

Rheological Properties of Cellulose/Chitin Xanthate Blend Solutions and Properties of the Prepared Fibers

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ABSTRACT: The focus of this article is the rheological properties of cellulose xanthate, chitin xanthate, and their blend solutions with cellulose/chitin blend weight ratios of 9.5 : 0.5, 9 : 1, 8 : 2, and 5 : 5 (mostly 9 : 1 blend solutions). The preparation and properties of fibers from 9 : 1 blend solutions and cellulose xanthate solutions are also discussed. The non-Newtonian index of the investigated solutions was found to vary in the following order: chitin < cellulose < 9.5 : 0.5 blend < 9 : 1 blend < 8 : 2 blend < 5 : 5 blend. Showing a tendency contrary to that of the non-Newtonian index, the structure viscosity index varies in the following order: chitin > cellulose > 9.5 : 0.5 blend > 9 : 1 blend > 8 : 2 blend > 5 : 5 blend. For 5–9 wt % 9 : 1 blend solutions, increasing the solution temperature aids the improvement of the fluidity of 9 : 1 blend solutions in the temperature range of 10–40°C. The zero shear viscosity decreases in an index manner with the solution temperature

increasing. The 7–8 wt % 9 : 1 blend solutions have good filtering and rheological properties and are ideal for spinning fibers. The mechanical properties of blend fibers spun from 7% 9 : 1 blend solutions are lower than those of pure cellulose and are much higher than those of Crabyon fiber, and they still reach the national criteria and fit the need for further processing. This proves that the viscose method which we have developed here is an efficient way of preparing cellulose/chitin blend fibers with satisfactory mechanical properties and processing properties. Scanning electron microscopy photographs show that the surface of 9 : 1 blend fibers is coarser than that of pure cellulose fibers. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 1208–1215, 2008

Key words: blends; fibers; polysaccharides; rheology

INTRODUCTION

As the two most abundant sources of polysaccharides found in nature, cellulose and chitin are biologically degradable resources that have received more and more interest since the 1980s. One major direction is in fiber preparation because the handle of both prepared fabrics is excellent. New spinning methods such as electrospinning have been used to prepare fibers, and Schiffman and Schauer¹ used one-step electrospinning to obtain crosslinked chitosan fibers with improved production and a decreased average fiber diameter of 128 ± 40 nm.¹ Cellulose fabric is moisture-absorbent, comfortable, and cheap, and chitin and chitosan fibers are biostatic, inflammation-diminishing, odor-resistant, odor-preventing, and itch-resistant. Therefore, blends of cellulose and chitin seem very attractive to us. However, chitin and cellulose cannot dissolve in ordinary solvents,² and this limits their widespread use.

Bochek et al.³ discussed macromolecular interactions in chitin–cellulose blend solutions in which

dimethylacetamide/lithium chloride was used as a solvent and the macromolecules showed partial compatibility. Phonwong et al.⁴ prepared chitin/cellulose blend films in which dimethylacetamide/lithium chloride was used as a solvent. Zhang et al.⁵ prepared cellulose/chitin blend membranes from sodium hydroxide/thiourea aqueous solutions, and the morphology and structure of the blend membranes were investigated; 5 wt % ammonium sulfate was used as a coagulation bath.

Fuji Spinning Co. (Osaka, Japan) has prepared fibers with the commercial name Chitopoly by wet spinning from cellulose viscose containing a fine chitosan powder. Chitopoly fibers have antimicrobial activity and deodorant properties.⁶ Hirano and coworkers^{7,8} prepared fibers from *N*-acetylchitosan xanthate, its derivatives, and their blends with cellulose xanthate. However, they mainly used *N*-acetylchitosan (a regenerated chitin) prepared by the chemical *N*-acetylation of chitosan as a raw material, rather than chitin, because the intramolecular and intermolecular hydrogen bonds of *N*-acetylchitosan are weaker than those of natural chitin, and this results in good solubility in a 14% aqueous sodium hydroxide solution.

Noguchi et al.⁹ prepared chitin–cellulose blend fibers by spinning an aqueous blend solution of

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sodium cellulose xanthate with sodium chitin xanthate in a coagulation bath. Omikenshi Co., Ltd. (Osaka, Japan) has prepared cellulose–chitin blend fibers named Crabyon in a similar ordinary viscose way with antimicrobial activity, deodorant properties, high water regain, and high adsorption ability for metal ions. However, the mechanical properties of Crabyon are still low.^{10,11}

Because the mechanical properties of Crabyon are low¹¹ and the viscose method is still most widely used for industrial production, we want to develop a distinct viscose method for preparing fibers with improved mechanical properties. As we know, the rheological properties of the spinning solution are very important, in that they determine the spinning conditions. Therefore, the rheological properties of blend solutions of cellulose xanthate with chitin xanthate provide instructional information for further fiber formation, and this will provide us with the preferred solution composition, temperature, concentration, and so on. Here for the first time, the rheological properties of cellulose/chitin xanthate blend solutions have been investigated to provide data for further fiber spinning. The mechanical properties of the prepared fibers are reported.

EXPERIMENTAL

Materials and reagents

The cellulose samples were cotton linters with a degree of polymerization of 1200; they were obtained with the cupriethylene diamine method and had an α -cellulose content of 90.8%. The chitin samples were made from shrimp with a degree of polymerization of 1000; this was obtained with the equation $[\eta] = 2.1 \times 10^{-4} \times M^{0.88}$ (where $[\eta]$ is the intrinsic viscosity), which was provided by Chen et al.¹² Dimethylacetamide containing 5% lithium chloride (w/w) was chosen as the solvent, ground into a powder that could pass through #60 mesh, and kept over fresh phosphorous pentoxide in a desiccator before use. Reagent-grade carbon disulfur and sodium hydroxide were supplied by Shanghai Feida Chemical Co. (Shanghai, China).

Preparation of the blend solutions

The cellulose viscose was prepared as follows: Cellulose was dipped into a 20 wt % sodium hydroxide solution for an hour at 20°C and then crushed until its weight was 3 times that of the original cellulose; it was then crumbled and aged for 8 h at 40°C. Then, a certain amount of carbon disulfide [CS₂; 37 wt % of the original weight of α -cellulose] was put into the mixture and stirred continually until a transparent solution was obtained.

The chitin viscose was obtained according to Thor's¹³ method: a certain amount of chitin powder was mixed with a 43 wt % sodium hydroxide solution in a flask at room temperature for 300 min, and the ratio of the mass of the chitin to the volume of the sodium hydroxide solution was 1 : 20. Then, some residual sodium hydroxide solution was removed through a vacuum supply, and the weight ratio of the obtained product to the original chitin powder was 3/6. Then, the product was mixed with chopped ice under violent stirring at 0°C until the whole system became transparent. A certain amount of CS₂ was added under stirring until a stable, orangelike solution of chitin xanthate was obtained. During the reaction process, different factors, including the alkali concentration, alkalization time and temperature, amount of CS₂, and solution concentration, have an important influence on the quality of chitin xanthate, which influences the solubility of chitin xanthate in dilute alkali solutions and finally influences the prepared fibers.

After both solutions were filtered, the cellulose xanthate solution was blended with the chitin xanthate solution according to different mass ratios (9 : 1, 9.5 : 0.5, 8 : 2, and 5 : 5 weight ratios of cellulose to chitin). Homogeneous, orangelike solutions were obtained and kept at 0°C until they were used.

Rheological measurements

The rheological properties of the solutions were measured on an SR5 stress-controlled rheometer (provided by Rheology Science Co., New Jersey, USA) at 10–40°C. The realized region of the shear rate ($\dot{\gamma}$) was in the range of 0.5–200 s⁻¹.

The slope of the flow curve in the form of $\lg \delta_t = f(\lg \dot{\gamma})$ obtained at infinitely small $\dot{\gamma}$ is the non-Newtonian index (n): δ_t is the shear stress, and $\dot{\gamma}$ is the shear velocity. n indicates the extent of fluid deviation from a Newtonian fluid.

The apparent viscosity (η_a) was obtained as follows:

$$\eta_a = \delta_t / \dot{\gamma}$$

The structure viscosity index ($\Delta\eta$), which was obtained at infinitesimal $\dot{\gamma}$, was obtained as follows:

$$\Delta\eta = (-d \lg \eta_a / d \dot{\gamma}^{1/2}) \times 10^2$$

Preparation of the fibers from the blend spinning solutions

Spinning conditions

The spinning routine is described in Figure 1. The spinning equipment was ordinary viscose spinning

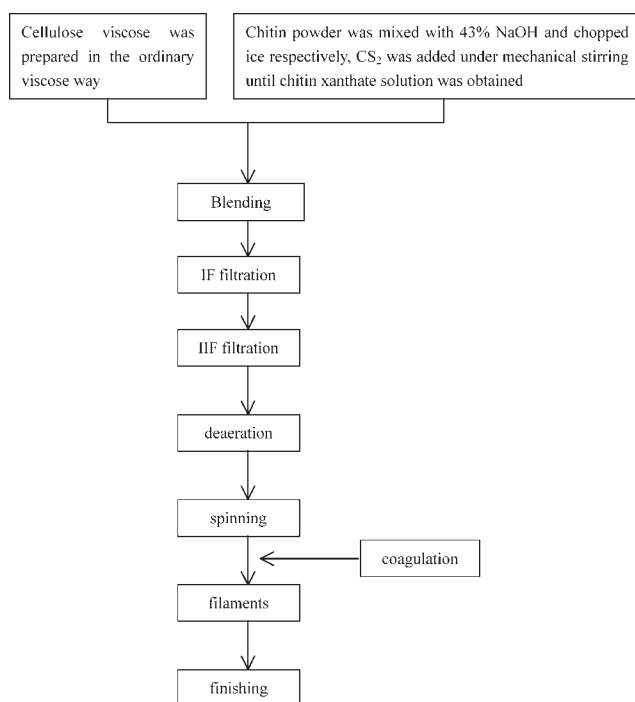


Figure 1 Production process of cellulose/chitin filaments from cellulose/chitin xanthate blend solutions.

equipment manufactured by Shanghai Chengdong Limited Co. (Shanghai, China). The spinneret was provided by Changzhou Spinneret Co. (Changzhou, China).

Cellulose viscose and chitin viscose were prepared, and they were blended under strong mechanical stirring in a postdissolving machine in different mass ratios until homogeneous solutions were obtained. The dissolving temperature was 14–25°C, and the alkali content was controlled to around 4–12 wt %. Then, the blend solution was filtered for the first time which was defined as (IF), deaerated, and aged until a certain extent of aging was reached. The spinning solution was deaerated under a 0.08-MPa vacuum for 15 h to remove the dissolving air in the spinning solution. The aging period lasted for 6–12 h at 15–26°C. The spinning solution was filtered again which was called IIF filtration and spun through a spinneret into a coagulation bath. After solidification and partial regeneration, the original fiber was stretched, plasticized, cut, washed, desulfurized, bleached, washed with acid, oiled, and dried; cellulose/chitin fibers were obtained.

The pore diameter of the filter cloth for IF filtration was 30–25 µm; the pore diameter of the filter cloth for IIF filtration was about 20–25 µm. The diameter of the adopted spinneret was 0.08 mm. The spinning velocity was 70 m/min.

The jet stretch ratio (the ratio of the take-up speed to the spinning speed) was 3%, the stressed draw ratio was 2.69%, and the draw ratio between the drawing gadget and coagulation roller was 20%, respectively.

The coagulation bath had the following composition: 100–135 g/L sulfuric acid, 6–25 g/L zinc sulfate, and 200–330 g/L sodium sulfate. The temperature of the coagulation bath was 40–55°C.

Finishing

The finishing procedure included washing with water at 35–55°C, desulfurization, bleaching, washing with acid, and oiling. The concentration of the aqueous sodium hydroxide solution for desulfurization was 3–9 g/L, and the temperature was 50–90°C. The concentration of hydrogen peroxide for bleaching was 1–5 g/L, the temperature was 20–40°C, and the pH value was controlled at 7–10. The acid washing temperature was 25–45°C. The concentration of the oil bath was 1–2 g/L, and the temperature was 25–45°C.

Equipment and testing conditions

Scanning electron microscopy (SEM) photographs

A JEOL JSM-5000LV scanning electron microscope (Tokyo, Japan) was used to study the surface morphology of the fibers. The fibers were extracted with ether at 35°C for 4 h, and then the fiber surface was coated with gold and used for observation.

Mechanical properties

The measurements of the mechanical properties were carried out as follows. The tensile strength was measured on a tensile testing machine provided by Changzhou Textile Machine Co. (Changzhou, China). The breaking intensity (δ_b) was calculated as follows:

$$\delta_b = F/A$$

where F is the breaking force of the machine and A is the cross-sectional area of the fiber. Ratio of dry stretching, ε_b , was calculated as follows:

$$\varepsilon_b = (L - L_0)/L_0 \times 100\%$$

where L_0 is the original length of the fiber and L is the length of the fiber at breakage.

About 20 fiber samples were tested for both the pure cellulose and 9 : 1 blend fiber, and the averages and standard errors are reported.

RESULTS AND DISCUSSION

Dependence of the rheological properties on the composition

Dependence of n on the composition

For the investigated solutions, n increases with increasing chitin content and is maximum when the chitin content is 50% (here 50% means that the chitin

mass percentage in the mass sum of cellulose and chitin is 50%), whereas n of a chitin xanthate solution is much lower than that of other solutions (Fig. 2). During the aging process, cellulose xanthate and chitin xanthate decompose consistently, and this leads to an increasing amount of free cellulose and chitin macromolecules in the solution. Because there exist hydrogen bonds among acetamino groups and hydroxyl groups in chitin macromolecules and there exist hydrogen bonds among hydroxyl groups in cellulose macromolecules, the interaction in or among chitin macromolecules is stronger than that of cellulose, so n of a chitin xanthate solution is lower than that of a cellulose xanthate solution. The addition of chitin macromolecules destroys the crosslinks among cellulose macromolecules, and the resistance among flow fields decreases; this leads to an increase in n , and this tendency increases with increasing chitin content in the blend solution. It is favorable for the spinning process. When n increases, the elastic deformation of the fluid in the spinneret duct decreases, the stretching property of the spinning fluid is improved, and the spinning velocity is increased.

Dependence of $\Delta\eta$ on the composition

All solutions appear as shear-thinning fluids. $\Delta\eta$ indicates the extent of solution structuralization. The higher $\Delta\eta$ is, the worse the fluid spinnability is, the more difficult fiber formation is, and the lower the fiber mechanical properties are. The spinnability of a spinning fluid is determined with the values of n and $\Delta\eta$. Figure 3 shows a tendency contrary to that of n : $\Delta\eta$ of a chitin xanthate solution is apparently higher than that of other solutions, and $\Delta\eta$ of the blend solutions decreases with increasing chitin content. This means that the spinnability of the investigated solutions varies in the following order: chitin < cellulose < 9.5 : 0.5 blend < 9 : 1 blend < 8 : 2 blend < 5 : 5 blend (here the ratios indicate the mass ratios of cellulose to chitin in the blend solu-

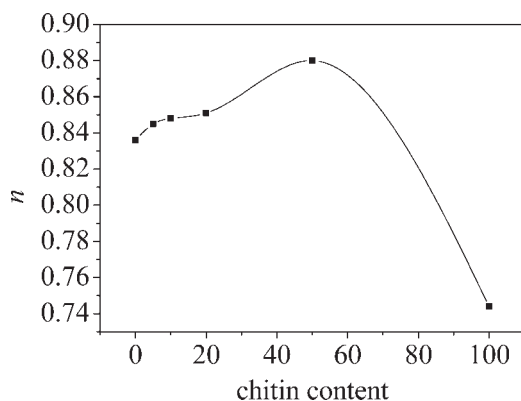


Figure 2 Dependence of n on the chitin content of blend solutions at 25°C.

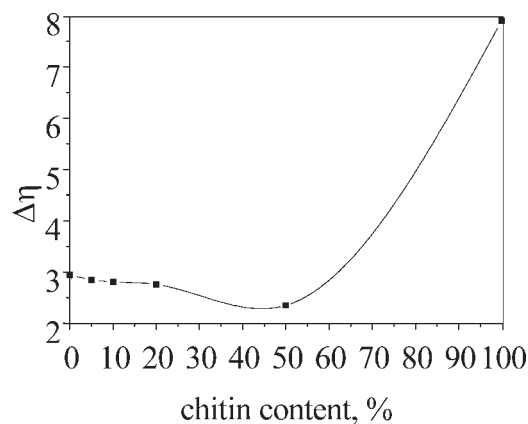


Figure 3 Dependence of $\Delta\eta$ on the blend ratio at 25°C.

tions). The addition of chitin destroys the network that is formed through cellulose macromolecules, and this leads to a decrease in $\Delta\eta$. Resistance among flow layers is weak, and n increases; this favors the spinning process. Elastic deformation of the spinning solution decreases with increasing n when it flows in the spinneret, and the stretching property of the spinning solution is improved; this favors the increase in the spinning velocity.

As shown in Figure 4, the tensile stress of the 9 : 1 membrane is higher than that of the other membranes.

Considering the rheological properties of the investigated solutions and the higher intensity of the 9 : 1 membrane in comparison with the other membranes, we chose 9 : 1 blend solutions to prepare blend fibers. Therefore, we investigated the rheological properties of 9 : 1 blend solutions to provide data for further fiber spinning.

Rheological properties of 9 : 1 blend solutions

Dependence of n on the temperature

n of 5–8 wt % 9 : 1 blend solutions varies slowly with changes in the solution temperature (Fig. 5). n of a 9 wt % solution is much lower than that of other solutions at the same solution temperature,

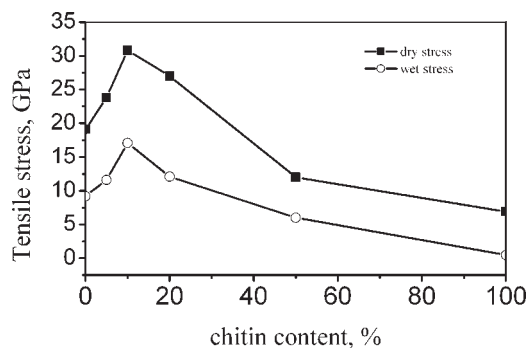


Figure 4 Dependence of the membrane tensile strength on the chitin content.

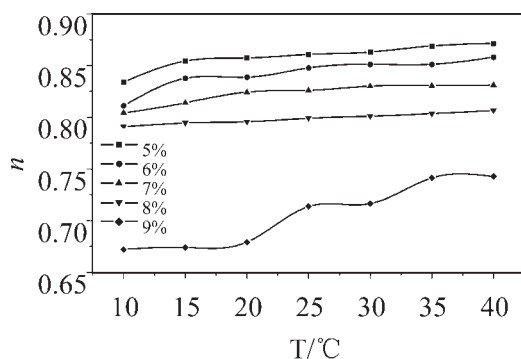


Figure 5 Dependence of n on the 9 : 1 solution temperature.

which is kept around 10–20°C, and then increases with the solution temperature increasing.

Dependence of $\Delta\eta$ on the temperature of the solution

In a certain solution, $\Delta\eta$ decreases slowly with increasing solution temperature (Fig. 6). At a certain temperature, $\Delta\eta$ increases uniformly with increasing concentration. $\Delta\eta$ of a 9 wt % 9 : 1 solution is apparently higher than that of the other investigated solutions, which increases from 4 to 17 at 25°C. This involves a high solution viscosity at 9 wt % and a quick increase in the amount of entanglement among macromolecules. Thus, the flow property of a 9% blend solution worsens.

As we know, the solution viscosity determines the spinnability and maximum spinneret stretching of a spinning solution. On the one hand, a solution cannot be used for spinning if the solution viscosity is too low. On the other hand, the elasticity of the viscose increases with viscosity, and this leads to an increase in the spinnability and maximum spinneret stretching of the solution. When the solution viscosity reaches a certain range, the shear stress and extent of extrusion swelling of the solution also increase, and this leads to a decrease in the maximum spinneret

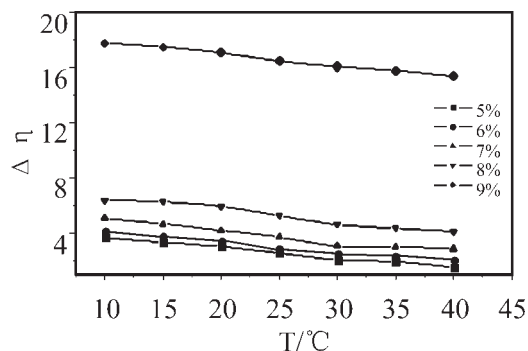


Figure 6 Dependence of $\Delta\eta$ on the 9 : 1 solution temperature.

stretching and spinnability. When the viscosity reaches its highest value, the filtration of the solution is difficult to carry out, and this leads to difficulty with filtration and deaeration, high energy consumption, a low spinning velocity, and so forth. The solution at such a high viscosity is not fit for spinning.

Therefore, 7–8 wt % is the best concentration for a 9 : 1 blend solution; at this concentration, the rheological property is stable.

Activation energy of flow (E_η)

According to the Arrhenius equation, the temperature and viscosity have the following relationship:

$$\eta = A \exp(E_\eta/RT)$$

or

$$\ln \eta = \ln A + E_\eta/RT$$

where E_η is a measure of the dependence of the viscosity on the temperature, T is the absolute temperature of the solution in the unit K, R is the gas constant and the value is 8.314 J/K·mol. The bigger the E_η value is, the stronger the influence is of the temperature on the viscosity, and we may find the possibility of changing the flow ability through the temperature. The temperature increase aids the motion of macromolecules and the expansion of the solution space, so the space among macromolecules is increased and the flow ability of the solution increases; this leads to the decrease in η_a .

Figure 7 shows that the dependence of the zero shear viscosity (η_0) on the temperature of a chitin xanthate solution is higher than that of the other solutions. The relationship between η_0 and temperature T can be described as follows:

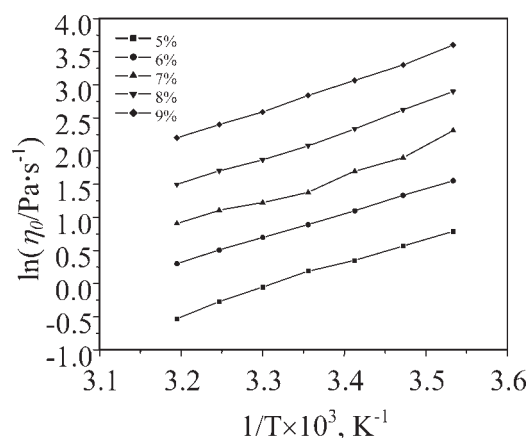


Figure 7 Dependence of η_0 on the 9 : 1 solution temperature.

TABLE I
Statistical Results for the Disassembling Frequency of the Filtering Machine

Sample	Spinning solution	Disassembling frequency of the filtering machine
1	Ordinary cellulose xanthate solution (7–8 wt %)	5–6 batch
2	9 : 1 blend solution (7–8 wt %)	5–6 batch

For a 5% 9 : 1 solution

$$\ln \eta_0 = -12.46 + 3.75 \times (1/T \times 10^3)$$

For a 6% 9 : 1 solution

$$\ln \eta_0 = -11.50 + 3.70 \times (1/T \times 10^3)$$

For a 7% 9 : 1 solution

$$\ln \eta_0 = -11.88 + 3.98 \times (1/T \times 10^3)$$

For an 8% 9 : 1 solution

$$\ln \eta_0 = -11.72 + 4.13 \times (1/T \times 10^3)$$

For a 9% 9 : 1 solution

$$\ln \eta_0 = -10.93 + 4.10 \times (1/T \times 10^3)$$

Therefore, the values of E_η are derived as follows:

For a 5% solution

$$E_\eta = R \times 3.75 = 31.18 \text{ kJ/mol}$$

For a 6% solution

$$E_\eta = R \times 3.70 = 30.76 \text{ kJ/mol}$$

For a 7% solution

$$E_\eta = R \times 3.98 = 33.09 \text{ kJ/mol}$$

For an 8% solution

$$E_\eta = R \times 4.10 = 34.09 \text{ kJ/mol}$$

For a 9% solution

$$E_\eta = R \times 4.13 = 34.34 \text{ kJ/mol}$$

The E_η values for 5–9% 9 : 1 solutions do not vary a lot despite experimental error, and they are all lower than that of relatively pure chitin xanthate (unpublished data) and higher than that of relatively pure cellulose xanthate.

Therefore, the cellulose/chitin solution showed unique rheological behavior that was very sensitive to the temperature, solution concentration, and solution composition. By changing them, we could improve the fiber structure during the regeneration process. Here, this improved viscose method was adopted to spin cellulose/chitin blend filaments from 9 : 1 cellulose/chitin blend solutions according to their rheological character.

Filtering property of 9 : 1 blend solutions

Table I presents the statistical results for the disassembling frequency of the filtering machine. The disassembling frequency of a 7–8 wt % 9 : 1 cellulose/chitin xanthate blend solution does not change more in comparison with that of a pure cellulose xanthate solution, and this means that the addition of 10% chitin xanthate to a cellulose xanthate solution does not vary the filtering properties of the spinning solution greatly. Therefore, a 9 : 1 blend solution has an excellent filtering property as a pure cellulose viscose spinning solution should be feasible for further spinning production.

Mechanical properties of the fibers

Table II presents the mechanical properties of 9 : 1 blend fibers and pure cellulose fibers. The dry and wet strengths of pure cellulose fibers are 2.4 and 1.2 cN/dtex, respectively, which are higher than those

TABLE II
Physical Properties of Fibers Spun from 7% Solutions

Sample	Chitin content (%)	Denier (dtex)	Dry intensity (cN/dtex)	Wet intensity (cN/dtex)	Ratio of wet intensity to dry intensity (%)	Ratio of dry stretching (%)	Uniformity of dyeing (%)
1	0	1.28	2.4 ± 0.2	1.2 ± 0.2	50.0 ± 2.0	16.0 ± 2.0	3.4
2	10	1.37	1.96 ± 0.3	1.08 ± 0.2	55.1 ± 4.0	14.8 ± 1.0	3.6
3	10	1.00	0.66	0.15	22.7	5.7	—

Sample fibers 1 and 2 were prepared in our production line. Data for sample 3 were taken from Shimizu et al.¹¹

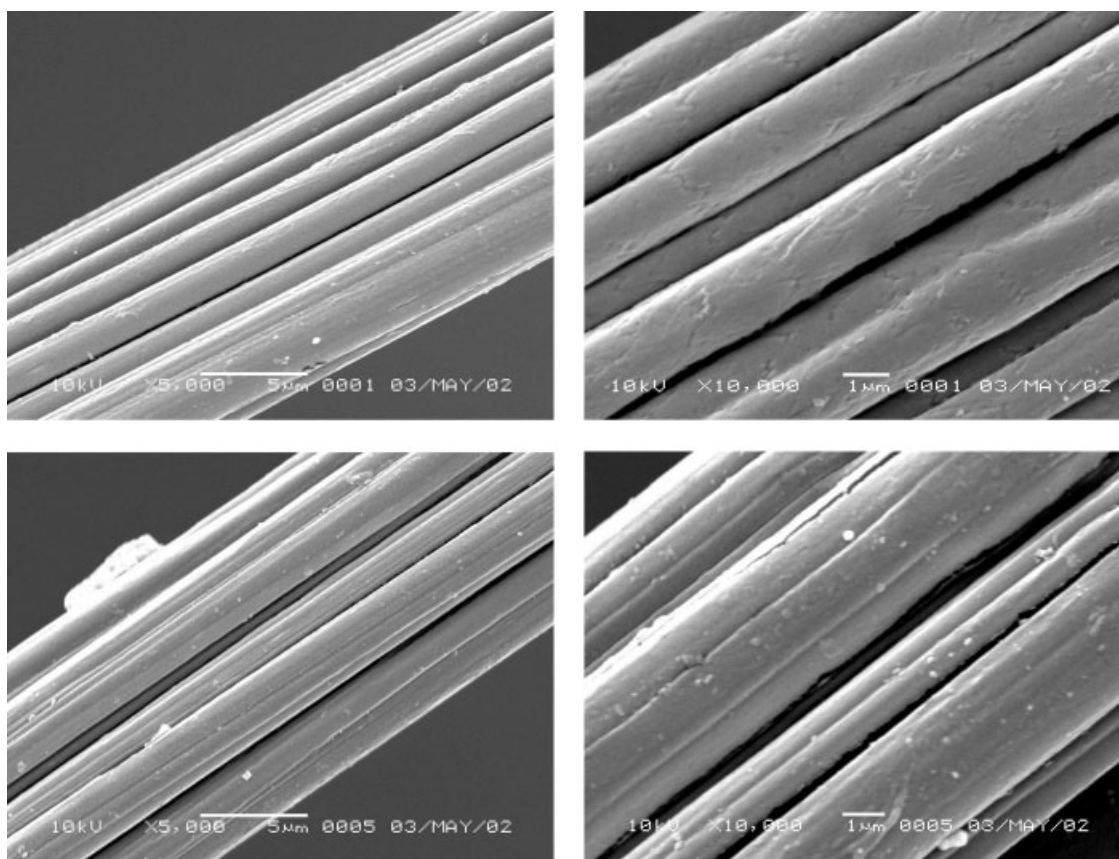


Figure 8 SEM photographs of pure cellulose and 9 : 1 blend fibers.

of the 9 : 1 blend fibers, 1.96 and 1.08 cN/dtex, respectively. It is proved that even a small addition of chitin interferes with the orientation of cellulose macromolecules, which is closely related to the orientation of the macromolecules. However, the mechanical properties of the blend fibers still reach the national criteria and fit the need for further processing. The ratio of the wet intensity to the dry intensity of pure cellulose fibers is higher than that of 9 : 1 blend fibers, and this means that the mechanical property loss of 9 : 1 blend fibers in the wet state is more than that of pure cellulose fibers. The ratios of dry stretching for the pure cellulose and 9 : 1 blend fibers are almost the same, and this means that 9 : 1 blend fibers have excellent further processing properties, that is, excellent weaving properties, just like pure cellulose fibers.

As also shown in Table II, the blend fiber spun from a 7% 9 : 1 blend solution has much higher dry and wet intensities, that is, 1.96 and 1.08 cN/dtex, respectively, than those of Crabyon fiber, that is, 0.66 and 0.15 cN/dtex, respectively,¹³ and this shows that Crabyon is difficult to weave alone, but the prepared blend fiber can be processed alone because its mechanical properties are strong enough. The ratios of the wet intensity to the dry intensity of the blend and Crabyon fibers are 55.1% and 22.7%,

respectively, and this shows that the mechanical loss of Crabyon fiber in the wet state is much higher than that of the blend fiber. The ratios of dry stretching of the 9 : 1 blend fiber and Crabyon fiber are 14.8% and 5.7%, respectively, so the weaving property of the blend fiber is much better than that of Crabyon fiber. All these data prove that the viscose method which we have adopted is an efficient way of preparing cellulose/chitin xanthate blend fibers with satisfactory mechanical properties and processing properties in comparison with Crabyon.

Figure 8 shows SEM photographs of pure cellulose and 9 : 1 blend fibers. The SEM photographs of the fibers show grooves on the surface of the 9 : 1 blend fibers, and the surface of the 9 : 1 blend fibers is coarser than that of the pure cellulose fiber. The 9 : 1 blend fiber could not endure 10 kV during SEM observation, whereas the pure cellulose sample could endure a longer time; this may suggest that the thermal stability of the 9 : 1 blend fiber sample was worse than that of the pure cellulose fiber sample.

CONCLUSIONS

The n values of cellulose xanthate, chitin xanthate, and their blend solutions with different blend ratios

vary in the following order at a constant concentration of 5 wt % and 25°C: chitin < cellulose < 9.5 : 0.5 blend < 9 : 1 blend < 8 : 2 blend < 5 : 5 blend. On the contrary, $\Delta\eta$ of the investigated solutions varies in the following order: chitin > cellulose > 9.5 : 0.5 blend > 9 : 1 blend > 8 : 2 blend > 5 : 5 blend.

For a 9 : 1 blend solution, n increases and $\Delta\eta$ decreases with the solution temperature increasing. Increasing the solution temperature improves the fluidity of a 9 : 1 blend solution in a certain temperature range. η_0 decreases in an index manner with the solution temperature increasing.

A 9 : 1 blend solution has good filtering and rheological properties and is ideal for spinning fibers.

The dry and wet intensity ratio of the blend fiber spun from a 7% 9 : 1 blend solution is lower than that of a pure cellulose fiber and is much higher than that of Crabyon fiber; it still reaches the national criteria and fits the need for further processing. It proves that the viscose method that we have developed here is an efficient way of preparing cellulose/chitin blend fibers with satisfactory mechanical properties and processing properties. SEM

photographs show that the surface of 9 : 1 blend fibers is coarser than that of pure cellulose fibers.

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