Anti-felting Oxidation Treatment of Cashmere Fibers

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ABSTRACT
Cashmere fiber produces felting during laundering because of its scale. In this work, anti-felting treatment of cashmere fibers was investigated using the potassium permanganate oxidizing method, and the optimum oxidizing treatment parameter was obtained through orthogonal experiment. The fibers felting, tensile property, scale morphology, X-ray photoelectron spectroscopy, and directional frictional effect of oxidized cashmere fibers were also tested. Experimental results showed that optimum anti-felting condition of cashmere fiber was 3g/L potassium permanganate ($KMnO_4$) for 20min under the condition at temperature 50℃ and pH3. The felting assembly volume of oxidized cashmere decreased. XPS test showed that hydroxyl group (-OH) content of oxidized cashmere fiber lowed.

Keywords: cashmere fibers, anti-felting treatment, oxidation

INTRODUCTION
Scales of animal fibers are believed to be the major contributor to the felting shrinkage of products made from these fibers [1]. Cashmere fiber, as a special animal fiber, exhibits soft handle and good elasticity. However, it has felting property because of its scale, which influences appearance and dimensional stability of cashmere fabric. The dimensional stability of textile goods influences the comfort and look after repeated laundering of such articles [2]. Moreover, Schlink [3] reported that knitted fabric from low felting wool was more resistant to pilling and shrinkage than fabric from high felting wool. So, it is necessary to decrease felting shrinkage of cashmere fiber.

The mechanism of fiber felting is very complicated and findings from literatures are not always consistent. In general, felting is a form of tangling produced by the persistent rootward migration of the individual fibers, which is caused by the directional frictional effect of fibers [4, 5]. No felting is produced without directional frictional effect [6]. The scale structure of wool fiber is the major component responsible for the tendency of wool goods to undergo felting shrinkage. During laundering, wool fibers experience small movements in the direction of the fiber root, as a consequence of the difference in coefficient of friction of fibers in the 'with-scale' or 'against-scale' directions. This progressive entanglement mechanism ultimately results in felting [2]. Studies on wool fiber anti-felting treatment have been reported widely. One of the most important developments in shrink-resist or anti-felting treatments for wool has been the Hercosett process. This process, which involves a mild chlorination treatment to modify the surface properties of the wool fibers followed by the application of a cationic polyamide, has led to the availability of machine-washable wool garments [7]. Proteases (such as subtilisins) have been employed in the industrial treatment of wool goods to impart desirable properties, such as improving handle properties and imparting shrink-resistance [8]. Chitosan, a polysaccharide that exists as a cationic polyelectrolyte in acid solution has been examined as a possible alternative to the synthetic polyelectrolytes currently used in anti-felting treatments [9]. An alternative method involves coating of the wool fibers using a polymer or monomers that are polymerized on the fiber surface, masking the scale structure of the fibers [10]. Such methods achieve a significant level of shrink-resistance to wool textiles, but may affect adversely the handling properties, as well as generating damaging substances that may be released into the environment. As an oxidizing agent, potassium permanganate ($KMnO_4$) is nontoxic and usually used for sterilization in medicine. In this work, potassium permanganate ($KMnO_4$) was selected to oxidize the scale of cashmere fiber, and the anti-felting property, strength and scale morphology of oxidized cashmere fiber were tested.

EXPERIMENTAL

Material

China white cashmere was selected for the experiment. The anti-felting cashmere was supplied by Inner Mongolia China-Garment Co. Ltd. The micro fineness of the cashmere was 15.6μm.

Oxidizing Treatment

The process of potassium permanganate ($KMnO_4$) oxidizing cashmere fiber is as follows:

Cashmere →potassium permanganate ($KMnO_4$) oxidizing → washing (three times with water at 30℃) → reduction → washing (three times with water at 30℃) →drying

The Orthogonal experiment method was used to get optimum potassium permanganate ($KMnO_4$) oxidizing parameters. The orthogonal parameters are showed in *Table I*. Oxidizing treatment bath ratio was 1:50. Reduction treatment condition was 30g/L sodium sulfite (Na_2SO_3) and 20 ml/L acetic acid (CH_3COOH) for 15min at 45℃.

5g cashmere was used for each treatment, and dried in an oven of 85℃ for 2h. Then the properties of treated cashmere were tested.

Fiber Felting Test

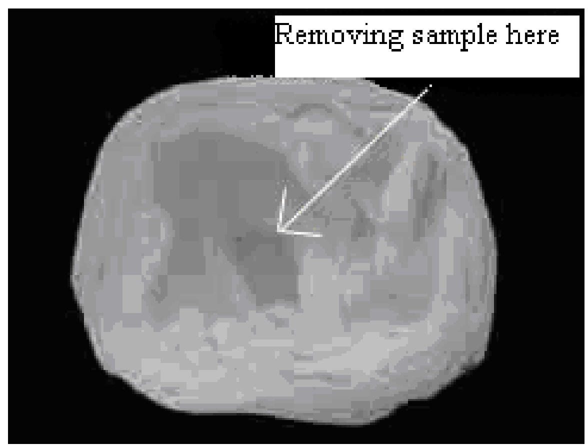
The treated cashmere was placed into a 0.5g plastic bottle (65mm×65mm×85mm), then poured felting agent solution 50ml at 40℃ into the bottle, and fixed the bottle cap. Put the bottle into a wooden box. The box rotates at 60rounds/min for 30min. Then cashmere sample was poured out from the bottle, and dried in an oven.

The felting cashmere assembly volume was tested as follows:

A paper napkin was changed into paper pulp and the dried cashmere sample was covered by the paper pulp *Figure* 1(a). Then put the cashmere sample covered by paper pulp into an oven for drying. Removed the sample from the dried paper pulp, and a model was created *Figure* 1(b). Poured sand (density ρ) into the model and then the weight of sand (*m*) was measured. 10 tests were done for each model. Mean weight ($\bar{m}$ =*m*/10) of sand was determined. The felting cashmere assembly volume (*V*) was calculated.

$$V = \frac{\bar{m}}{\rho} \tag{1}$$

(a) dried paper pulp covering felting sample



(b) model after removing felting sample

FIGURE 1. Model for felting sample test.

TABLE I. Orthogonal parameters.

Factor	Level 1	Level 2	Level 3
Temperature（A），(℃)	40	45	50
Time（B），(min)	10	15	20
potassium permanganate ($KMnO_4$) content（C），(g/L)	1	3	5
pH（D）	3	5	7

Tensile Properties

The single fiber tensile properties of cashmere fiber were tested using a YG001 single fiber tensile tester. The test length of fiber was 10 mm; tensile speed 10 mm/min. 100 fibers were tested for each result.

Scale Morphology

The scale morphology of oxidized cashmere fiber was observed using a JSM-5800 scanning electronic microscope.

X-Ray Photoelectron Spectroscopy

The X-ray photoelectron spectroscopy (XPS) of cashmere fibers was tested using a PHI-5400 instrument. In fixed analyzer transmission mode, the photoelectron line indicated pass energy of 89.45eV.

RESULTS AND ANALYSIS ORTHOGONAL EXPERIMENT

The results of the oxidizing orthogonal experiment are showed in *Table II*. Based on reference [11], the object analysis results for felting assembly volume and strength of fibers are showed in *Table III*.

TABLE II. Results of orthogonal experiment.

Experimental No.	Factor and Level				Felting assembly volume(mm^3)	Fiber strength(cN)
	A	B	C	D		
1	1	1	1	1	10.61	4.25
2	1	2	2	2	12.16	3.85
3	1	3	3	3	14.84	3.55
4	2	1	2	3	12.69	3.53
5	2	2	3	1	14.99	3.76
6	2	3	1	2	10.61	4.74
7	3	1	3	2	13.80	3.46
8	3	2	1	3	9.53	4.59
9	3	3	2	1	15.65	4.17

TABLE III. Object analysis result.

Factor	Result of felting assembly volume	Result of fiber strength
A	k_{1A}=12.537 k_{2A}=12.763 k_{3A}=12.993 Max difference=0.456	k_{1A}=3.883 k_{2A}=4.010 k_{3A}=4.073 Max difference=0.190
B	k_{1B}=12.367 k_{2B}=12.227 k_{3B}=13.700 Max difference =1.473	k_{1B}=3.747 k_{2B}=4.067 k_{3B}=4.153 Max difference =0.406
C	k_{1C}=10.250 k_{2C}=13.500 k_{3C}=14.543 Max difference =4.293	k_{1C}=4.527 k_{2C}=3.850 k_{3C}=3.590 Max difference =0.937
D	k_{1D}=13.750 k_{2D}=12.190 k_{3D}=12.353 Max difference =1.560	k_{1D}=4.060 k_{2D}=4.017 k_{3D}=3.890 Max difference =0.170

According to the reference [11], the order influencing felting volume of fiber is: potassium permanganate ($KMnO_4$) content ＞pH＞time＞temperature. The order influencing strength of fiber is: potassium permanganate ($KMnO_4$) content ＞ time ＞ temperature ＞ pH. So, potassium permanganate ($KMnO_4$) content is a main factor influencing felting volume and strength of oxidized cashmere fibers.

Only considering felting volume of oxidized cashmere fiber, the optimum oxidizing condition is 3g/L potassium permanganate ($KMnO_4$) for 20min under the condition of 50℃ and pH3. Only considering the strength of oxidized fiber, the optimum oxidizing condition is 1g/L potassium permanganate ($KMnO_4$) for 20min under the condition of 50℃ and pH3. So, the optimum oxidizing condition is different for each index. However, the two indexes of oxidized cashmere must be considered simultaneously. Therefore, the fuzzy judging method is used to get optimum conditions. Based on Fuzzy analysis [12], the felting assembly volume and fiber strength is changed into related index. The results are listed in *Table IV*.

TABLE IV. Result of fuzzy analysis.

Experimental No.	Felting assembly volume	Fiber strength
1	0.678	1.897
2	0.777	0.812
3	0.948	0.749
4	0.811	0.745
5	0.958	0.793
6	0.678	1
7	0.882	0.730
8	0.609	0.968
9	1	0.880

Using Eq. (3) and Eq. (4), the matrix of each experimental level is obtained.

$$e_{ij} = \frac{\sum Y_{ij}(k)}{l}, (i = 1,2,...,m; j = 1,2,...,n; k = 1,2,...,K) \quad (3)$$

Of which, $Y_{ij}(k)$ is the related index for k level of different factors. k is experiment level number. m is index number.

Mean level matrix for different factors is following as:

$$E = \begin{pmatrix} e_{11} & e_{12} & ... & e_{1K} \\ e_{21} & e_{22} & ... & e_{2K} \\ ... & ... & ... & ... \\ e_{m1} & e_{m2} & ... & e_{mK} \end{pmatrix} \quad (4)$$

So, the mean level matrix for A factor is:

$$E_A = \begin{pmatrix} 0.801 & 0.816 & 0.830 \\ 0.819 & 0.846 & 0.859 \end{pmatrix}$$

The mean level matrix for B factor is:

$$E_B = \begin{pmatrix} 0.790 & 0.781 & 0.875 \\ 0.790 & 0.858 & 0.876 \end{pmatrix}$$

The mean level matrix for C factor is:

$$E_C = \begin{pmatrix} 0.655 & 0.863 & 0.929 \\ 0.955 & 0.812 & 0.757 \end{pmatrix}$$

The mean level matrix for D factor is:

$$E_D = \begin{pmatrix} 0.879 & 0.779 & 0.789 \\ 0.857 & 0.847 & 0.821 \end{pmatrix}$$

Based on Eq. (5), above mean level matrix for different factors are normalized, and the membership of three indexes for different factors is obtained. Formed fuzzy judging matrixes are following as:

$$T = \begin{pmatrix} \frac{e_{11}}{\sum_{j=1}^{K} e_{1j}} & \frac{e_{12}}{\sum_{j=1}^{K} e_{1j}} & ... & \frac{e_{1K}}{\sum_{j=1}^{K} e_{1j}} \\ \frac{e_{21}}{\sum_{j=1}^{K} e_{2j}} & \frac{e_{22}}{\sum_{j=1}^{K} e_{2j}} & ... & \frac{e_{2K}}{\sum_{j=1}^{K} e_{2j}} \\ ... & ... & ... & ... \\ \frac{e_{m1}}{\sum_{j=1}^{K} e_{mK}} & \frac{e_{m2}}{\sum_{j=1}^{K} e_{mK}} & ... & \frac{e_{mK}}{\sum_{j=1}^{K} e_{mK}} \end{pmatrix} \quad (5)$$

$$T_A = \begin{pmatrix} 0.327 & 0.333 & 0.339 \\ 0.324 & 0.335 & 0.340 \end{pmatrix}$$

$$T_B = \begin{pmatrix} 0.323 & 0.319 & 0.358 \\ 0.313 & 0.340 & 0.347 \end{pmatrix}$$

$$T_C = \begin{pmatrix} 0.268 & 0.353 & 0.380 \\ 0.378 & 0.322 & 0.300 \end{pmatrix}$$

$$T_D = \begin{pmatrix} 0.359 & 0.318 & 0.323 \\ 0.339 & 0.336 & 0.325 \end{pmatrix}$$

Based on weight vector and judging matrixes, the results of fuzzy comprehension judgment vectors for different factors are obtained.

$$M_A = W \cdot T_A = (0.5268 \quad 0.4732) \cdot \begin{bmatrix} 0.327 & 0.333 & 0.339 \\ 0.324 & 0.335 & 0.340 \end{bmatrix}$$
$$= (0.3256 \quad 0.3339 \quad 0.3395)$$
$$M_B = W \cdot T_B = (0.5268 \quad 0.4732) \cdot \begin{bmatrix} 0.323 & 0.319 & 0.358 \\ 0.313 & 0.340 & 0.347 \end{bmatrix}$$
$$= (0.3183 \quad 0.3289 \quad 0.3528)$$
$$M_C = W \cdot T_C = (0.5268 \quad 0.4732) \cdot \begin{bmatrix} 0.268 & 0.353 & 0.380 \\ 0.378 & 0.322 & 0.300 \end{bmatrix}$$
$$= (0.3200 \quad 0.3383 \quad 0.3421)$$
$$M_D = W \cdot T_D = (0.5268 \quad 0.4732) \cdot \begin{bmatrix} 0.359 & 0.318 & 0.323 \\ 0.339 & 0.336 & 0.326 \end{bmatrix}$$
$$= (0.3495 \quad 0.3265 \quad 0.3239)$$

It is indicated that optimum parameters are M_{A3}, M_{B3}, M_{C3}, and M_{D1}. Therefore, the optimum condition of anti-felting treatment for cashmere fiber is 5g/L potassium permanganate ($KMnO_4$) for 20min under the condition of 50℃ and pH3.

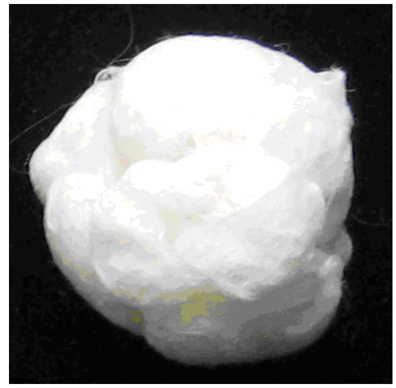
The properties of oxidized cashmere fiber under optimum oxidizing condition are showed in *Table V*.

TABLE V. Comparison of cashmere properties.

Sample	Felting assembly volume(mm^3)	Fiber strength(cN)
Cashmere	8.10	4.22
Oxidized cashmere	15.65	4.17

Fiber Felting Assembly Morphology

The fibers felting assembly morphology was photographed using a camera. *Figure* 2 shows the fibers felting assembly morphology. It is shown that the felting of oxidized cashmere fiber is poorer than that of cashmere fiber. The felting assembly of cashmere fiber is round, but oxidized cashmere fiber is irregular. The directional frictional effect of oxidized cashmere fiber is less than that of cashmere fiber. So, oxidized cashmere fiber has anti-felting property.



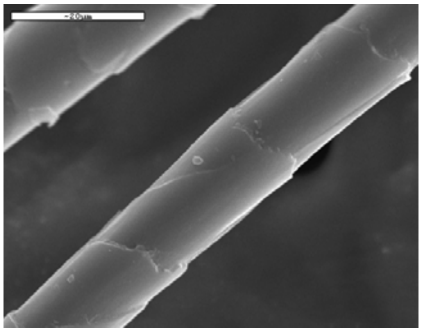
(a) cashmere felting assembly

(b)oxidized cashmere felting assembly

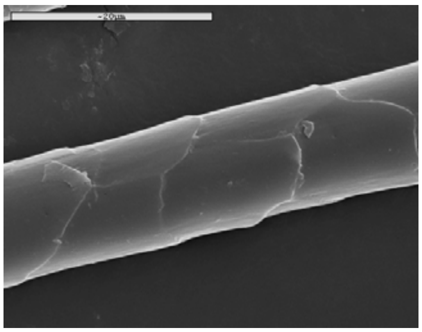
FIGURE 2. Fiber felting assembly morphology.

Fiber Scale Morphology

The scale morphology of cashmere fiber is regarded as a basic characteristic of cashmere content test in textile goods. So, the scale of oxidized cashmere fiber cannot be damaged seriously. The scale morphology of cashmere fiber and oxidized cashmere fiber are showed in *Figure* 3. Oxidized cashmere fiber has clear scale morphology, but the scale edge is irregular because of oxidizing action.



(a) scale morphology of cashmere



(b) scale morphology of cashmere treated

FIGURE 3. Scale morphology.

Fiber Xps Test

Through XPS testing, Carbon element content and its chemical bond type of the oxidized cashmere fibers are shown in *Table VI*. Compared with cashmere fiber, carbon-carbon bonds (C-C) content of oxidized cashmere is higher.

Oxygen element content and its chemical bond type are shown in *Table VII*. Oxygen anion (O^{2-}) content of oxidized cashmere is higher than that of cashmere fiber. However, hydroxyl group (-OH) content of oxidized cashmere is lower. The hydroxyl group (-OH) on the surface of fiber can absorb moisture. When a fiber absorbs moisture, the moisture diffuses into the fiber, causing the molecules to separate and fiber volume to increase, which is called swelling [1]. The stiffness of fiber reduces as a result. The absorbed water molecules act as a lubricant, reducing the coefficient of with-scale friction, and fiber migration will be easier [13]. So, the felting of oxidized cashmere fiber decreases.

TABLE VI. Carbon element chemical bond.

Chemical bond	Chemical bond content (wt%)	
	Cashmere	Oxidized cashmere
O-C=O	7.14	5.76
C=O	4.24	3.39
C-O	8.50	5.70
C-C	80.12	85.15

TABLE VII. Oxygen element chemical bond.

Chemical bond	Chemical bond content (wt%)	
	Cashmere	Oxidized cashmere
-OH	87.4	85.77
O^{2-}	12.5	14.23

CONCLUSIONS

Potassium permanganate content is a main factor that influences the anti-felting property of oxidized cashmere fibers. Under the optimum treating condition, the felting assembly volume and the directional frictional effect of oxidized cashmere are lower than those of cashmere fibers. Oxidized cashmere fiber has a clear scale structure. Hydroxyl group (-OH) content of oxidized cashmere is lower compared with cashmere fiber. Oxidized cashmere fiber has anti-felting property.

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