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Upgrading Paper-Grade Pulp as Dissolving Pulp for Lyocell Fiber Preparation

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Abstract: Lyocell fiber has emerged as a new generation of green fiber due to its preparation process and unique properties in comparison with viscose fiber. The raw material for the preparation of Lyocell fiber has a great impact on the quality of the finished product. However, unlike viscose-grade dissolving pulp, there is no evaluation system for Lyocell-grade dissolving pulp, making it difficult to assess the quality of the raw material. This study examined the approach to upgrade the paper-grade pulp to dissolving pulp for the preparation of the raw material for Lyocell fiber. Under the sequence of caustic extraction, acid treatment, and enzymatic treatment, the pulp was prepared with competitive properties compared to the commercial Lyocell-grade dissolving pulp. The assessment of prepared pulp was also accomplished by characterizing the pulp properties, the dissolution properties, and the spinnability and stability of Lyocell solution using the prepared sample. In addition, the dissolution mechanism and influencing factors of pulp in the system were elucidated, providing a theoretical basis for upgrading paper-grade pulp to dissolving pulp for Lyocell fiber production.

Keywords: dissolving pulp; Lyocell fiber; assessment; dissolution properties; spinnability



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1. Introduction

Lyocell fiber is produced by solvent spinning, using dissolving pulp and cotton fiber as the main raw materials, and N-methylmorpholine-N-oxide (NMMO) as the solvent. It is a physical dissolution of cellulosic materials without chemical changes, but with various side reactions and byproducts, mainly referring to homolytic and heterolytic side reactions, accomplished by trapping radicals, formaldehyde, and N-(methylene)iminium ions [1]. It is called the “green fiber” of the 21st century due to the lack of harmful reagents added during preparation such as carbon disulfide in viscose production, thus lessening toxic and harmful wastewater, waste gas, and waste residue discharge. Lyocell fiber combines the advantages of natural fiber and synthetic fiber; it has the properties of moisture absorption, air permeability, and comfort akin to cotton fiber, while its strength performance is much higher than that of cotton and ordinary viscose fiber, but close to that of polyester fiber. Considering the unique properties of Lyocell fibers, a variety of high value-added woven and knitted products have been developed, widely applied in the fields of clothing, textile, nonwoven fabric, industrial filter cloth, industrial silk, specialty paper, and other industrial fields [2].

The market prospect of Lyocell fiber is extremely broad, growing from 760 million EUR in 2016 to over 1.35 billion EUR by 2024 [3]. With the rapid development of Lyocell fiber, many problems in the production process need to be solved. In addition to the spinning process and solvent recovery process, the poor dissolution properties of dissolving pulp have become the bottleneck for the massive industrialization of Lyocell fiber [4]. Improving the dissolution properties of raw material is of great significance to the production of Lyocell fiber, not only broadening the source of raw materials, but also enhancing the quality of spinning solution, which further upgrades the properties of finished product [5]. In addition, unlike viscose fiber, there is no evaluation system for the raw material of

Lyocell fiber, which cannot effectively connect the upstream pulp products of Lyocell fiber with the downstream spinning performance.

The impact of pulp on Lyocell fiber production and product quality can be summarized in several aspects [6]. First, the pulp contains impurities other than cellulose, such as hemicelluloses, lignin, extractives, and metal ions. Most of these impurities have negative impacts. As the pulp enters the Lyocell solution system, these impurities cannot be removed during the filtration process, which affects the spinnability of the Lyocell solution. At the same time, the increasing concentration of impurities in the system can further affect the solvent recovery. In order to meet the use requirements, the raw materials are generally treated by purification, depolymerization, and metal ion removal. The available methods include cold caustic extraction [7], enzymatic treatment [8], and acid treatment [9]. Secondly, the stability and rheology of Lyocell solution are key factors in the preparation of Lyocell fiber. The mobile phase of Lyocell solution is mainly affected by the degree of polymerization and the cellulose content of the pulp, which eventually affect the spinnability of the Lyocell solution [10].

Therefore, the approach to upgrade paper-grade pulp to dissolving pulp was performed by conducting cold caustic extraction, acid treatment and enzymatic treatment in sequence. By elucidating the dissolution mechanism and influencing factors of pulp in NMMO solution system, and analyzing the dissolution properties and spinnability of Lyocell solution, the treated pulp was well assessed for application as Lyocell-grade dissolving pulp, suggesting a practical approach to provide raw material for Lyocell fiber production.

2. Materials and Methods

2.1. Upgrading Approach

The raw material used was a bleached softwood kraft pulp (paper grade) provided by Shandong Sun Holdings Group, China. The studied upgrading process was as follows: the raw material was first treated with alkali extraction (using sodium hydroxide) to purify the cellulose, and then with acid (using sulfuric acid) to remove the metal ions. The enzymatic treatment was also performed at the last stage to elucidate its influence on the pulp properties and dissolution properties. The control sample was commercial Lyocell-grade dissolving pulp supplied by Grecell Co., Ltd., Shenzhen, China. The parameters of the experiment are listed in Table 1. All chemicals were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. with analytical grade. Enzymes (mainly hemicellulase) were provided by Leveking, China. The samples were treated with alkaline extraction, different acid treatments, and with/without enzymatic treatments, as shown in Table 2.

Table 1. The parameters of treatments.

Treatment	Enzyme Dosage (ppm)	Alkaline Concentration (%)	Acid Concentration (%)	Temperature (°C)	Pulp Consistency (%)	Time (min)
Enzymatic treatment (E)	100	—		50	5	120
Caustic treatment (C)	—	6		40	10	60
Acid treatment I (A1)			2	80	10	60
Acid treatment I (A2)			0.25	100	10	60
Enzymatic treatment (E)	100	—		50	5	120

Table 2. The treatments for sample 1# to 4#.

No.	Sequence
1#	C + A ₁
2#	C + A ₁ + E
3#	C + A ₂
4#	C + A ₂ + E

2.2. Characterization of Treated Pulp

The pulp properties of treated samples were characterized according to standards: ISO 699-1992 for alkali resistance (R10 and R18), ISO 2144-2015 for ash content, ISO 5351-2010 for intrinsic viscosity (degree of polymerization), ISO 779-2001 for iron content, ISO 777-2001 for calcium and magnesium content, and ISO 778-2201 for copper content.

2.3. Lyocell Solution Preparation

Lyocell solutions were prepared by distilling 76% (*w/w*) NMMO aqueous solution with treated pulp under continuous mixing and vacuum, removing water in a rotary evaporator (YaRong RE-2000A, Shanghai, China) until an 87% (*w/w*) NMMO aqueous solution was obtained. Then, 0.5% *n*-propyl gallate was added as an antioxidant. A 1% concentration of lyocell solution was obtained. A higher 7% concentration of treated samples was prepared by shear mixing and vacuuming in a three-neck bottle heated in an oil bath at 105 °C. The dissolution was completed when an 87% (*w/w*) NMMO aqueous solution was reached, determined using a polarizing microscope (DM2700P Leica, Heidelberg, Germany) until no bright spot was observed; the dissolution time was recorded, and the Lyocell solution was saved for further characterization.

2.4. Characterization of Lyocell Solution

2.4.1. Determination of Yield of Regenerated Cellulose

Taking m_1 (g) of Lyocell solution into water to precipitate, the precipitate was then filtered, washed with hot water until the filtrate was colorless, and dried in an oven at 105 °C to constant weight m_2 (g). The yield was calculated as the ratio of m_2 to m_1 .

2.4.2. Determination of Stability of Lyocell Solution

Lyocell solution was placed in a certain amount of deionized water to precipitate, and then vacuum-filtered to collect the filtrate. The color index (E) was calculated by quantifying the concentration (C) of NMMO referring to [11] and the absorbance (ABS) at 633 nm using an ultraviolet spectrophotometer (DR 6000, HACH, Loveland, CO, USA). The color index value (E) reflected the stability of the Lyocell solution as calculated in Equation (1); a lower E value implied a better stability of the Lyocell solution.

$$E = (ABS \times 4000)/C. \quad (1)$$

2.4.3. Rheology Properties of Lyocell Solution

The shearing rheology at 90 °C was determined using a HAAKE RS600 rheometer (P35/TiL plate, ThermoFisher Scientific, Waltham, MA, USA). The steady-state rheological properties were performed at a shear rate, γ , of 0.001–100 s^{−1}, from which the Newton index (n), consistency fraction (K), zero-shear viscosity (η_0), and structural viscosity index ($\Delta\eta$) were calculated. The dynamic-state rheological properties were performed in a frequency range, ω , of 0.1–100 rad/s, from which the storage modulus G' , loss modulus G'' , G_c at intersection, polydispersity coefficient π , and frequency f_c corresponding to the intersection were calculated.

3. Results and Discussion

3.1. Characterization of Treated Pulp

The paper-grade pulp was upgraded to dissolving pulp via the sequence of alkaline extraction, followed by acid treatment with/without enzymatic treatment. The results are shown in Table 3. The applied treatments were beneficial to purify the cellulose content; R10 was enhanced by 4.80% to 5.96%, and R18 was enhanced by 5.09% to 7.05% compared to the raw material. Acid treatment is commonly applied to remove ash and metal ions [10]; the applied acid treatment successfully reduced the ash content to as low as 0.01%, while reducing iron, magnesium and calcium ions. Meanwhile, the intrinsic viscosity of samples was diminished by 33.05% to 33.48% by acid treatment, indicating its impact on adjusting

the polymerization degree of pulp. The enzymatic treatment had a positive effect on the low-molecular-molecular weight carbohydrate removal, as the difference between R10 and R18 was slightly lowered after treatment [12].

Table 3. Characterization of pulp properties.

Sample	Intrinsic Viscosity (mL·g ⁻¹)	R10 (%)	R18 (%)	R18–R10 (%)	Ash (%)	Fe (mg·kg ⁻¹)	Mg (mg·kg ⁻¹)	Ca (mg·kg ⁻¹)	Cu (mg·kg ⁻¹)
1#	430	90.1	91.8	1.7	0.01	ND	39	ND	ND
2#	436	89.6	90.9	1.3	0.01	4.0	34	ND	ND
3#	468	90.6	92.6	2.0	0.01	21	41	ND	ND
4#	468	90.3	92.0	1.7	0.01	7.0	25	55	ND
Control	456	89.8	95.0	5.2	ND	5.6	34	ND	ND
Raw material	699	85.5	86.5	1.0	0.16	9.0	143	197	ND

ND-not determined.

The pulp properties of treated samples complied well with those of commercial Lyocell-grade dissolving pulp. The degree polymerization of 1# and 2# pulp was lower due to the use of a higher concentration of acid treatment. The 3# pulp had a relatively higher amount of metal ions, which may affect solvent recovery in the subsequent NMMO system [13]. The R18 of the four kinds of pulp samples was lower than that of the control sample, suggesting higher hemicellulose residual [12], which consequently might have a certain impact on the spinnability and stability of the Lyocell solution made from the sample [14,15].

3.2. Dissolution Properties of Treated Pulp

The dissolution experiment was carried out under a lower pulp consistency (1%, *w/w*), and the results are shown in Table 4. The dissolution processes of the four treated pulps were quite different from that of the control sample with regard to the dissolution time. It can be seen that the control samples were mostly dissolved at 30 min, and the complete dissolution was observed at 40 min. The treated pulps started to dissolve after 50 min, and then completely dissolved up by 85 min.

Table 4. Characterization of dissolution properties.

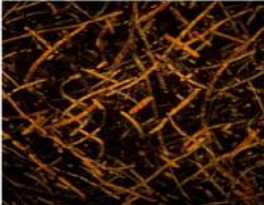
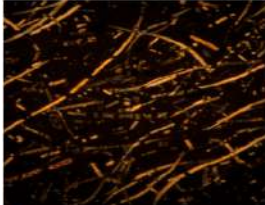
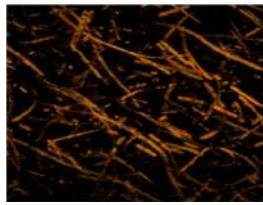
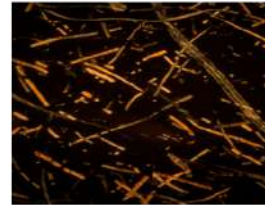
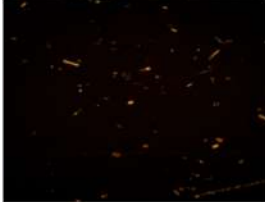
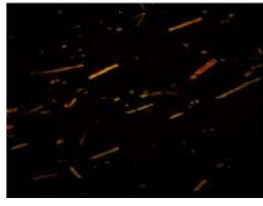
Sample	Lyocell Solution Viscosity (cp)	Dissolution Time (min)	Yield (%)	Stability
1#	125	85	94.56%	126.96
2#	114	70	93.28%	108.90
3#	127	80	90.91%	167.86
4#	125	60	89.47%	192.65
Control	164	40	93.17%	96.00

The viscosity of the spinning solution of the samples after dissolution was lower than that of the control sample. The viscosity was conventionally related to the degree of polymerization and the chemical composition (mainly cellulose and hemicelluloses) of pulp [16]. It can be seen that the hemicellulose content of the four samples was higher than that of the control sample, which contributed to the lower viscosity. The yield of regenerated cellulose simulated the effective utilization rate of the pulp converted into Lyocell fiber. The results showed that the 1# and 2# samples were equivalent to the control sample, while the yields of 3# and 4# samples were slightly lower; this result was different from the viscose fiber yield, represented as R10 in Table 3. In addition to cellulose, hemicelluloses and other organic compounds would remain in Lyocell fiber, contributing to the higher yield.

The stability of Lyocell solution was analyzed by comparing the color index of the liquid after spinning. The results indicate that samples 1# and 2# approached that of the control sample, while stabilities of samples 3# and 4# were slightly weakened, which was most likely due to the incomplete removal of metal ions under the mild acid treatment [1].

A higher concentration of Lyocell solution (7% pulp consistency) was prepared to simulate the actual Lyocell fiber production. As observed in Table 5, the complete dissolution times of the four samples were about 40 to 45 min, leading to clear and transparent spinning solutions.

Table 5. Observation of dissolution process.

Dissolution Time	1#	2#	3#	4#
20 min				
30 min				
40 min				
45 min				

3.3. Rheology Properties of Treated Pulp

The stability and rheology of Lyocell solution are the keys to prepare Lyocell fiber. By determining the rheological properties of Lyocell solution, the spinnability of the pulp in the production line could be simulated.

3.3.1. Comparison of Steady-State Shear Flow Performance

As shown in Figure 1, the apparent viscosity of cellulose solution decreased with the increase of shear rate, which was a typical pseudoplastic non-Newtonian fluid [17]. At the same shear rate, the apparent viscosities of cellulose solution were sorted in descending order: 2# > 1# > 3# > 4# > control sample. With the increase in shear rates, the apparent viscosities of cellulose solutions of different samples tended to be similar. This phenomenon could be explained by the physical crosslinking between polymer molecular chains via intertwining or van der Waals force [18]. These crosslinking points were in the dynamic equilibrium of continuous disintegration and reconstruction under the effect of molecular thermal motion; thus, the fluid had a transient spatial network structure. When the shear rate increased, the dynamic equilibrium moved, and some of the entanglement points between chains were removed, resulting in a decrease in the apparent viscosity of the fluid. As the content of polymer in the solution deviated from Newtonian fluid, the shear

thinning became more obvious. Therefore, at a high shear rate, the apparent viscosity of the solution tended to be similar.

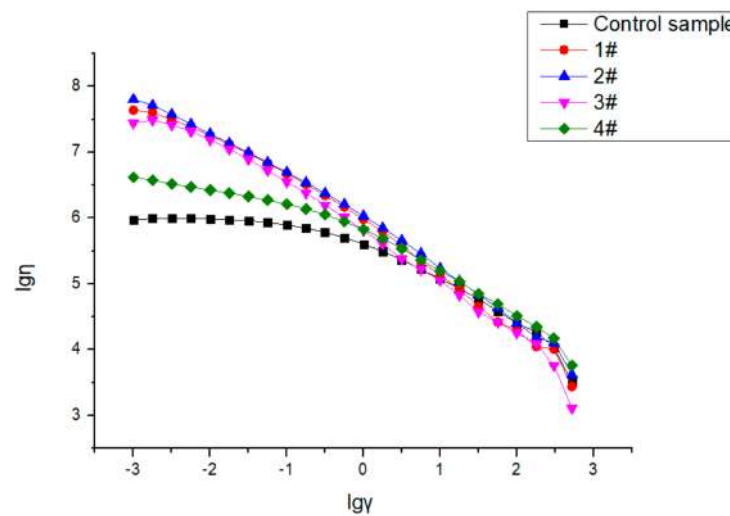


Figure 1. Comparison of steady-state rheological curves.

The Lyocell solution containing cellulose/NMMO/water is a viscoelastic fluid; thus, the related specifications including Newton index (n), consistency fraction (K), zero-shear viscosity (η_0), and structural viscosity index ($\Delta\eta$) were calculated, as listed in Table 6. Among them, n represents the sensitivity of Lyocell solution to shear, whereby a smaller value denotes a more obvious phenomenon of shear thinning, and $\Delta\eta$ is used to characterize the structural degree of Lyocell solution, relating to the spinnability of the solution, whereby a smaller value denoted better spinnability. As shown in Table 6, sample 4# was less affected by shear, while 3# was more sensitive to shear. The Lyocell solution made from sample 4# had the best spinnability, which was equivalent to the control sample. It was suggested from the steady-state rheological data that a smaller n value led to greater K , η_0 , and $\Delta\eta$ values, with the Lyocell solution deviating more from Newtonian fluid, resulting in a poorer spinnability of the solution. The spinnabilities of samples were ranked in descending order: control sample > 4# > 2# > 1# > 3#.

Table 6. Rheological properties.

Sample	Steady State				Dynamic State				
	n	K	η_0 (Pa·s)	$\Delta\eta$	G_c (Pa)	π	f_c (Hz)	w_c (r·s ⁻¹)	t_c (s)
1#	0.271	644.6	43,600	17.88	6435	15.5	1.26	7.94	0.126
2#	0.289	787.4	63,900	17.29	-	-	-	-	-
3#	0.267	488.3	27,800	18.22	515	194.2	0.63	3.98	0.251
4#	0.529	365.2	4170	12.69	740	135.1	0.20	1.26	0.794
Control	0.614	197.8	941	11.40	994	100.6	1.22	7.64	0.131

3.3.2. Comparison of Dynamic-State Shear Flow Performance

In the production of Lyocell fiber, the flow of spinning fluid in the spinneret is a one-dimensional steady shear flow; hence, the apparent viscosity and elasticity obtained from the steady-state experiment have the most practical significance. However, the range of steady-state experiment is limited in the laboratory, and the actual spinning is difficult to obtain elasticity measurement directly. Therefore, the dynamic flow performance of the spinning solution was measured through the oscillation mode of the HAAKE rheometer under the action of normal rotation alternating stress, and the dynamic and steady experimental data were combined to characterize the viscosity and elasticity of the spinning solution.

The dynamic-state rheological curves of Lyocell solutions prepared from different pulps were measured under the oscillating mode to compare with control sample, as shown in Figure 2. The storage modulus G' represents the elasticity of the material, and the loss modulus G'' represents the viscosity of the material. The modulus G_c at the intersection of G' and G'' is related to the relative molecular weight distribution of polymers. As π is equal to $10^5/G_c$, a larger π value indicates a wider relative molecular weight distribution of polymers. The relaxation time t_c expressed by the reciprocal of the intersection frequency w_c corresponds to the fluid entanglement network [19].

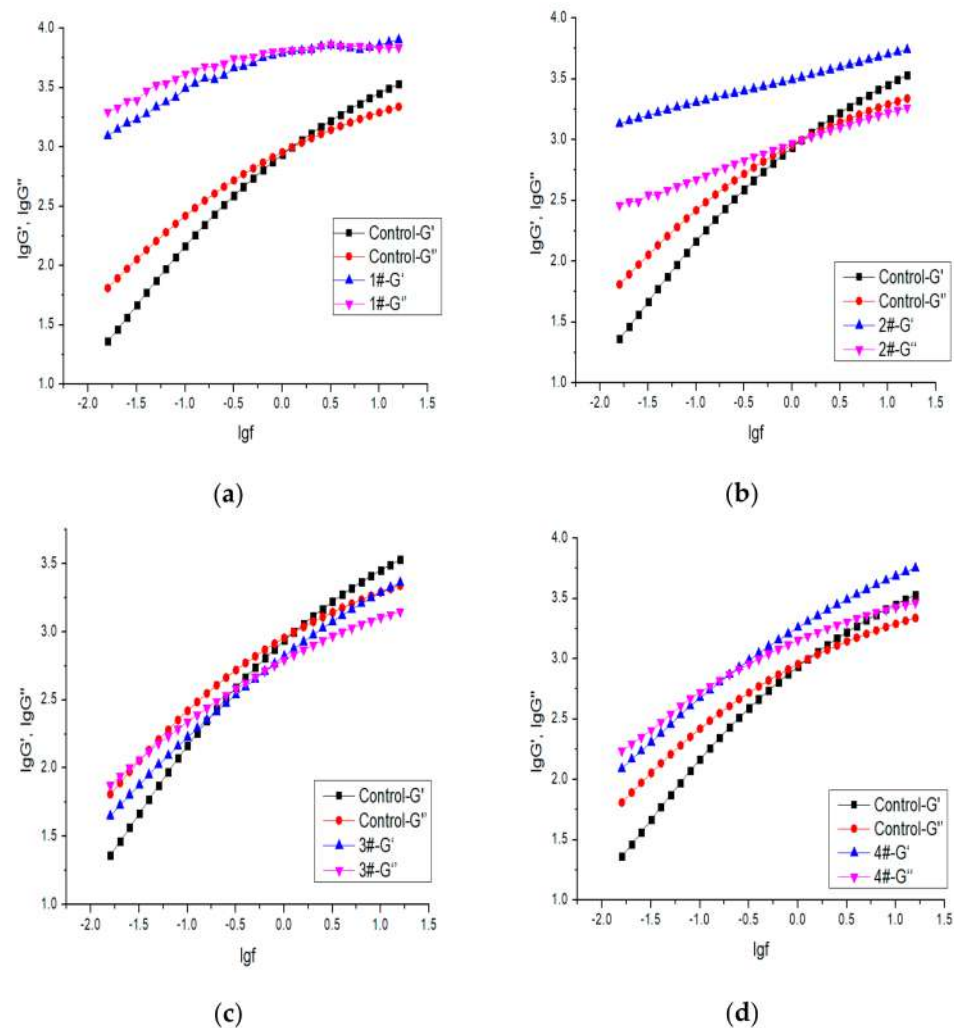


Figure 2. Comparison of dynamic-state rheological curves: (a) sample 1# vs. control sample; (b) sample 2# vs. control sample; (c) sample 3# vs. control sample; (d) sample 4# vs. control sample.

It can be seen from Figure 2 that, with the increase in vibration frequency, the storage modulus G' and loss modulus G'' of sample 1# and 2# solution exhibited a more obvious increase than the control sample solution. This indicates that the viscosity and elasticity of these two solutions would greatly increase during the spinning process, which was not conducive to the spinning of Lyocell fibers. The intersection modulus G_c of sample 4# was equivalent to that of the control sample (shown in Table 6), indicating the similar molecular weight distribution of the two pulps. In addition, sample 3# had the largest G_c value, correlating to the widest molecular weight distribution. As illustrated in the previous section, sample 3# had the poorest spinnability; the wider molecular weight distribution would weaken the spinnability of the Lyocell solution. It should be noted that the relaxation time of sample #4 solution was the largest among others, inferring the most complex fluid entanglement network, which may be attributed to the enzymatic treatment effect. The

entanglement network was a function of degree of polymerization of cellulose; meanwhile, the cellulose content and other components may have also contributed to the complexity. Although enzymatic treatment had a slight effect on the performance of the pulp properties, it had a greater impact on its dissolution and spinning performance.

4. Conclusions

The approach to upgrade paper-grade pulp to dissolving pulp for Lyocell fiber preparation was studied. The Lyocell-grade dissolving pulp was prepared under the sequence of caustic extraction, acid treatment, and enzymatic treatment. The prepared pulp was comparable to commercial Lyocell-grade dissolving pulp with regard to the pulp properties and dissolution properties. The spinnability of pulp was analyzed on the basis of its rheological properties after dissolution. By analyzing the steady-state and dynamic-state rheological data, it was suggested that the pulp with a higher purity with regard to a higher cellulose content, a lower hemicelluloses content, and a trace content of metal ions, along with a relatively narrow molecular weight distribution, was beneficial to the solubility and stability of the spinning solution. Enzymatic treatment is suggested for a better dissolution of pulp for Lyocell fiber production.

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