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Preparation of viscose/wool powder blended fibre and optimization of its acid dyeing

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The viscose/wool powder fibres blended with different superfine wool powder content have been produced using wet spinning method. The viscose/wool powder blended fibres have been dyed with acid red dye and the effects of dyeing conditions such as dyeing temperature, dye bath *p*H, salt concentration, dyeing time and wool powder content are studied. The higher dye exhaustion is obtained at 90°C, with 5% sodium sulfate and 4 *p*H. The per cent of exhaustion increases with wool powder content. All the dyed fibres show good colour fastness to washing and rubbing. The blended fibres show characteristics of both cellulosic fibre and wool fibre.

Keywords: Acid dyeing, Blended fibre, Superfine wool powder, Viscose

1 Introduction

Wool fibre is a highly valued natural protein fibre, and it can be biodegradable and renewable, which shows green trend emerging in textiles. Every year, a large amount of wool fibres is abandoned as waste for different reasons. This is a vast waste for natural protein and perhaps results in environment pollution. Therefore how to reuse the abandoned wool fibre has received more and more attentions[1-3]. Among these studies, most wool protein was regenerated by chemical method, and the compound protein material was prepared through the blending of regenerated protein with other polymers[4-7]. However, chemical process is relatively complicated and costly. More economical and practical ways are needed in application of reused protein. One possible way is to prepare fine protein powder mechanically and use the powder directly[8,9].

One earliest developed chemical fibre (viscose) is used extensively for both apparel and non-apparel applications. Viscose is regenerated cellulose and has many good performances such as high moisture regain and silk-like handle. To make full use of main attributes of viscose fibres, it is common in blend spinning yarn to use viscose fibres[10,11]. Blending wool protein in viscose dope is a new way to produce protein-based cellulose fibre with improving wool-like characters.

In previous studies[12-14], different proteins were used, produced in powder form and then characterized. In this study, blended fibres with superfine wool powder and viscose fibres were produced. To investigate the feasibility and practicality of the compound fibre, the dyeing properties of the fibres with acid dye were studied. The dyeing conditions such as temperature, dye bath *p*H, salt concentration, and dyeing time were investigated.

2 Materials and Methods

2.1 Materials

Wool powder was prepared using self-made mechanical pulverizer. The wool powder particle size is shown in Fig.1. Particle size has a narrow

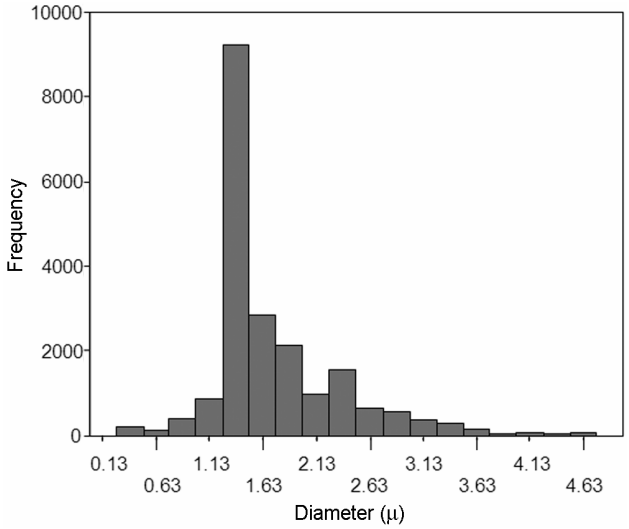

Fig. 1–Diameter distribution of superfine wool powder

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distribution. Average particle diameter was 1.7μm and size of 95% of particles was kept lower than 3.0μm.

The acid red dye(GR) was commercially obtained. Sodium sulfate, sodium carbonate, sodium chloride and all other reagents were of general purpose grade and supplied by China Reagent Company.

2.2 Fibre Preparation

The fibres were produced by wet spinning in Shandong Hailong Incorporated Company with common spinning machine of small size (200kg/day productivity, made in Japan).

Before adding into spinning dope, the wool powder was stirred mechanically in mixed solution of 1% twelve benzene sulfonate, surfactant, protective agent, and alcohol for 30min.

Pulp was impregnated and after aging, yellowing, and dissolution, wool powder dispersion solution was added into the pulp and mixed evenly for 10min. After mixing, filtration, deaeration(for 4h), the viscose pulp blended with wool powder was extruded through spinneret (holes number: 15000) and submerged in a coagulating bath containing sulphuric acid (H_2SO_4), sodium sulphate (Na_2SO_4) and zinc sulphate ($ZnSO_4$). Chemical composition in coagulating bath neutralized alkaline content in the viscose and made cellulose regenerated. The temperature of coagulating bath was kept at 48°C and spinning was done at an ambient temperature. The total draft was 103.87% and the linear density of the obtained fibres was 1.5D.

2.3 SEM Study

Fibre longitudinal and cross-sectional morphology were observed with a scanning electron microscope (SEM)[JSM-6510LV(JEOL) at 20 kV with gold powder coating on the samples].

2.4 Physical Properties Evaluation

The physical properties of viscose fibre and the blended fibres with different wool powder content (5% and 15% respectively) were evaluated according to viscose staple fibre standard GB/T 14463-2008 (similar to BISFA-2004).

2.5 Dyeing

Dyeing was performed under appropriate condition on a laboratory dyeing machine. The dyebath contained 2%(o.w.f) dye and some other auxiliaries. The fibres samples(1g) were dyed in a dye bath having material-to-liquor ratio 1:50 and the *p*H value of dyeing bath was adjusted using phosphate buffer. The samples were immersed into the dyeing solution at 40°C, heated up to 90°C at a rate of 2°C/min, and then maintained at this temperature for 60 min. After dyeing, the fibres were rinsed in water and dried in air. The fibres were dyed at various dyebath conditions (temperature, auxiliary content and *p*H value) in order to investigate their effects on the acid dyeing properties of the viscose/wool powder fibres.

2.6 Dyeing Rate

The exhaustion (E) was determined using the following equation:

$$E\% = \frac{A_0 - A_1}{A_0} \times 100$$

where A_0 and A_1 are the absorbance of the dyebath before and after dyeing.

Dyeing property of the fibres was also studied according to their dye uptake rate curves.

2.7 Colour Fastness Testing

The colour fastness to washing and rubbing was determined according to GB/T3921-2008 (ISO 105-C10:2006) and GB/T 3920-2008 (ISO 105-X12:2001).

3 Results and Discussion

3.1 Fibre Morphology Observation

The viscose/wool powder fibres were spun successfully without clogging up the spinneret holes. The SEM images (Fig.2) shows the morphology of different blended fibres. As shown in SEM images, the blended fibre is similar to the common viscose fibre. During viscose process, regeneration and coagulation start at the surface of the fibre and thus form a skin-core structure. As the core shrinks on regeneration of cellulose, the skin also contracts and becomes wrinkled, resulting in serrated cross-sectional shape and striations along fibre length.

With the addition of the wool powder, some minor cracks are observed in the fibre section, striations along fibre length become uneven and some fine powder appears in blended fibre surface. With wool powder content increase, spinning process and thus fibre structure are affected.

3.2 Physical Properties of the Blended Fibre

The physical properties of the typical blended fibres were tested according to production standard and the results are shown in Table 1. The tested properties include mechanical properties (dry and wet break strength, coefficient of variation of dry break

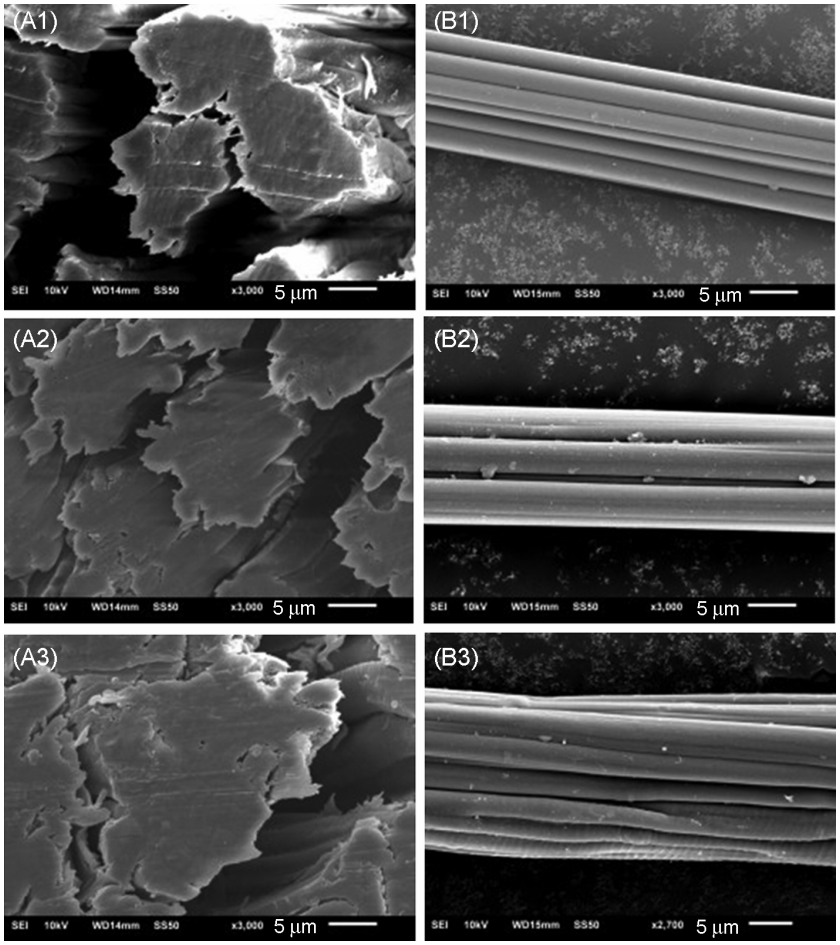

Fig.2–Cross-sectional and longitudinal morphology of viscose fibre with different wool powder contents (A1-A3 cross-section of fibre with 0, 5%, 15% wool powder contents respectively, and B1-B3 corresponding longitudinal morphology

Table 1–Physical properties of typical blended fibres

Property	Wool powder addition content, %		
	0	5	15
Dry break strength (≥), cN/dtex	2.38	2.11	2.02
Wet break strength (≥), cN/dtex	1.30	1.27	1.11
Elongation rate (≥), %	18.5	17.6	20.8
Linear density deviation (±), %	2.99	2.86	3.21
Length deviation (±), %	2.11	1.97	2.71
Over-length fibre rate (≤), %	0.6	0.8	0.5
Double-length fibre (≤), mg/100g	14.2	10.7	12.2
Residual sulfur content (≤), mg/100g	8.9	9.2	9.6
Defect (≤), mg/100g	0	0	0
Oil contamination fibre (≤), mg/100g	0	0	0
CV% (≤)of breaking strength	15.4	13.2	15.7
Whiteness (≥), %	76.3	74.2	71.9

strength, and elongation at dry break), fibre shape parameter(fibre linear density and its deviation, fibre length and its deviation and over-length, double-length rate), fibre appearance quality(whiteness, defect and oil contamination fibre) and residual sulfur content. The data show that all items have minor difference. For example, though fibre breaking strength declines slightly, the strength(2.02cN/dtex) of fibre with 15% wool powder content is still above the standard value. Above all, after wool powder addition the physical properties of the fibres keep stable and can meet the requirements of the downstream textile application.

3.3 Acid Dyeing

3.3.1 Effects of Dyeing Temperature on Acid Dye Uptake

Figure 3 shows the exhaustion behavior of acid dyes on pure viscose fibres and viscose/wool powder blended fibres during dyeing at different temperatures. Dye exhaustion on all fibres increases as the temperature rises from 60°C up to 90°C. Indeed, heating increases fibre swelling, and reduces dye molecule aggregates in the solution, thus the diffusion of the dye molecules to the fibre is easier and dye uptake values increase. It is obvious that the dye uptake of the blended fibres is much higher than that of pure viscose fibres, which means that the

substantivity of acid dye to viscose fibre is improved by the addition of wool powder. In addition, with the increase of wool powder content, the per cent of exhaustion increases obviously.

3.3.2 Sodium Sulfate Addition Effects on Dyeing

Figure 4 shows the effects of sodium sulfate on the exhaustion of acid dye uptake on fibres. For both pure viscose fibre and the blended fibre, the exhaustion values decrease with the increase of sodium sulfate concentration until a relatively stable absorption occurs. It means that sodium sulfate requirements are lower for acid dyes on viscose/wool powder blended fibres. Too much salt may affect the solubility of dyes and results in dye aggregation, thus reducing dye uptake rate. It is also evident from Fig. 4 that the exhaustion of the dyeing on viscose/wool powder blended fibre is higher than that on viscose rayon and the dye uptake increases with wool powder content up to 15%. The substantivity of acid dye for viscose fibre is enhanced by protein powder addition, and this result is also found consistent with the previous work[13].

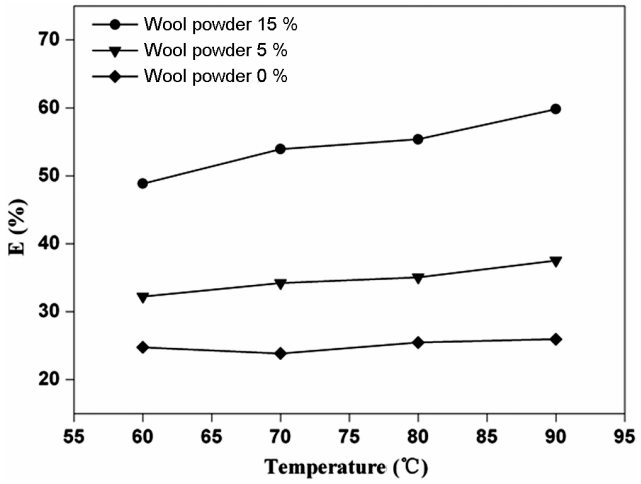

Fig.3–Effects of dyeing temperature on acid dye uptake of viscose/wool powder fibre

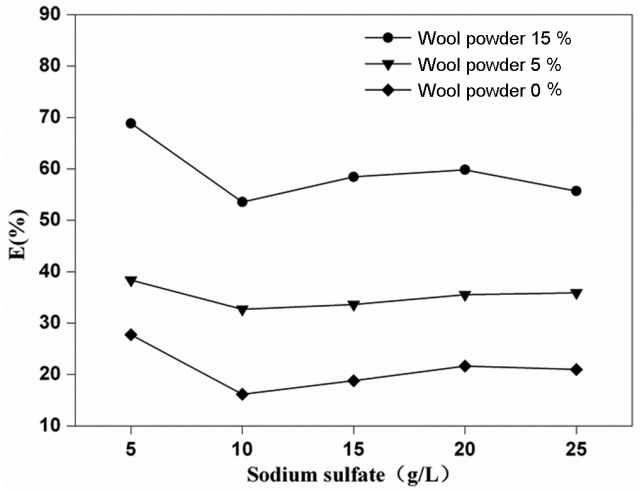

Fig.4–Effect of sodium sulfate addition on exhaustion of acid dye

3.3.3 pH Values

Figure 5 shows that during acid dyeing, the ***p***H values of the dye bath play an important role on the adsorption process. The effects of ***p***H values are attributed to the correlation between dye structure, fibres used and dye stability. For pure viscose fibres, when dyed with acid dyes, the whole dye exhaustion is lower than that of the viscose fibre blended with wool powder and during dying at ***p***H 4~6, the highest dye exhaustion is obtained at ***p***H 4. Dye uptake increases as the ***p***H value increases up to 4, and then it goes down. It is attributed to the dyeing environment requirement for weak acid dye. For the blended fibre, because of the existence of wool powder, when dyed around isoelectric points, the number of the protonated terminal amino groups of wool powder is increased and then the fibre is positively charged. Therefore, the blended fibre has stronger attraction to the sulfonic acid groups in acid red dye. This is due to the fact that the blended fibre has higher dye exhaustion than pure viscose fibre.

3.3.4 Effects of Dyeing Time

Figure 6 shows that the dye uptake of pure viscose fibre and the blended fibre obtained depend on the dyeing time. It is observed that dye uptake of all fibres reaches equilibrium in a short time. Dye adsorption in pure viscose fibre has the same trend as

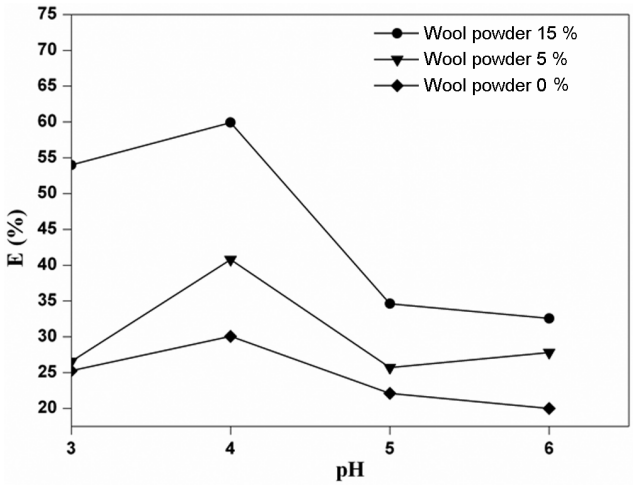

Fig. 5–Effect of *p*H value on exhaustion of acid dyeing

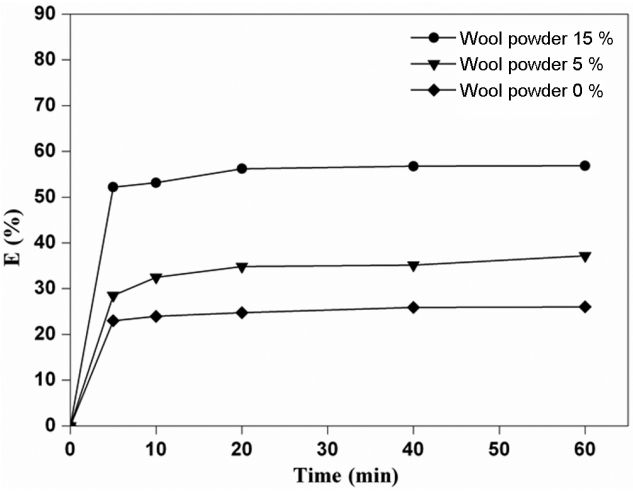

Fig.6–Acid dye uptake of viscose/wool powder blended fibre

in the blended fibre. However, it is obvious that dye uptake increases with the increase in wool powder content. The dye uptake of the fibre with 15% wool powder reaches 55% at the end of the dyeing process, which is almost 1.5 times to that of the blended fibre with 5% wool powder. Since wool proteins are made out of different combinations of the twenty essential amino acids, many different dyes, especially acid dye can be used to dye wool fibre. Now the results show that the viscose/wool powder fibre can also be dyed with acid dyes.

3.4 Colour Fastness

All the fibres were dyed under the same conditions as dyeing rate measurement for 60min in order to test the colour fastness. Colour fastness test results (Table 2) show that acid dyes on the viscose/wool powder fibre present good washing fastness: the change to colour is above 4, staining adjacent value is below 1-2, and all fastness to rubbing is above 4, which indicates that the blended fibres are suitable for commercial application.

4 Conclusion

Wool powder of 1.7μm average diameter has been prepared and blended with viscose dope to spin blended fibre. The viscose/wool powder blends are produced successfully and wool powder addition had little effects on fibre shape. Fibre strength decreases slightly with the increase of wool content. The viscose/wool powder fibre can be dyed with acid dye. When the blended fibre is dyed at 90°C with 5% sodium sulfate and *p*H value of 4, higher dye exhaustion can be obtained. Colour fastness to washing and rubbing are good enough for commercial use. The results obtained suggest that the blended fibres are novel and have both characters of cellulosic fibre and wool fibre.

Table 2–Colour fastness of acid dyes on blended fibres

Wool powder content, %	Wash fastness		Rub fastness	
	Colour change	Staining to cotton	Dry	Wet
0	3-4	1-2	4-5	4
5	4	1	4-5	4
15	4	1	4-5	4

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