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Preparation and characterization of persimmon leaves/cellulose blend fiber and comparison with cellulose fiber

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Abstract

With respect to the fact that persimmon leaves having better effects on medical area applications, a persimmon leaves/cellulose blend fiber was prepared by wet spinning from a DMSO/polyoxymethylene solvent under different spinning conditions, e.g. the temperature of coagulation bath, amount of persimmon leaves added and concentration of spinning solution. Based on mechanical measurement, X-ray diffraction and DSC characterization, it was found that this blend fiber had similar mechanical and thermal properties as referenced cellulose fiber obtained using the same method.

Keywords: Persimmon leaves; Cellulose; Blend fiber; Wet spinning; Mechanical properties

1. Introduction

Since persimmon leaves have been found to have beneficial effects on haemostasis, constipation and hypertension, they have been broadly applied in food and medicine area, especially recently (Funayama & Hikino, 1979; Kameda, Takaku, & Okuda 1987; Kotani, Fujita, & Tanaka 1999; Kotani et al., 2000; Matsumoto et al., 2002; Tanaka et al., 2003). Therefore, it is our consideration to convert this material to a fiber to extend its application area.

The aims of this paper are to: (1) prepare a persimmon leaves/cellulose blend fiber, and (2) primarily study the influences from spinning on physical properties of this blend fiber and compare to reference cellulose fiber.

Recently, studies of persimmon leaves have been carried out in our laboratory, and their contents and several properties, e.g. surface and dynamic dissolution in two solvents, e.g. DMSO and DMAc, have been characterized (Hu, Zuo, Zhang, & Shen, 2002; Shen, Wang, & Gu, 2004; Shen, Wang, & Liu 2003). These studies provided results that allowed us to prepare a persimmon leaves/cellulose blend fiber by means of wet spinning as aimed. A reference cellulose fiber was also prepared using the same method and condition as that of blend fiber.

2. Experimental

2.1. Materials

As previously used and described, persimmon leaves picked from Shanghai and prepared as powder with a size of about 40 mesh (Shen et al., 2004, 2003). The cellulose used was a commercial product provided by Shanghai Chemical Fiber Co. and known to have degree of polymerization (DP) of about 500, and α-cellulose content about 90%.

Polyoxymethylene (PF) and DMSO were analytical grade solvents purchased from a local chemical company (Shanghai) were used as received without further purification.

2.2. Dissolution and spinning of persimmon leaves/cellulose blend fiber

Weighed amounts of persimmon leaves, PL (2–10% wt), cellulose (10% wt) and DMSO/PF10% were used to prepare

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spinning solutions. Solutions were initially stirred and heated to 60 °C held for 1 h, then heated to 110 °C and held until that temperature transparent. The residual formaldehyde within solution was removed using a vacuum pump at 80 °C for several hours. Before spinning, this solution was stirred again and filtered to remove insoluble solid materials.

Laboratory scale wet spinning was employed. The process was similarly as that of Focher et al. (1994). Under a pressure of about 30 kPa and controlled by a metering pump, e.g. 0.2 g/min, spinning solution was allowed to flow into a 100 μm spinnerette die, L/D ratio of 1.0, to form a persimmon leaves/cellulose blend filament. Then, this filament was passing and solidified by through two water baths. The first was a coagulation bath with a temperature of about 40–60 °C; take up rate, V_1, was about 1.2 m/min. The second was a boiling water bath; take up rate, V_2, about 2.4 m/min. Finally, the filament was collected on a winder at a rate of about 10 m/min. After that, the filament was washed with distilled water, then dried in a vacuum oven at 50 °C for 24 h. Before analysis and characterization, the filament was held at 20 °C and 65% relative humidity.

A cellulose filament was also prepared using the same conditions as a reference.

2.3. Fiber analysis and characterization

The mechanical properties for obtained filament were measured using several Chinese made fiber analysis instruments, e.g. XQ-1, YG086 and YG001A made by Donghua University, Shanghai. During measurement, the gauge length and crosshead speed were chosen at 500 mm and 7.5 mm/min, respectively, in accordance with ASTM D3822-90. Reported values are averages of 10–30 independent runs.

The X-ray diffractograms of fibers were recorded by means of a Rigaku III Dmax 2500 type X-ray diffractometer; Nickel-filtered ($\lambda = 0.1542$ nm) Cu Kα radiation was used for all measurements. The Sherrer equation (Alexander, 1969) was employed to calculate crystal grain size corresponding to $2\theta = 14°$.

For DSC analysis, a Mettler-Toledo 822e Differential Scanning Calorimeter was employed. The temperature was raised from 20 to 300 °C at a rate of 10 °C/min.

To analyze the orientation of fiber, a sonic velocity measurement was carried out using a self-made tester and the method of Moseley (1960).

3. Results and discussion

3.1. Influence of coagulation bath temperature on preparation and mechanical properties of cellulose referenced fiber

To apply wet spinning to prepare a filament, the influence from the temperature of coagulation bath was initially investigated. As Table 1 reports, a referenced cellulose filament was prepared. In Table 1, we find that both the lowest and highest temperatures are unacceptable for preparation of fiber. The data in Table 1 also suggest that the best temperature for coagulation bath seems to be in the range of 40–50 °C.

Table 1
Influence of the temperature of coagulation bath on preparation of referenced cellulose fiber

Temperature of coagulation bath (°C)	Fiber drawing behavior	Fiber breaking intensity (cN/dtex)
20	Unable to draw	–
30	Breaks easily	1.6
40	Good drawing	2.1
50	Good drawing	2.2
60	Breaks easily	1.9
65	Unable to draw	–

3.2. Influence of amount of persimmon leaves added on preparation and mechanical properties of blend fiber

To produce a persimmon leaves/cellulose blend fiber, understanding this material's compositions is generally required because they are usually in variety probably to influence blend fiber. Previously, we have investigated and found that persimmon leaves have cellulose 68.28%, hemicellulose 7.54% and lignin 11.70%, respectively (Hu et al., 2002). Since the percent of persimmon leaves added to the blend fiber was expected high for bioactivity, the bio-fiber was prepared with three different percents of PL.

Table 2 indicates that the persimmon leaves affect the orientation and mechanical strength. This is reasonable because a rigid polymer cellulose is mixed with a random coil, low molecular weight matrix. According to Flory (Billmeyer, 2000), poor compatibility occurs upon blend polymers beyond a critical concentration in the absence of strong intermolecular interactions based on the interference of the random coil polymer with a mutual orientation of the molecules of the rod-like polymer molecules. In fact, in this case the low-molecular-weight polymer of persimmon leaves seemed to have acted as a plasticizer facilitating rotating of polymer chains. The same phenomenon was also

Table 2
Influence of the amount of persimmon leaves, PL, added on the mechanical properties of PL/cellulose blend fibers

Fibers	Added PL amount (wt%)	Fiber orientation (%)	Fiber breaking intensity (cN/dtex)
Cellulose	0	70.0	2.20
PL/cellulose blend	2	69.0	2.10
PL/cellulose blend	5	67.5	2.00
PL/cellulose blend	10	64.5	1.95

Table 3
Influence of the spinning solution concentration on preparation and mechanical properties of persimmon leaves/cellulose blend fiber

Concentration of spinning solution (wt%)	Added PL amount (wt%)	Fiber drawing behavior	Fibers breaking intensity (cN/dtex)
6	5	Difficult drawing	–
8	5	Breaks easily	1.8
10	5	Good drawing	2.0
12	5	Good drawing	2.2

PL, persimmon leaves.

observed in a case of wood fiber preparation by Focher et al. (1994). Based on Table 2, the decrease in mechanical properties seems to be less than expected from the amount of PL added.

3.3. Influence of concentration of spinning solution on mechanical properties of blend fiber

By fixing the percent of persimmon leaves added, e.g. 5 wt%, and varying the concentration of spinning solution from 6 to 12 wt%, its influence on fiber preparation and mechanical properties of the blend fiber was also investigated. As summarized in Table 3, a lower concentration, e.g. 6%, seems to be inappropriate for preparation of fiber. Moreover, the mechanical properties of the blend fiber increased with an increase of the concentration of spinning solution, in good agreement with the above-mentioned Flory theory.

3.4. A comparison of persimmon leaves/cellulose blend fiber with referenced cellulose fiber

Table 4 compares X-ray data of the reference and blend fibers resulted from Fig. 1. From Fig. 1 and Table 4, it was observed that the blend fiber crystalline was less difference, e.g. mainly displayed at $10\bar{1}$ and 102 peaks comparing to reference cellulose fiber.

3.5. A comparison of the thermal behaviors for persimmon leaves/cellulose blend fiber and referenced cellulose fiber

Fig. 2 showed DSC curves for these two fibers. A comparison of these curves found that the glass transition

Table 4
A comparison of the crystallinity and crystal grain size for persimmon leaves/cellulose blend fiber and referenced cellulose fiber

Fibers	Added PL amount (%)	Crystallinity (%)	Crystal grain size (nm)		
			101	$10\bar{1}$	102
Cellulose	0	69.6	1.17	1.66	1.19
PL/cellulose blend	10	66.5	1.17	1.61	1.23

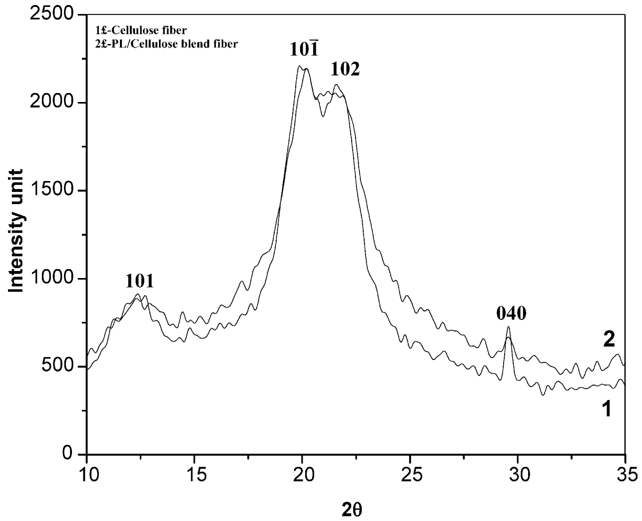

Fig. 1. A comparison of X-ray diffractograms for referenced cellulose fiber (1) and persimmon leaves/cellulose blend fiber (2).

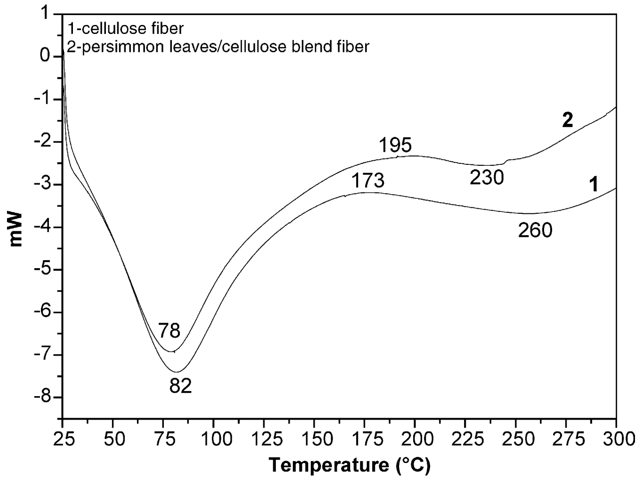

Fig. 2. A comparison of DSC curves for referenced cellulose fiber (1) and persimmon leaves/cellulose blend fibers (2).

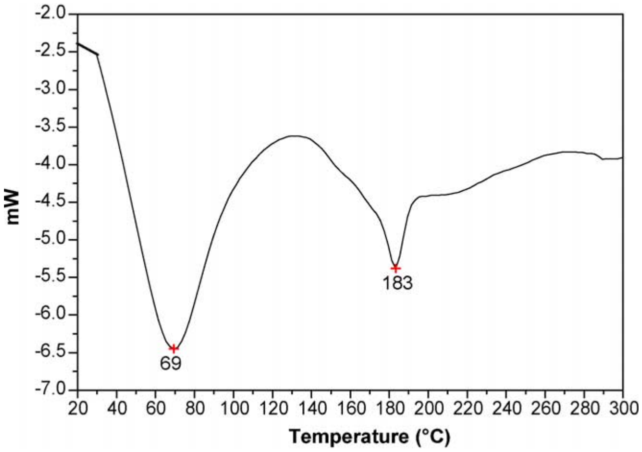

Fig. 3. A DSC curve of persimmon leaves.

temperature, T_g, of the blend fiber is about 4 °C less than that of reference cellulose fiber. For comparison, Fig. 3 is the curve of persimmon leaves alone.

4. Conclusion

Using DMSO/PF solvent and wet spinning method, and after investigating several influences on reference cellulose fiber, persimmon leaves/cellulose blend fiber was prepared. By physical measurements, it was found that persimmon leaves/cellulose blend fiber can be prepared and its properties are generally compared to those of a reference cellulose fiber.

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