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Study on the Structure and Properties of Viscose/Wool Powder Blended Fibre

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Abstract

Viscose/wool powder blended fibres with 0, 5, 10, 15 and 20% wool powder were produced with the method of wet spinning. SEM, FT-IR spectroscopy, WAXD and TG analysis were used to study the chemical composition and structure of viscose/wool powder blended fibre. The experimental results show that the macromolecule composition, aggregation structure, morphological structure and thermal properties of these viscose/wool powder blended fibres were similar, which mainly show the characteristics of viscose fibre. Absorption studies were carried out to investigate the protein characteristics of the blended fibre. The results showed that the affinity of the blended fibre to acid dye increased with the wool powder content.

Key words: wool powder; viscose fibre; blended fibre; dyeing.

tein was inevitably destroyed. Therefore the protein compound fibre might lose the original properties of the protein. On the other hand, since protein powder could keep the original properties of materials without destroying the microstructure, it has been widely applied in modern industries and hi-tech related fields with some special properties [7 - 9].

Viscose fibre is one of the widely used chemical fibres. It is based on the most naturally occurring organic polymer: cellulose. Viscose fibres are hydrophilic, absorbent, and skin-friendly. They are widely used in blended spinning with synthetic and wool fibre to exert their good performance. Blending proteins is a technological approach to generate protein-based cellulose fibre with a more complete set of specific properties, such as improving the wool-like effect and dyeing properties with acid dyes. In the previous study, we developed and characterised different protein powder [3, 10, 11], while in this study, to produce polymer/protein blend fibre without losing its original properties, superfine wool powder was produced by a special mechanical pulveriser and powder was added to viscose dope to spin viscose/wool-powder blend fibre. In this paper we only give the fibre structure and dyeing properties of the fibres; the mechanical properties of the fibres will be discussed in detail in another paper.

Experimental

Materials

Australian wool fibres were cut into short pieces on a rotary blade and then the pieces were ground into superfine wool powder using a self-made mechanical pulveriser. **Figure 1** shows the particle

size distribution of the wool powder. 95% of the powder is less than 3.0 mm in diameter, and the average particle size is 1.7 mm.

Spinning

Spinning was carried out at the Shandong Hailong Incorporated Company using a traditional wet spinning procedure with a common spinning machine of small size (200 kg/day productivity, made in Japan). Firstly the wool powder was added to a mixture solution of 1% twelve benzene sulfonate, surfactant, protective agent and alcohol, and stirred mechanically for 30 min.

The pulp was impregnated and after aging, yellowing and dissolution, wool powder (different contents: 0, 5, 10, 15, 20 %wt) dispersion solution was added into the pulp and mixed evenly for 10 min. After mixing, filtration and deaeration (for 4 h), the viscose pulp blended with wool powder was extruded through a spinneret (hole number: 15000) into a spinning bath. The bath was made up of sulphuric acid (H_2SO_4), salts (sodium sulphate (Na_2SO_4) and zinc sulphate

Introduction

Recently there has been great interest in studying compound protein fibres to develop new textile materials and to recycle redundant protein materials [1 - 3]. In most of these studies, the protein was regenerated by chemical dissolving, and the compound protein fibre is prepared through the grafting of the regenerated protein on some fibre, or the regenerated protein and other polymers were blended to produce blend fibres [4 - 6]. However, the protein was prone to decomposing in the process of dissolving and spinning, and the inherent microstructure of pro-

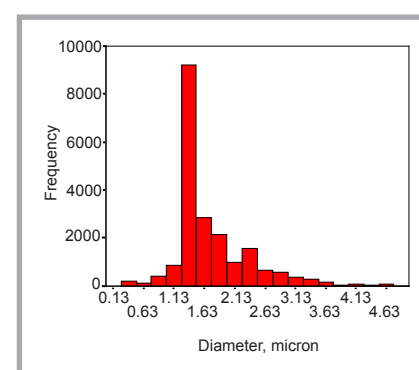


Figure 1. Diameter distribution of superfine wool powder.

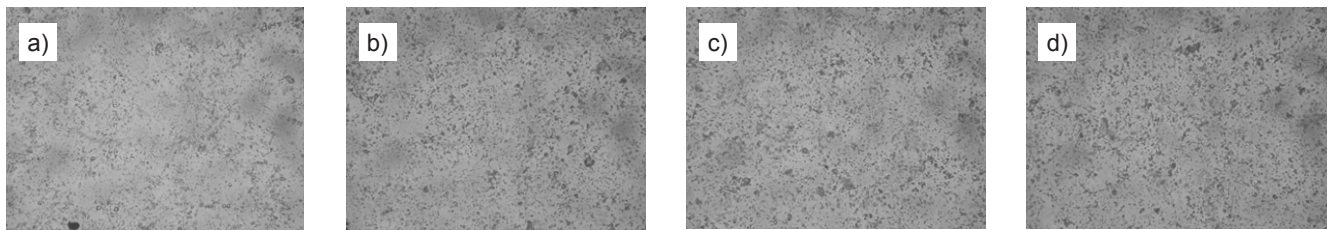


Figure 2. Stability of wool powder in alkaline solution during different time intervals: a) 0 h, b) 6 h, c) 12 h, d) 24 h.

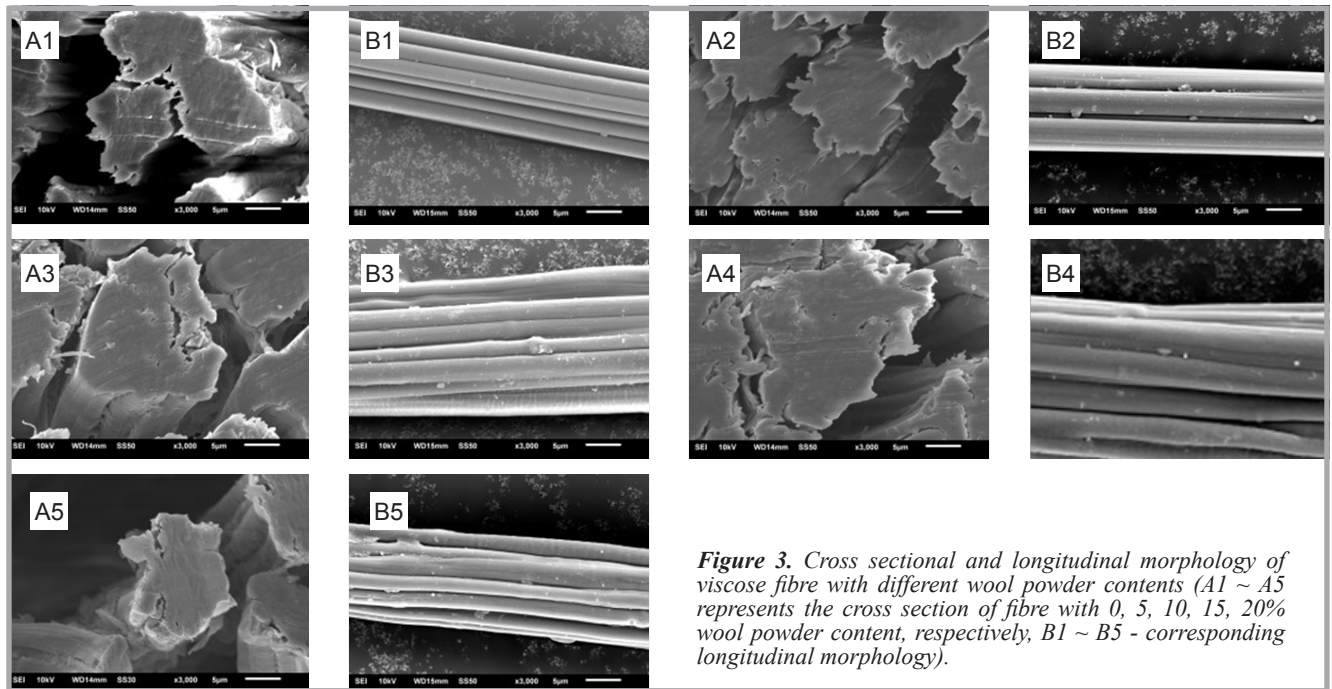


Figure 3. Cross sectional and longitudinal morphology of viscose fibre with different wool powder contents (A1 ~ A5 represents the cross section of fibre with 0, 5, 10, 15, 20% wool powder content, respectively, B1 ~ B5 - corresponding longitudinal morphology).

(ZnSO₄), which neutralised the alkaline content of the viscose and regenerated the cellulose. The bath temperature was 48 °C. Viscose dope was kept at 20 °C and the spinning temperature was that of the room. The total draft was 103.87% and the linear density of the fibres obtained was 1.67 dtex.

Characterisation

To observe wool powder stability in the alkaline solution, after the treated wool powder was added to the pulp and mixed evenly, the blended solution (NaOH content 5.8%) was observed under an optical microscope during different time intervals.

The longitudinal and cross sectional morphology of the fibre was investigated using a scanning electron microscope (SEM), JSM-6510LV (JEOL, Japan) at 20 kV with gold powder coating on the samples.

Physical properties of the blended fibres (wool powder addition content: 0, 5, 15%) were evaluated according to vis-

cose staple fibre standard GB/T 14463-2008.

Infrared (IR) spectra of 500 cm⁻¹ and 4000 cm⁻¹ were obtained on a VERTEX70 (German Bruker) with a potassium bromide presser.

X-ray diffraction (XRD) was employed in studying the crystallinity by using a X'Pert PRO X-diffractor (PANalytical B.V., the Netherlands) at 40 kV and 30 mA. XRD studies were carried out at Bragg's angle for 2 h from 10° to 45° at a rate of 8 deg/min.

Thermal properties of the samples were measured on a Pyris 1 TGA analyser (PerkinElmer, USA) and testing was carried out from 20 °C to 600 °C with inflowing nitrogen atmosphere (50 ml/min) at a heating rate of 10 °C/min.

The dyeing property of the fibres was studied according to their dyeing rate curves. The fibre samples were dyed with Great Red (GR) acid dye at a bath ratio of 1:50 at 90 °C. The initial dye concentration was 2% (o.w.f). At some time in-

tervals, the solution was withdrawn and the absorbency measured by a UV-visible spectrophotometer 2550 (Shimadzu, Japan). The exhaustion rate (E in %) was estimated 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 initial bath and the bath at intervals, respectively.

Results and discussion

Wool powder stability evaluation

Figure 2 shows the wool powder situation in the blended solution. It was observed that after dispersion protection treatment, the wool powder was stable in the 5.8% alkaline solution after 24 h, which meant the wool powder is stable in viscose dope, and thus spinning can go ahead. Wool fibres can be dissolved in boiling 30% NaOH solution, whereas they just swell in 5% NaOH solution at 20 °C. Although the viscose solution contains 5% NaOH, compared with pure 5% NaOH aqueous solution, damage to

Table 1. Physical properties of typical blended fibres.

Item	Unit	Wool powder addition content		
		0	5%	15%
Dry tenacity \geq	cN/dtex	2.38	2.11	2.02
Wet tenacity \geq	cN/dtex	1.30	1.27	1.11
Elongation rate \geq	%	18.5	17.6	20.8
Linear density deviation \pm	%	2.99	2.86	3.21
Length deviation \pm	%	2.11	1.97	2.71
Over-length fibre rate \leq	%	0.6	0.8	0.5
Double-length fibre \leq	mg/100 g	14.2	10.7	12.2
Residual sulphur content \leq	mg/100 g	8.9	9.2	9.6
Defects \leq	mg/100 g	0	0	0
Oil contamination of fibre \leq	mg/100 g	0	0	0
Coefficient of variation of breaking strength (CV) \leq	%	15.4	13.2	15.7
Whiteness \geq	%	76.3	74.2	71.9

the wool powder is much less, which is due to the relatively high viscosity of the viscose solution, thus preventing alkali penetration into the fibre.

Fibre morphology investigations

Blended viscose fibres were spun successfully without a filter and spinneret clogging. SEM photos of the cross sectional and longitudinal morphology of different blended fibres are presented in **Figure 3** (see page 27), with a magnification of 3000 \times . As shown in the figure, the cross sections of all the fibres are serrated and almost solid. With an increase in the wool powder content, cracks appear in the sections. Observed from the longitudinal direction, viscose fibre has a smooth surface and straight and evenly distributed grooves. With the addition of wool powder, grooves along the longitudinal direction become uneven and some wool powder appears on the surface, which means that too much wool powder

addition would affect the spinning process and thus the fibre structure.

Physical properties of the blended fibre

Physical properties of typical blended fibres were tested according to the production standard and the results are shown in **Table 1**. The data show that all items have a minor difference, which means that after wool powder addition physical properties of the fibres remain stable and can meet the requirements of downstream textile application.

IR spectrum analysis

Figure 4 shows IR curves of pure viscose fibre and blended fibres. All curves exhibit similar absorption peaks: stretching vibration of the -OH group in viscose appears at 3400 cm^{-1} , stretching vibration of the CH_2 group at about 2920 cm^{-1} , bending vibration of the - CH_3 group at 1367 cm^{-1} , bending vibration of -OH and stretching vibration of -C-O-C- at

1019 cm^{-1} . On the whole, the spectrum of the ordinary viscose fibre and blended fibre are quite similar, which reflects the infrared absorption peak of pure viscose fibre and indicates that the addition of wool powder has little effect on the infrared spectra of viscose fibre. The wool powder content also shows little effect. In the blended fibre, the viscose and wool powder are just a physical mixture, thus there are no new absorption peaks in their IR spectrum.

X-ray diffraction analysis

XRD patterns of the viscose and blended fibre with various contents of wool powder are shown in **Figure 5**. For pure viscose fibre, there is one main diffraction peak at about 20.42 $^\circ$, which belongs to 101 crystalline peaks of celluloses II. The weaker diffraction peak at about 11.19 $^\circ$ belongs to the 10I crystalline peak of celluloses II. All the viscose/wool powder fibres show a similar diffraction pattern. With an increase in the wool powder content, a 101 peak almost disappears, and a new diffraction peak appears at about 25.7 $^\circ$, which is attributed to the wool keratin crystalline peak. A decrease in the diffraction peak intensities indicates the effect of wool powder addition on the aggregation structure of viscose fibre.

Thermal analysis

The thermal degradation patterns of viscose fibre and blended fibre with various contents of wool powder are illustrated in **Figure 6**. All samples have similar thermal behaviour and demonstrate endothermic reactions. It is obvious that

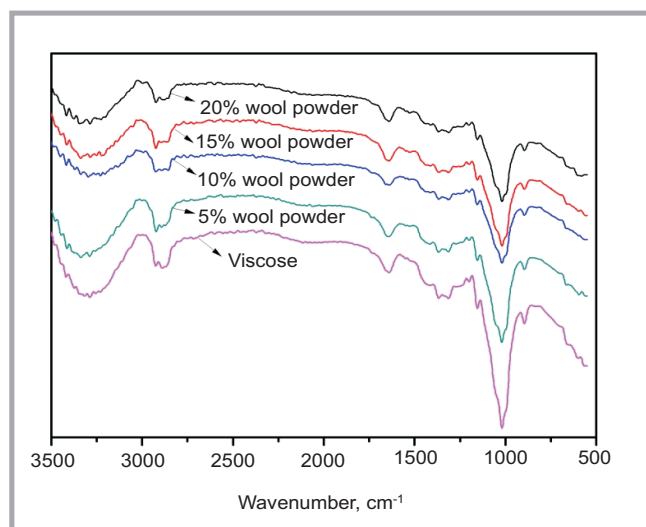


Figure 4. IR spectrum of different fibres.

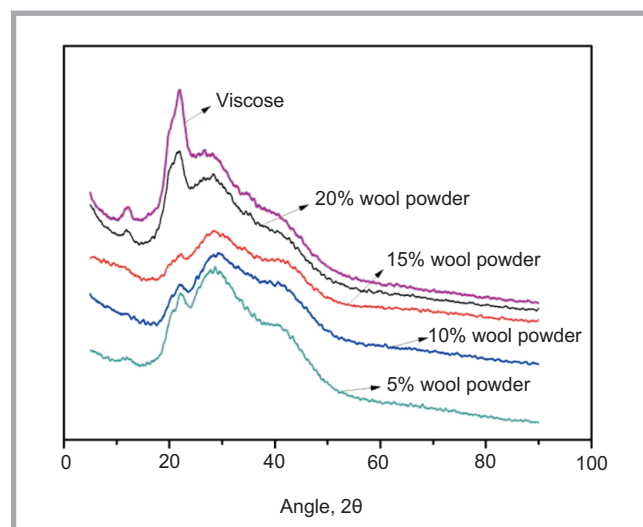


Figure 5. XRD profiles of viscose fibre and viscose/wool powder fibre.

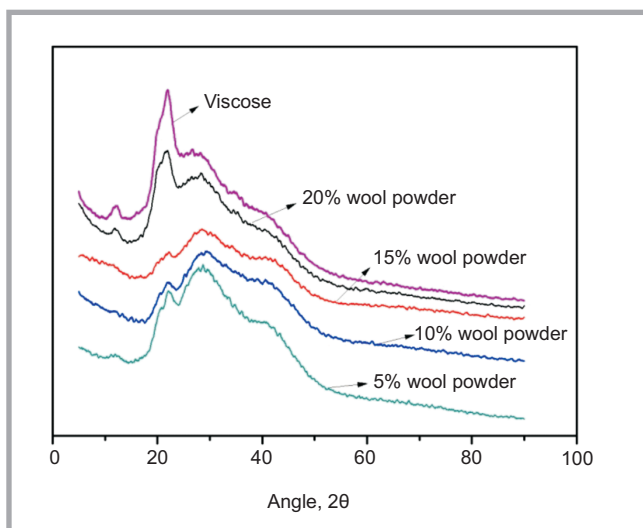


Figure 6. TG results of viscose fibre and viscose/wool powder fibre.

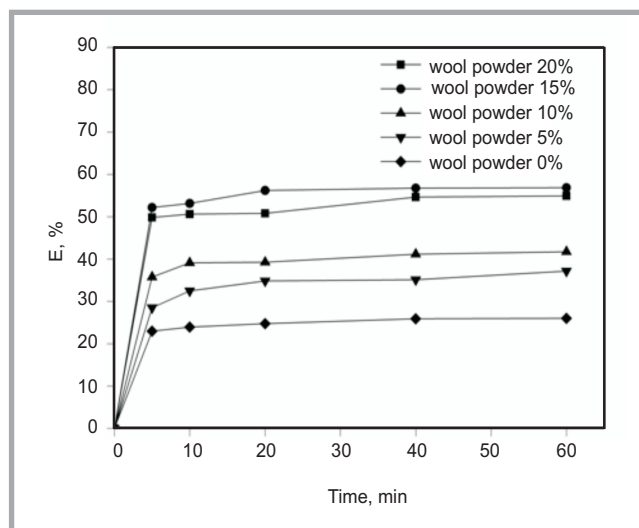


Figure 7. Dye uptake of viscose /wool powder blended fibre with different powder contents.

all the samples have only one big weight loss during thermal degradation, which happens mainly between 270 °C and 380 °C. It starts to lose weight at room temperature and the weight loss resulting from water elimination is about 3%. Then there is a big drop in the TG curves, which means that the thermal degradation of the viscose fibre and blended fibre are completed in one step. After 380 °C, the weight loss rate becomes very slow, and the residual quantity is nearly invariable at 400 °C; namely the decomposition is complete. With an increase in wool powder content, the main degradation rate and peak temperature of the weight loss are nearly invariable. Due to the existence of wool powder, the decomposition residue is slightly different. The entire thermal degradation behaviour indicates that the addition of wool powder has little effect on the thermal stability of viscose fibre.

Dyeing rate of various fibres

As natural protein fibre, wool fibre can be dyed by acid dyes easily, hence it is expected that viscose/wool powder blended fibre could be dyed with acid dyes. Pure viscose fibre and blended fibre were dyed by acid dyes under the same experimental condition, and the exhaustion curves of all the samples are shown in Figure 7. It can be seen that all curves show the same trend, namely the slope of each curve is steep for the first few minutes and slows down after that, quickly reaching equilibrium. It is evident that the affinity of the blended fibre is better than pure viscose fibre, and the dye uptake is obviously enhanced as the wool powder content increases. The dye uptake of the fibre

with 20% wool powder reaches 55% at the end of the dyeing process, which is 2.2 times that of pure viscose fibre.

Conclusion

Superfine wool powder was added to viscose dope and blended viscose fibres of different wool powder content were successfully produced. With an increase in wool powder, the surface of the fibre became uneven and a crack in the fibre appeared. A characteristic peak of cellulose was observed in the blended fibres because wool powder was a minor component there. With an increase of wool powder content, the peak intensity, especially 101 crystalline peaks of celluloses II, became weaker. TG results showed that the thermal stability of the viscose fibre and wool powder blended fibres were identical. The viscose/wool powder fibre showed a similar dyeing property to that of wool fibre and can be dyed with acid dye.

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References

- Poole AJ, Church JS, Huson, MG. Environmentally sustainable fibres from regenerated protein. *Biomacromolecules* 2009; 10:1–8.
- Kon'kov A, Pustovalova O, Agapov I. Biocompatible materials from regenerated silk for tissue engineering and medicinal therapy. *Applied Biochemistry & Microbiology* 2010; 46:739–744.

- Xu WL, Cui WG, Li WB, Guo WQ. Development and characterizations of super-fine wool powder. *Powder Technology* 2004; 140: 136–140.
- Zhu Z, Ohgo K, Reiko W, Toshiaki T, Asakura T. Preparation and characterization of regenerated Bombyx mori silk fibroin fibre containing recombinant cell-adhesive proteins; Nonwoven fibre and monofilament. *Journal of Applied Polymer Science* 2008; 109, 5: 2956 – 2963.
- Yang YQ, Reddy N. Properties and potential medical applications of regenerated casein fibres crosslinked with citric acid. *International journal of biological macromolecules* 2012; 51, 1-2: 37 – 44.
- Aluigi A, Vineis C, Varesano A, Mazzuchetti G, Ferrero F, Tonin C. Structure and properties of keratin/PEO blend nanofibres. *European Polymer Journal* 2008; 44, 8: 2465 – 2475.
- Novak S, Kobe S, McGuinness P. The effect of chemically bonded organic surface layers on the behaviour of fine powders. *Powder Technology* 2004; 139: 140–147.
- Miyajima T, Yamamoto K-I, Sugimoto M. The effect of particle orientation and/or position on two-dimensional shape measurements. *Advanced Powder Technology* 2001; 12: 413–423.
- Zhang Y, Liao S, Fan Y, Xu J, Wang F. Chemical reactivities of magnesium nanopowders. *Journal of Nanoparticle Research* 2001; 3: 23–26.
- Wang X, Xu WL, Ke GZ. Preparation and dyeing of superfine down-powder/viscose blend film. *Fibres & Polymers* 2006; 7: 250–254.
- Xu WL, Ke GZ, Peng XQ. Studies on the Effects of the Enzymatic Treatment on Silk Fine Powder. *Journal of Applied Polymer Science* 2006; 101: 2967–2971.

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