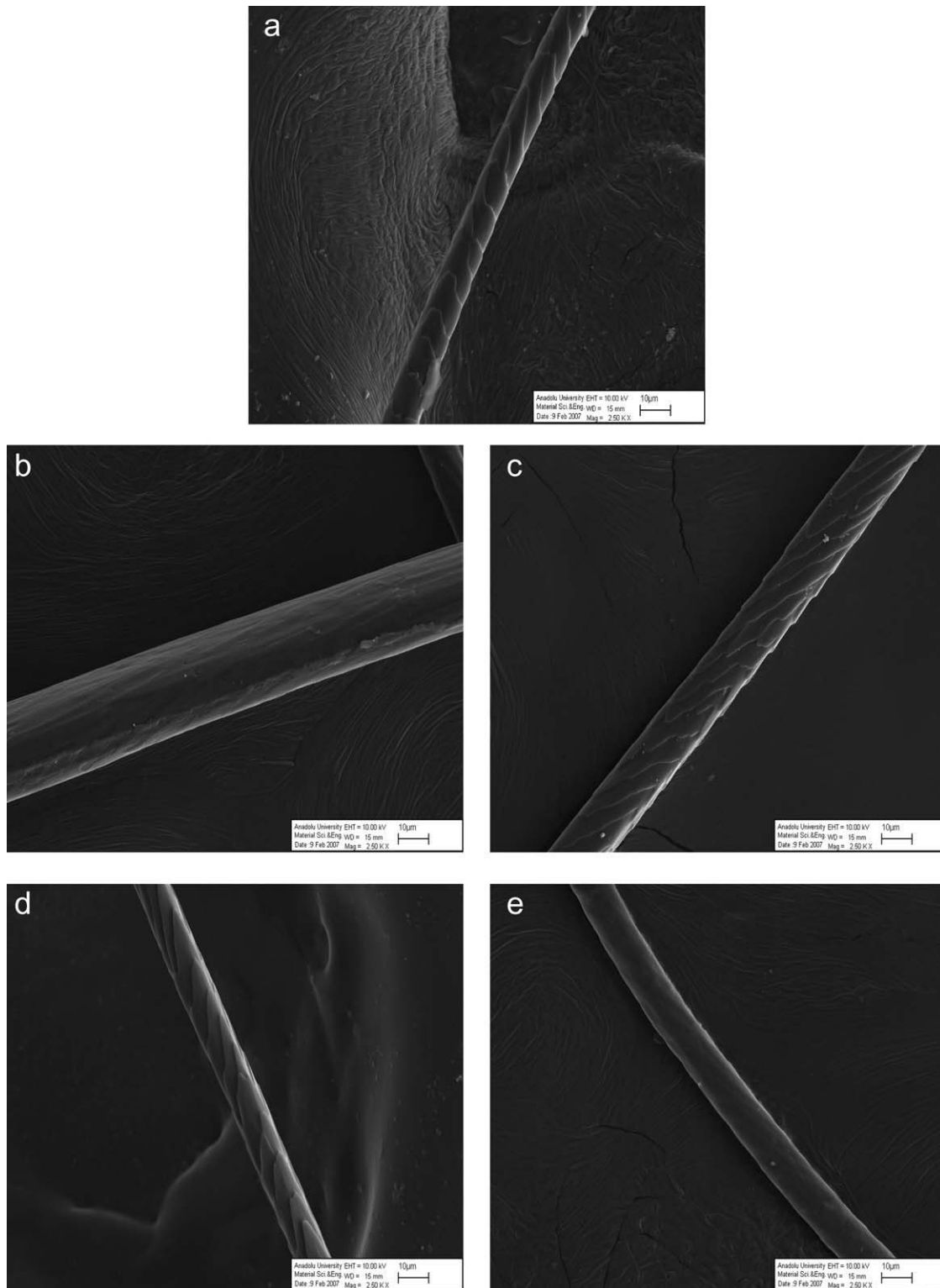


Figure 3 Moisture absorption/desorption properties of the treated Angora rabbit fibers.



**Figure 4** SEM photographs of the Angora rabbit fibers: (a) untreated, (b) ozonated, (c) conventionally dyed, (d) dyed with power US, and (e) ozonated and dyed with power US.

absorption at 20°C and 65% relative humidity from 30 min to 24 h was investigated. Although there were great differences in the absorption of moisture among the tested fibers, the desorption of moisture was not significantly different.

Figure 3 clearly shows that the ozonation process before dyeing caused important changes in the moisture absorption/desorption properties of the Angora rabbit fibers. The reason is the removal of the cuticle layers, which are responsible for the barrier effect

**TABLE III**  
**IR Transmittance Peaks ( $\text{cm}^{-1}$ ) of the Untreated and Treated Angora Rabbit Fibers<sup>27,30–38</sup>**

Untreated fiber		Ozonated fiber		Possible assignment
Dyed with US	Dyed conventionally	Dyed with US	Dyed conventionally	
3278.84	3272.25	3274.57	3278.43	OH stretching
2926.77	2928.56	2929.14	2927.96	C–H stretching
1633.94	1634.88	1634.31	1631.31	Amide I
1516.71	1515.81	1525.90	1525.54	Amide II
1450.73	1449.10	1449.66	1449.17	CH <sub>2</sub> and CH <sub>3</sub> stretching
1232.17	1233.32	1189.03	1191.27	Amide III
—	—	1120	1120	Cystine dioxide
—	—	1070	1070	Cystine monoxide
—	—	1040.75	1041.09	Cysteic acid

during moisture absorption/desorption and also for dye uptake. SEM photographs (Fig. 4) supported this idea, too. As a result, after ozonation, the moisture absorption and desorption of the Angora rabbit fiber became easier.

On the other hand, the treatment with US without ozonation reduced the amount of absorbed moisture by Angora rabbit fiber when the untreated fiber was considered. The results of US treatment depend on cavitation and mechanical effects. It is thought that during US dyeing, unlike ozonation, the cuticle layer of the fiber is not removed; moreover, the scales in the cuticle layer become parallel to the fiber, and a tighter structure occurs as a result of this mechanical effect. This situation shows a significant decrease in the moisture adsorption of Angora rabbit fibers.

### FTIR spectroscopy

The FTIR transmittance spectra of treated Angora wool samples were prepared as a function of the wave number in the range of 650–4000  $\text{cm}^{-1}$ . Angora wool samples were analyzed by the consideration of some chemical groups with FTIR spectroscopy. These groups are presented in Table III.

In all cases, strong absorption peaks were observed at 3278, 2926, 1633, and 1516  $\text{cm}^{-1}$ . The peak at 3278  $\text{cm}^{-1}$  was due to —OH stretching vibrations.<sup>32</sup> The absorption band at 2926  $\text{cm}^{-1}$  was assigned to the asymmetric stretching of C–H. The peaks at 1634 and 1545  $\text{cm}^{-1}$  were assigned to the amide band (H–NCO stretching vibrations). It was clear from the spectrum of the unozonated Angora rabbit fiber that US dyeing caused no significant chemical change in the fiber.

However, the chemical composition of Angora wool undergoes significant changes during ozonation. Cysteic acid is formed after ozonation as a result of the cleavage of the disulfide linkage.<sup>40</sup> The presence of the cysteic acid on the polypeptide chain provides a polar surface for the Angora wool fiber,

which in turn helps to improve the wettability.<sup>35</sup> Furthermore, the cleavage of the disulfide bonds helps to remove the surface barrier of Angora wool fiber.

Apart from the cysteic acid, other interesting cystine residues, cystine monoxide and cystine dioxide, arise after ozonation. Both cystine residues are believed to be intermediate cystine oxidation products (disulfide  $\rightarrow$  monoxide  $\rightarrow$  dioxide  $\rightarrow$  sulfonic acid). Cystine monoxide and cystine dioxide are interesting because they represent more reactive forms than the parent disulfide. The formation of cystine monoxide and cystine dioxide in Angora fiber thus generates a more reactive substrate, which provides a suitable site for introducing agents such as dyes and softeners carrying nucleophilic reactive groups.<sup>35</sup>

Ozonated and dyed Angora fibers with and without US showed some increases in the —OH stretching absorbance. Bands for amide I (1633  $\text{cm}^{-1}$ ), amide II (1516  $\text{cm}^{-1}$ ), and amide III (1232  $\text{cm}^{-1}$ ) were also observed in the spectrum.

The results show that the ozonation process produced a significant increase in the cysteic acid signal at 1040  $\text{cm}^{-1}$  in the FTIR spectra of the ozonated Angora fiber, but no peak was observed in the spectra of the untreated Angora fiber (Table III). Consequently, the ozone oxidized the cystine linkage present on the Angora fiber's surface to cysteic acid, and an improvement in the dyeability and chemical damage was observed.<sup>32–34,41</sup> This is in good agreement with the SEM photographs of the Angora fiber and correlates with the observed significant improvement in the dyeability and moisture conductivity.

### Determination of the damage to Angora rabbit fiber

SEM photographs of the treated Angora rabbit fiber showed the damage to the fiber caused by ozone. Roughness occurred on the cuticle of the ozonated

fiber (Fig. 4). In other words, the outer layer of the Angora rabbit fiber was damaged by ozone. Meanwhile, after dyeing with power US, the cuticle layer of the Angora fiber was removed completely. Also, the cleaning effect of US could be seen in the photographs [Fig. 4(c,d)]. When the Angora rabbit fiber was conventionally dyed, some particles were observed on the fiber surface. However, these were removed with the help of US.

### CONCLUSIONS

In the ozonation process, there was an increase in the dyeability property of Angora rabbit fiber with increasing ozonation time. The ozonation time had to be adjusted according to the required color yields, but a minimum of 30 min was preferred. Also, the effect of WPV on the dyeability property of Angora rabbit fiber was observed. The study showed that the maximum color yields were obtained with ozonation at 23–25°C of Angora rabbit fiber impregnated with water at pH 7 and 60% WPV before dyeing. The reason is thought to be that the cuticle of the Angora rabbit fibers was removed during ozonation. SEM photographs, FTIR analysis, and moisture absorption/desorption behaviors of the fibers supported this result. The amounts of moisture absorbed and desorbed by the fibers improved after ozonation.

According to SEM photographs, ozone processes damaged the outer layer of the Angora fiber surface. Also, the cleaning effect of US was observed. On the other hand, US enhanced the penetration of dye molecules into the fiber. Finally, ozone and US provided good improvements in the color yields of Angora rabbit fiber.

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