

Chan Liu<sup>1</sup>,  
Xuzhong Su<sup>1,2,\*</sup>,  
Chunping Xie<sup>1</sup>,  
Xinjin Liu<sup>1</sup>

# Effects of Optimal Mordanting on the Bleaching and Spinning of Black Yak Fibres

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<sup>1</sup>Jiangnan University,  
Key Laboratory of Eco-Textile,  
Ministry of Education,  
Wuxi 214122, P. R. China  
\*E-mail: [suxuzhong@jiangnan.edu.cn](mailto:suxuzhong@jiangnan.edu.cn)

<sup>2</sup>Wuhan Textile University,  
National Engineering Laboratory for Advanced  
Textile Processing and Clean Production,  
State Key Laboratory for Hubel,  
Wuhan 430200, P. R. China  
\*E-mail: [wxxchp@vip.163.com](mailto:wxxchp@vip.163.com)

## Abstract

*In this paper, the orthogonal analysis method was adopted to analyse the optimal mordanting condition for the bleaching of black yak fibres. Without changing the subsequent oxidative/reducing bleaching technology, the optimal mordanting condition was as follows: per weight of water, the ferrous ion concentration is 16%, the sodium bisulfate concentration 3 g/l, the mordanting temperature 60 °C and the liquor pH value is 2. Based on the optimal mordanting condition, yak fibres were bleached. The properties of bleached and untreated yak fibres and yarns were tested by a fibre tensile tester, scanning electron microscopy, yarn strength tester, USTER Tester 5 and so on. By comparing the properties of untreated and bleached yak fibres and yarns, the effects of optimal mordanting on the bleaching and spinning of black yak fibres were presented. The results show that bleached yak fibres have poorer mechanical and morphology properties, and it is harder to felt for bleached fibres. For the spinning of 16.7 tex yarn, a complete condensing spinning system was adopted, and the performances of bleached fibre yarn decreased in the permissible range compared with the untreated fibre yarn.*

**Key words:** mordanting, black yakwool, whiteness, alkali solubility, fibre properties, yarn properties.

ble, and excellent moisture absorption, the price of yakwool is just a quarter of cashmere, and thus products made from yakwool are welcomed by the consumers. However, the limited colours of yak fibres hinder their application. Most yaks live in high and cold areas, and as the natural colour of the animal is closely related to the character of the environment in which it lives [3], most yak fibres are black or brown, with just a small amount of yakwool being white. Thus, based on the premise that the spinnability of yak fibres is not seriously damaged, bleaching pigmented yak fibre can broaden the range of its applications, and it can also improve its economical benefits.

At present, many metal ions have been applied to investigate the catalytic effect on hydrogen peroxide [4-8]. After comparison, Fe<sup>2+</sup> was found to be the most appropriate metal catalyst. Laxer and Whewell [8] concluded that the amount of Fe<sup>2+</sup> absorbed by coloured fibres was more than for white fibres. C Earland and A. S. Little [6] found that a complexation reaction happens between the metal ions and melanin during the pretreatment process, and Fe<sup>2+</sup> can be absorbed by melanin granules, which is preferential to keratin proteins. Thus hydrogen peroxide can be catalysed selectively by ferrous ions, thus minimising the damage to yak fibres.

During the pre-mordant process, redundant ferrous ions can be oxidised to ferric ions and deposited on fibres. After being oxidized, ferrous ions not only cause the

decomposition of hydrogen peroxide but also bring about a tiny amount of red in discolored yak fibres. In order to reduce the oxidation of ferrous ions, appropriate reducing agents should be added [8-10]. Khishigsuren A and co-workers [9] pointed out that during the mordant process, sodium bisulfate was the most appropriate auxiliary. By using sodium bisulfate, better whiteness and less fibre damage can be got. And the efficiency of fibre bleaching can be improved by using sodium bisulfate during the rinsing process.

In addition, the temperature and pH values of the mordanting bath have also been discussed [4, 11-14]. In order to minimise damage to fibres, M Giesen and K Ziegler [11] pointed out that pH values of 3.0-3.5 are appropriate. Trollip and co-workers [14] found that temperature between 60-70 °C may achieve the bleaching effect desired.

Motivated by all these research works above, optimisation mordant conditions were investigated in the paper, and black yak fibres were taken as samples. Yak fibres were bleached under different conditions. And by analysing the whiteness and alkali degradation of bleached fibres under different conditions, optimisation conditions were obtained. Finally the mechanical properties, surface morphologies and spinnabilities of untreated fibres and bleached fibres under the optimisation condition were compared and analysed, which may be useful for industrial production.

## Introduction

Yakwool is a kind of precious fibre used as an inner coat to keep the yak warm [1]. The properties of yakwool are comparable to cashmere, with the average fineness of yakwool being about 20 µm and the average length between 25-35 mm [2]. Not only do fine yak fibres have good elasticity, smooth han-

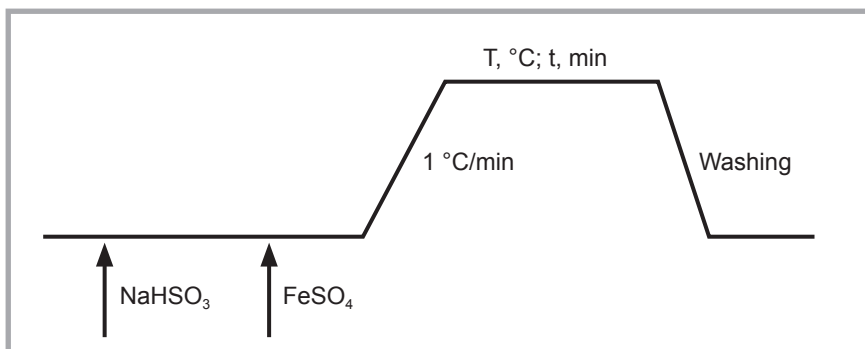


Figure 1. Mordanting process.

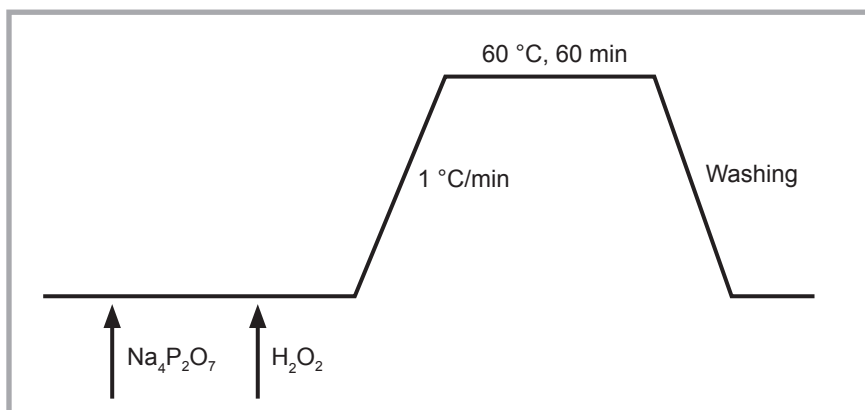


Figure 2. Oxidation bleaching process.

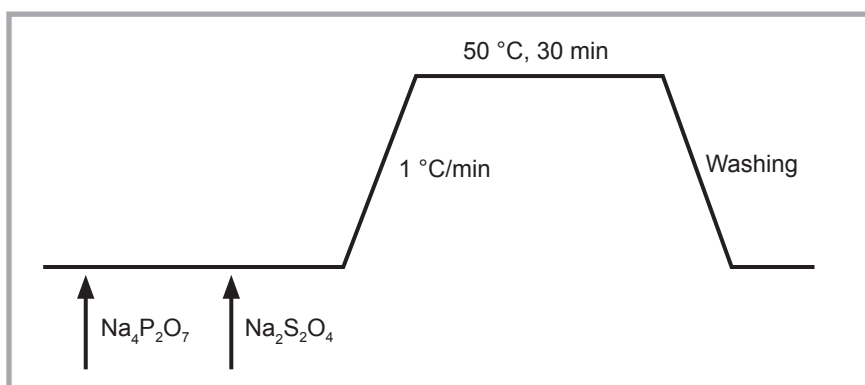


Figure 3. Reduction bleaching process.

## Experimental

### Material

Black yak fibres from Inner Mongolia were selected as raw material, provided by Zhongfuda Textile Co., Ltd. All chemicals were of analytical reagent grade. Ferrous sulfate, sodium bisulfate, sodium

pyrophosphate and sodium hydrosulfite were purchased from Sinopharm Group Chemical Reagent Co., Ltd. Hydrogen peroxide (30%) was from Wuxi Zhanwang Chemical Co., Ltd. SEIBana 4554V wool lubricating oil was from Henkel, and FX-AS20 Antistatic agent was from Beijing Fangxing Accessory Ingredient Co., Ltd.

Table 1. Orthogonal list of mordanting process.

Level	Fe <sup>2+</sup> , %	NaHSO <sub>3</sub> , (g·l <sup>-1</sup> )	T, °C	pH
1	8	1	40	2
2	12	2	60	3
3	16	3	80	4

## Bleaching technology

### Mordanting

The experiment was designed according to four factors and three levels of the orthogonal table. Based on the weight of water, different mordant baths containing ferrous ions and sodium bisulfate with various pH values and temperatures were set up, shown in Table 1. Untreated yak fibres were immersed in the baths for 30min, where the liquor/yakwool ratio was 50:1. Finally the mordanted yak fibres were taken out and rinsed, the reaction curve of which is shown in Figure 1.

### Oxidation bleaching

Oxidation bleaching was carried out in an alkaline condition with hydrogen peroxide (25 g/l) at 60 °C for 60 min, and sodium pyrophosphate (5 g/l) was added to the liquor as a stabiliser. The mordanted fibres were bleached for 60min and the liquor PH value adjusted to 8.5, with a liquor/yakwool ratio of 50:1; the reaction curve of which is shown in Figure 2.

### Reduction bleaching

Subsequent reduction bleaching was conducted with sodium hydrosulfite (6 g/l) and sodium pyrophosphate (4 g/l) at 50 °C for 30 min. The pH of the bath was 5.5, and the liquor/yakwool ratio 50:1. Finally the treated fibres were removed from the bleaching bath, rinsed with cold water and then dried, the reaction curve of which is shown in Figure 3.

## Testing methods

### Fibre performance

#### (1) Whiteness

The whiteness index  $W$  was calculated from parameters  $L$ ,  $a$  and  $b$ , as shown in Equation (1). Here parameter  $L$  (lightness) represents the lightness index, and  $a$  (redness) and  $b$  (yellowness) – chromaticity coefficients. Parameters  $L$ ,  $a$  and  $b$  were measured by means of a WSD-III-automatic whiteness meter under a D65 illuminant (at 10° standard observer). Every sample was tested 5 times, and then an average of 5 values was taken as the test value.

$$W = 100 - \sqrt{(100 - L)^2 + a^2 + b^2} \quad (1)$$

#### (2) Alkali solubility

The degree of damage to the fibre caused by bleaching was evaluated from the alkali solubility. Two grams of treated yak fibres were weighed and immersed in 100 mL of sodium hydroxide (0.1 mol/l) at (65±0.5) °C for 60 min. The remaining materials were filtered, rinsed with

sodium hydroxide (0.1 mol/l) twice, and then rinsed with distilled water six times. Finally the remaining substances were dried at (105±3) °C [15]. The alkali solubility was calculated by the following formula:

$$s = \frac{2 \times (1 - G) - W}{2 \times (1 - G)} \times 100\% \quad (2)$$

$$G = \frac{a - b}{a} \times 100\% \quad (3)$$

Here,  $S$  represents the alkali solubility,  $G$  the moisture ratio of untreated yak fibres,  $W$  the weight of the remaining substance,  $a$  the weight of untreated yak fibres, and  $b$  represents the weight of dried yak fibres.

### (3) Diameter and length

The fibre diameters were tested by means of an electronic microscopy fineness meter 30 times and the average value calculated.

The fibre lengths were measured using the hand drawing method. Firstly a bundle of fibres was taken, and combed gently with a steel comb, with coarser hairs being removed by repeated combing. When fibres were organised into a beam and one side of the velvet was neat, the principle length of the fibre could be tested. Putting the fibre bundles on a black velvet board, a ruler was used to measure the length of the fibre bundles. The result should be accurate to 0.5 mm.

### (4) Tensile property

Samples were measured with an XQ-2 tensile tester. As the average length of yak fibres is less than 35 mm, the clamp distance was set at 10 cm and the drawing speed at 10 mm/min.

### (5) Surface property

Vertical SEM images and frictional properties of untreated and bleached fibres were measured to evaluate the extent of damage to the fibre surface scales. SU1510 scanning electron microscopy was employed at a magnification of 2000 times.

The frictional property was measured with a Y151 friction coefficient gauge, with a weight of the tension clamp of 200 mg and speed of the rudder roll of 12 r/min. The frictional coefficient can be calculated as follows:

$$\delta = \frac{u_2 - u_1}{u_1 + u_2} \times 100\% \quad (4)$$

**Table 2.** Whiteness and alkali solubility analysis of mordanting orthogonal experiments. *Note:* \*  $K$  and  $M$  represent the average values of whiteness and alkali solubility.  $K_1$ ,  $K_2$  and  $K_3$ , represent the values of levels 1, 2 & 3 (Table 1), and  $M_1$ ,  $M_2$  &  $M_3$  respectively represent the values of level 1, 2 and 3, respectively (Table 1).  $R_1$  &  $R_2$  represent the ranges of whiteness and alkali solubility.

Treatment		Fe <sup>2+</sup> , %	NaHSO <sub>3</sub> , (g·L <sup>-1</sup> )	T, °C	pH	Whiteness, %	Alkali solubility, %
1		8	1	40	2	78.06	65.62
2		8	2	60	3	74.83	56.55
3		8	3	80	4	69.83	64.89
4		12	1	60	4	68.89	64.59
5		12	2	80	2	75.45	51.48
6		12	3	40	3	75.32	56.82
7		16	1	80	3	74.02	62.14
8		16	2	40	4	73.19	70.84
9		16	3	60	2	76.99	52.41
Whiteness	K <sub>1</sub>	74.24	73.66	75.52	76.83		
	K <sub>2</sub>	73.22	74.49	73.57	74.72		
	K <sub>3</sub>	74.73	74.05	73.10	70.64		
	R <sub>1</sub>	1.51	0.83	2.42	6.19		
Alkali solubility	M <sub>1</sub>	62.35	64.13	64.43	56.50		
	M <sub>2</sub>	62.30	59.62	57.85	58.50		
	M <sub>3</sub>	61.80	58.04	59.50	66.77		
	R <sub>2</sub>	0.55	6.09	6.58	10.27		

**Table 3.** Effect of bleaching on fibre mechanical properties.

	Diameter, µm	Length, mm	Strength, cN	Elongation, %	Tenacity, cN/dtex
Untreated	17.09	35.4	6.21	30.43	2.05
Bleached	16.03	33.7	5.36	34.66	2.01

Here,  $\delta$  represents the friction effect value, and the coefficient from the root to tip is defined as  $u_1$ , and that from the tip to root is defined as  $u_2$ .

### (6) Crimp property

The crimp is defined as the longitudinal irregular bending of fibres, the properties of which were measured by a YG362B crimp gauge.

### (7) Spinning performance

The tensile properties of yarns were measured with a yarn strength tester (YG068C), where the drawing speed was set at 500 mm/min, the drawing distance 500 cm and the pre-tension was 0.5 cN/tex. The hairiness was measured by a hairiness tester (USTER Zweigle HL400), and the speed was set at 400 m/min. Yarn evenness was measured by an USTER Tester 5 and twists of yarns were measured by a yarn twist counter (Y331A).

## ■ Results and discussion

### Optimal mordanting technology

Results of the whiteness and alkali solubility of yak fibres treated in nine dif-

ferent mordanting conditions are shown in **Table 2**.

From the results of **Table 2**, it can be seen the fibre whiteness values and fibre damage were mainly influenced by the pH values. As yak fibres are acid-resistant but alkaline non-resistant, and ferrous ions may be stable under strongly acidic conditions [15], the pH of the liquor should be maintained below 3. When the ferrous ion concentration, sodium bisulfate concentration or liquor temperature were definite, with increasing pH values, the whiteness and damage of the fibres were poorer. When the alkali solubility of the fibre was about 50%, the spinnabilities of yak fibres may not be influenced. Thus, based on the correlation between whiteness and alkali solubility in order to get the optimal bleaching conditions within the permissible limits of fibre damage, this clearly demonstrates that the alkali solubility of the fibre should be small enough – nearly 50%.

By comparing all the results in **Table 2** and considering the fibre damage, the optimal mordanting condition is as follows: per weight of water, the ferrous ion concentration is 16%, the sodium bisulfate

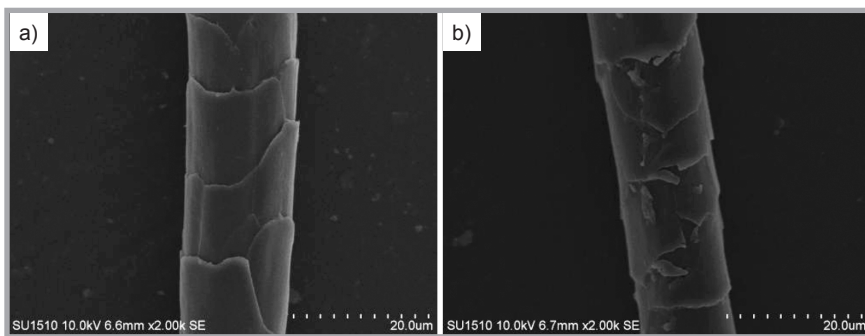


Figure 4. SEM images of untreated and bleached yak fibres: a) untreated, b) bleached.

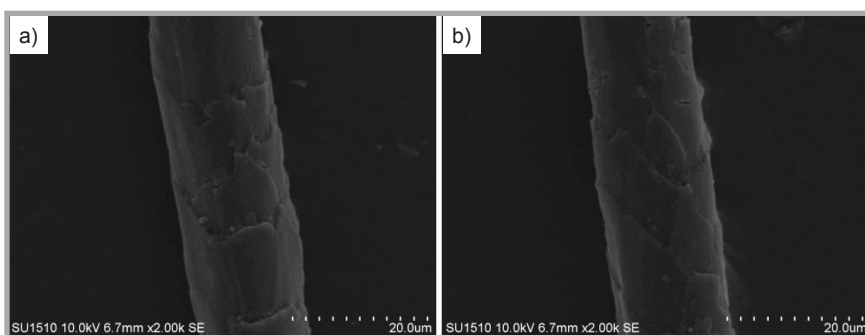


Figure 5. SEM images of bleached yak fibres: a) without wool lubricating oil, b) with wool lubricating oil.

concentration 3 g/l, the mordanting temperature 60 °C and the liquor pH value is set at 2.

### Fibre properties comparison

#### (1) Mechanical and crimp properties

Yak fibres were bleached according to the optimal mordanting condition selected above. Table 3 lists the fineness, tensile properties at break and crimp properties of untreated and bleached fibres.

From Table 3 above, it can be seen that bleached fibres are a little thinner than

untreated fibres. Due to the fibre damage caused by the bleaching process, fibre scales were destroyed or peeled off. And as the fibre got damaged during the bleaching process, bleached yak fibres are a little shorter than untreated ones.

When the tensile properties were compared, the loss of single fibre strength was 13.7%, with a loss of fibre strength of around 15% being acceptable. From Table 3, the elongation of the treated fibre is larger than the untreated one. After bleaching, the cleavage of the disulphide bond on the fibre surface can soften the fi-

Table 4. Effect of bleaching on fibre crimp properties.

	Crimp, 25 mm	Crimp ratio, %	Crimp recovery, %	Crimp elasticity rate, %
Untreated	8.5	15.02	12.97	87.51
Bleached	7.9	18.07	15.51	87.66

Table 5. Friction coefficient and frictional effect values of untreated and bleached yak fibres. Note:  $u_{1s}$  &  $u_{1k}$  represent the frictional effect values from the root to tip under static and kinetic conditions,  $u_{2s}$  &  $u_{2k}$  the frictional effect values from the tip to root under static and kinetic conditions, and  $\delta_s$ ,  $\delta_k$  represent the friction coefficients under static and kinetic conditions, respectively.

	Static			Kinetic		
	$u_{1s}$	$u_{2s}$	$\delta_s$ , %	$u_{1k}$	$u_{2k}$	$\delta_k$ , %
Untreated	0.1307	0.1948	19.69	0.2483	0.3000	9.43
Bleached	0.1533	0.2017	13.63	0.2763	0.3044	4.84

bre scales, making the fibres more elastic [16]. Thus fibres after bleaching cannot undergo brittle rupture during spinning.

We may also infer from Table 4 that for both untreated and bleached fibres, the crimp properties are close, demonstrating that bleaching had little influence on these properties.

#### (2) Surface morphology

SEM images and frictional properties of untreated and bleached fibres are presented in Figure 4 and Table 5.

The SEM images show the modification of the surface morphology of fibres before and after bleaching. During the bleaching process, the loss of protein/peptide material from the fibre can be indicated from SEM images [15]. The untreated yak fibre (Figure 4.a) had no surface damage and the scales were smooth and clear. After bleaching, it was evident that the corrosion and splitting of the fibre scales had occurred (Figure 4.b).

By testing the frictional properties, the surface properties can be reflected visually. Moreover the frictional properties can also influence the rate and degree of fibre felting [17-18]. Due to the directivity of the fibre scales, the static friction coefficients are smaller than the kinetic ones. In general, the scale coefficient of friction increases with the severity of treatment [19]. It can be seen from Table 5 that the scales of yak fibres were damaged and became rough, and that the friction coefficients of bleached yak fibres are more than those of untreated yak fibres. After bleaching, the friction effect values under static and kinetic conditions of fibres are all more than those of untreated fibres, which illustrates that it may be harder to felt bleached fibres than untreated fibres under the hot or wet conditions or under successive external mechanical forces.

### Comparison of spinning properties

Yak fibres are short, fluffy and have a low utilised percentage. In order to spin high-count yarns, the spinning procedure was as follows:

Oiling → Carding → Gilling (twice) → Combing → Gilling (twice) → Gilling (three times) → drawing (four times) → Roving → Spinning

As bleached yak fibres were damaged, and to guarantee smooth spinning and yarn quality, the amount of lubricating oil

should increase as appropriate. In general, wool lubricating oil can improve the toughness of yak fibre, decrease the friction among fibres and increase the moisture regain of yak fibres [20]. The surface morphologies with and without lubricating oil are shown in *Figure 5*.

Thus 0.15% more wool lubrication was added to bleached yak fibres than that of the untreated. The oiling process parameters are shown in *Table 6*.

From the carding process to the roving process, the same technologies were adopted for untreated and bleached yak fibres, where the roving quantum was 420 tex.

During the spinning process, a new kind of compact spinning system- a complete condensing spinning system was adopted, developed independently by Jiangnan University. In the spinning system, the front roller is replaced by a 50 cm hollow roller with a strip groove [21]. The spinning system and condensed area are shown in *Figure 6*. When the yakwool sliver reaches the front roller nip, with the help of airflow with negative pressure in the condensed area, fibres condense from the edge of the sliver to the center, which will help reduce the floating fibres and yarn hairiness further. The key advantage of the system is the cooperation between the hollow roller and suction components, the length of condensed area – 30 mm and uncontrolled area – 0 mm, thus yarns can be condensed over the whole condensed area [21]. Furthermore the hollow roller and width of the strip groove are the key technologies of the system. These may ensure stability and efficiency for high count yarn spinning.

The yarn manufacture parameters are presented in *Table 7*. In order to strengthen bleached yak fibre yarn, 2.59% more twists were added-helping to reduce short fibres from the surface of yarns. As bleached yak fibres are shorter and thinner than untreated ones (seen in *Table 3*), a decreased spinning speed may reduce the breakage of yarns and ensure the properties of bleached yarn. Yarn properties are presented in *Tables 8-10*.

According to the results shown in *Table 8*, with the same spinning technology, the falling rates of strength and tenacity are 8.23% and 8.35%. The reason for the lower tensile values of bleached yak fibre yarn is the decline in fibre strength, which

**Table 6.** Parameters of oiling process.

	Lubricating oil, %	Antistatic agent, %	Moisture regain, %	Reaction time, h
Untreated	0.40	0.2	20-21	12-24
Bleached	0.55	0.2	20-21	12-24

**Table 7.** Yak fibre yarn manufacture parameters.

	Untreated	Bleached
Model of spinning frame	DTM129	
Roller diameter, mm	27×27×50	
Roller distance, mm	29×11	
Ring model	PG1	
Ring diameter, mm	42	
Traveler type	U1UU <sub>DR</sub> 12/0	
Total draft multiple	25.19	
Back zone draft multiple	1.25	
Spindle speeds, (s.p.m)	8000	6000
Twist direction	Z	Z
Twists per 10 cm	90.76	93.11

**Table 8.** Tensile performances of yak fibre yarn.

	Strength, cN	Tenacity, cN/Tex	Elongation at break, %	Work of rupture, cNcm
Untreated	71.83	4.31	4.83	130.76
Bleached	65.92	3.95	4.40	106.79

**Table 9.** Evenness performances of yak fibre yarn.

	CVm, %	Thin, -50%	Thick, +50%	Neps, +200%
Untreated	19.79	400.0	440.0	146.7
Bleached	20.02	485.0	475.0	175.0

**Table 10.** Hairiness performances of yak fibre yarn.

	1 mm, (number · 100 m <sup>-1</sup> )	2 mm, (number · 100m <sup>-1</sup> )	≥3 mm, (number · 100m <sup>-1</sup> )
Untreated	13366	4345	1881
Bleached	12607	4587	1978

causes yarn rupture directly. Another possible explanation for the phenomenon is that damaged fibres can be ruptured into short fibres more easily, which may cause further yarn rupture. From the yarn's CVm values in *Table 9*, the deterioration of yarn evenness may also be caused by the increased ratio of short fibres. And the evenness of bleached yak yarn is still influenced by the shorter length and poor length uniformity of fibres.

In terms of fibre hairiness, in *Table 10* it is evident that bleached yak fibre yarn has more long hairiness (≥3 mm) than untreated yak yarn, and the numbers of short beneficial hairiness (1 mm and 2 mm) of untreated and bleached yarns were close. The long hairiness (≥3 mm) of bleached yak fibre yarn was 5.16% more than that

of untreated fibre yarns, which is because yarn hairiness usually increases with an increase in fibre diameter and decrease in fibre length [22]. But considering the fluffy and warm properties of yak fibre products, hairiness is not an important index for evaluation of yarn quality.

## Conclusions

In this paper, the optimal mordanting condition was analysed in detail. According to the analysis of the bleaching effect on bleached fibres, it can be concluded that fibre damage is mainly influenced by the pH values of the liquor. And then, with comprehensive consideration of fibre whiteness and alkali solubility, the appropriate mordanting condition was obtained.

Based on the optimal mordanting condition, the falling rate of fibre strength is relatively big, but fibre fineness and fibre crimp properties were apparently not damaged after bleaching. Meanwhile, due to the damage effect on the fibre internal structure, frictional properties of the fibres were also influenced.

During the spinning process, based on the damage degree of bleached yak fibres, the levels of wool lubricating oil, yarn twist, spinning gauges, and machine speeds were adjusted. The results show that the differences between these two yarns' evenness and hairiness are small. Furthermore the falling rate of strength, elongation and tenacity are 8.23%, 8.90% and 8.35%, which can all be acceptable for industrial production.



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## Institute of Textile Engineering and Polymer Materials



The Institute of Textile Engineering and Polymer Materials is part of the Faculty of Materials and Environmental Sciences at the University of Bielsko-Biala. The major task of the institute is to conduct research and development in the field of fibers, textiles and polymer composites with regard to manufacturing, modification, characterisation and processing.

The Institute of Textile Engineering and Polymer Materials has a variety of instrumentation necessary for research, development and testing in the textile and fibre field, with the expertise in the following scientific methods:

- FTIR (including mapping),
- Wide Angle X-Ray Scattering,
- Small Angle X-Ray Scattering,
- SEM (Scanning Electron Microscopy),
- Thermal Analysis (DSC, TGA)

Strong impact on research and development on geotextiles and geosynthetics make the Institute of Textile Engineering and Polymer Materials unique among the other textile institutions in Poland.

### Contact:

Institute of Textile Engineering and Polymer Materials  
University of Bielsko-Biala  
Willowa 2, 43-309 Bielsko-Biala,  
POLAND  
+48 33 8279114,  
e-mail: itimp@ath.bielsko.pl  
www.itimp.ath.bielsko.pl