

Preparation of Wet Strength Paper from Filter Paper with NaOH-Thiourea-Urea Aqueous Solution

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In this work, wet strength paper was prepared from quantitative filter paper pretreated with NaOH-thiourea-urea aqueous solution. The effects of alkali concentration, soaking time, freezing time, and washing time were evaluated through single factor experiments. The optimum conditions were found to be an alkali concentration of 8%, soaking time of 2 seconds, freezing time of 15 minutes, and washing time of 10 minutes. Under these conditions, the wet tensile strength of the modified paper could be increased to 33% of the dry tensile strength and 400% of the wet tensile strength of the body paper. Also, the wet burst strength could be improved to 200% of the dry burst strength and 2400% of the wet burst strength of the body paper. However, there were no significant effects on the structure of the functional groups or crystalline region. Also, there was no toxic material released during the treatment, and the treatment solution was recyclable and environmentally friendly.

Key Words: Wet-strength paper; NaOH-thiourea-urea aqueous solution; Gel; Cellulose

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INTRODUCTION

Recently, a variety of new applications have been found for wet strength paper due to its excellent biodegradability and environmental friendliness (Qian *et al.* 2008). Paper strength comes from the bonding strength of the fibers. However, fibers could be swelled easily by water and the bonds would be damaged, which could cause the paper strength to be decreased substantially, leading to poor wet strength (He 2010). Therefore, the problem has received significant attention.

Usually, wet strength agents have been employed to increase the wet strength of paper. It was reported that some aldehyde polymers (*e.g.* urea resin and melamine formaldehyde resin) (Stratton and Colson 1993; Epsy 1995; Zakaria 2004) could be used to modify paper wet strength. The sizing development of Alkyl Ketene Dimer (AKD) and Alkenyl Succinic Anhydride (ASA) (Obokata and Isogai 2005) were also found to be beneficial for wet strength paper. Recently, polyamide polyamine epichlorohydrin resin (PAE) has been applied as an excellent agent in the improvement of the paper wet strength in paper-making industries (Obokata and Isogai 2005). Also, all the wet-strength reagents mentioned above are synthetic polymers, which are difficult to be regenerated or recycled. For example, much of the urea resin or PAE would be lost in the pressure dewatering process, for both of the polymers are water-soluble. Some formaldehyde or organochlorine could also be released at high temperature, due to the instability of urea resin or PAE under higher temperature of the sheet forming process, which could be harmful to the environment (Obokata and Isogai 2007). For these reasons, the traditional wet strength reagents are difficult to be renewed or recycled (Obokata *et al.* 2005).

Therefore, efforts have been made to discover low-toxic, non-toxic, or biodegradable wet strength agents or paper treatment methods (Kitaoka *et al.* 1999; Qin *et al.* 2006; Aracri *et al.* 2011).

Cellulose products were obtained with a NaOH/H₂O system (Kamide *et al.* 1984; Yamashiki *et al.* 1992). It was found that regenerated cellulose membrane could be prepared from cellulose in NaOH/urea aqueous solution with various coagulants (Mao *et al.* 2006; Zhou *et al.* 2002). It was also reported that novel microporous membranes could be prepared from cellulose in NaOH/thiourea aqueous solution by coagulating with an ammonium sulphate aqueous solution (Ruan *et al.* 2004). NaOH-thiourea-urea aqueous solution, modified according to the three systems mentioned above, has also been used as a new solvent for cellulose (Jin *et al.* 2007) to generate cellulose membranes and other products due to its advantages of high solubility, low cost, rapid dissolution, and environmental friendliness.

To the best of our knowledge, there have been no reports on the improvement of paper wet strength with NaOH-thiourea-urea aqueous solution. It is known that NaOH could swell the amorphous regions of cellulose (Yang 2008) and urea or thiourea could soak into the crystalline region of swelled fibers, then cellulose would be dissolved with low cellulose concentration (Wang *et al.* 2008). However, the cellulose or paper could be jellified by the solution with higher cellulose concentration (Zhang *et al.* 2010). After solidifying treatment, it would become a solid-state gel paper (exhibiting increased water-resistance and stronger bonding) according to our tests. All of the reports above are beneficial to increasing the paper wet strength. In the present work, the reaction conditions were mild (*e.g.* low temperature and short treatment time), so the ratio of degraded cellulose was small and there was relatively little effect on yield (about 98% under the optimal conditions). The goal of the present work was to find out whether such a mild treatment has potential to modify the paper wet strength.

EXPERIMENTAL

Preparation of Material and Treatment Solution

Quantitative filter paper (FLP) (Hangzhou Whatman-Xinhua Filter Paper Co., Ltd.) was dried at room temperature for 72 hours (h) and then the solid content was measured.

NaOH, thiourea, and urea were dissolved in deionized water at the mass ratio of 8:6.5:8:77.5 (NaOH:thiourea:urea:H₂O; Zha *et al.* 2008), and the solution concentration was stabilized at room temperature for 72 h.

Experimental Procedure

The filter paper was added to the NaOH-thiourea-urea aqueous solution and was soaked at room temperature. The paper was removed from the solution and frozen at -10 °C (Wang *et al.* 2008). Then it was washed with anhydrous ethanol and plasticized using aminoalkyl silicone fluid emulsion. After the treatment of NaOH-Thiourea-urea aqueous solution in our tests, the brittleness of the treated paper would be increased significantly. As is known, while other qualities may be similar, paper with high brittleness has much lower strength than that of the paper with low brittleness (Casey 1980; Fengel and Wegener 1984). Therefore, paper plasticizing after the washing process was essential in the present work. The effects of the glycerinum, methyl silicone oil, and aminoalkyl

silicone fluid emulsion on the modification of plasticity were compared in preliminary work by the authors, and the aminoalkyl silicone fluid emulsion was applied in the work presented in this paper. Finally, the treated paper was air-dried at room temperature. The wet tensile and burst strength were measured to evaluate the effect of NaOH-thiourea-urea aqueous solution (The wet tensile strength or wet burst strength were measured according to the Chinese National Standard (GB/T 453-1989, GB/T 454-1989, GB/T1539-1989); and the soaking time in this work was chosen as 30 min. When the soaking time was longer than 30 min, the untreated paper could not be taken out, for the paper could have been gradually dispersed in water, and whole paper would not be prepared in our tests.).

The untreated and treated paper (under the optimal conditions in this work) were dried in a freeze dryer (Thermo Scientific Heto Power Dry PL6000) for 48 h. The temperature was $-30\text{ }^{\circ}\text{C}$ and the applied vacuum was -0.1 megaPascals (MPa). Freeze-drying was applied in this work to meet the requirement of XRD, SEM, and FT-IR analysis (the samples detected by these equipments should be freezing dried according to the technical specification). During the preparation of wet-strength paper, the product was air-dried, which was also a drying method at lower temperature. The fiber morphology or paper morphology might be changed or destroyed during the drying under high temperature, and fibers or paper would be shrunk sharply with the rapid water loss; or be oxidized by the air under the condition of higher temperature, which would affect the result of SEM, XRD, or FT-IR analysis (shrinking process might affect the morphology or the strength of fiber or paper (Nevell and Zeronian 1985), and an oxidation process would destroy the structure of the functional group or the crystalline region of the fibers (Wilke *et al.* 1983)). The morphology, strength, structure of crystalline region or functional group could be kept integrally with freezing drying method. Therefore, freezing drying method is more suitable for the accuracy of the following instrumental analysis or the modification effect of the NaOH-thiourea-urea aqueous solution on the paper wet strength.

The paper surface morphology was observed using scanning electron microscopy (SEM; FEI Quanta-200, USA). The magnification was 400X. Then, the sample was broken apart with a fiber mill and the powder was screened to obtain the components passing through the 100 mesh screen for analysis with FT-IR spectrum (Nicolet 380, USA) and X-ray diffraction (Bruker D8 Avance XRD device, Germany). The scanning range, in wave numbers, was from 1000 inverse centimeters (cm^{-1}) to 4000 cm^{-1} . The scanning angle of the XRD was from 5° to 50° .

RESULTS AND DISCUSSIONS

Effect of Alkali Concentration

Table 1 lists the wet strength at different alkali concentrations (soaking time of 3 seconds (s), freezing time of 20 minutes (min), and washing time of 20 min). The results indicated that the strength was improved with the increase of alkali concentration. With alkali concentration of 8%, the wet tensile and burst strengths were 668.7 Newtons per meter (N/m) and 171 kilopascals (kPa), respectively, reaching the maximum values. However, the wet strength decreased substantially with increasing alkali concentration.

The cellulose underwent the processes of gelatinization and dissolution during the treatments. The cellulose gelatinization was more obvious with lower alkali

concentration, and at the mild conditions the majority of the fibers gelled together and the number of individual fibers decreased; as this occurred, the wet strength increased gradually. However, with high alkali concentration, the dissolving process would play a main role and the wet strength would be decreased with the dissolution of cellulose. Therefore, alkali concentration of 8% was chosen as the optimal condition.

Table 1. Effect of Alkali Concentration

Alkali concentration (%)	Wet tensile strength (N/m)	Wet burst strength (kPa)
6	206.3	87
7	244.5	93
8	668.7	171
9	484.8	127
10	227.2	100

Effect of Freezing Time and Soaking Time

Table 2 lists the wet strength at different freezing times (alkali concentration of 8%, soaking time of 2 s, and washing time of 20 min) and Table 3 lists the wet strength at different soaking times (alkali concentration of 8%, freezing time of 15 min, and washing time of 20 min).

The strength was improved with the increase of freezing or soaking time. With freezing time of 15 min, the wet tensile and burst strengths were 688.2 N/m and 192 kPa, respectively. The wet tensile and burst strengths were 694.1 N/m and 181 kPa with soaking time of 2 s, respectively, reaching the maximum values for both conditions. However, the wet strength would be decreased substantially with longer soaking time or freezing time.

Table 2. Effect of Freezing Time

Freezing time (min)	Wet tensile strength (N/m)	Wet burst strength (kPa)
5	198.4	64
10	545.1	155
15	688.2	192
20	652.5	178
25	443.6	142

Table 3. Effect of Soaking Time

Soaking time (s)	Wet tensile strength (N/m)	Wet burst strength (kPa)
1	588.2	164
2	694.1	181
3	657.5	175
4	504.3	150

With shorter soaking time or freezing time, the cellulose gelatinization would play a main role during the freezing process with less absorbed solution or higher temperature of the system (paper and absorbed NaOH-thiourea-urea aqueous solution). Many fibers became jelly-like, the number of single fibers was decreased during the freezing process,

and the strength increased gradually. However, excess solution would be absorbed with increasing soaking time, and the system would be cooled continuously with longer freezing time. This would cause the effect of dissolving to be much more significant and the strength might be decreased with the loss of cellulose. Therefore, a soaking time of 2 s and a freezing time of 15 min were chosen as the optimal conditions in the present work.

Effect of Washing Time

Table 4 lists the wet strength at different washing times (alkali concentration of 8%, soaking time of 2 s, and freezing time of 15 min). With washing time of 10 min, the wet tensile and burst strengths were 675.5 N/m and 188 kPa, respectively, reaching the maximum values. However, the paper wet strength was decreased with longer washing time.

Cellulose was in the state of semigel when the freezing process was completed, and there was little strength added during production, so washing or solidification was necessary. With shorter washing time, cellulose semigel became a gel gradually. Then the bonding of the gels would be increased and the wet strength might be improved. However, with longer washing time, the cellulose gel would be solidified completely and then it might be aged. The paper strength could also be decreased by anhydrous ethanol (Yang 2008). Therefore, the wet strength was decreased and washing time of 10 min was determined as the optimal condition.

Table 4. Effect of Washing Time

Washing time (min)	Wet tensile strength (N/m)	Wet burst strength (kPa)
5	435.2	123
10	675.5	185
15	664.4	170
20	640.8	159

Strength Comparison of Body Paper and Treated Paper

Table 5 lists the paper strength of body paper and treated paper. Wet tensile strength of 178.2 N/m and wet burst strength of 7.66 kPa of the body paper were shown in Table 1, and were less than 10% of the dry tensile strength (2069.3 N/m) and dry burst strength (95 kPa). Therefore, the body paper could not be used as wet strength paper (He 2010).

In addition, the dry tensile and burst strengths of treated paper under the optimal conditions were 3348.5 N/m and 332 kPa respectively, indicating that the wet tensile strength was about 21% of the dry tensile strength and the wet burst strength was 57% of the dry burst strength of the treated paper. All of the results above showed that the NaOH-thiourea-urea aqueous solution pretreatment could be used to prepare wet-strength paper.

Table 5. Strength Comparison

Paper samples	Dry tensile strength (N/m)	Wet tensile strength(N/m)	Dry burst strength (kPa)	Wet burst strength (kPa)
body paper	2069.3	178.2	95	8
treated paper	3348.5	693.2	332	189

FTIR Analysis

Figure 1 portrays the FT-IR spectra of untreated and treated FLP and the peaks are assigned by comparing with the literature data (Schmidt *et al.* 2002; Li *et al.* 2010). As shown in Fig. 1, both samples exhibited similar spectra, which indicated that the structure of the functional group could not be altered by the solution under the optimal conditions in this work.

The absorption at 3420 cm^{-1} indicates the stretching of OH groups and the peak at 2900 cm^{-1} is attributed to C-H stretching in CH_2 groups in cellulose. The peak at 1330 cm^{-1} is attributed to C-C and C-O skeletal vibrations. The bands between 1200 and 1000 cm^{-1} are dominated by ring vibrations overlapped with stretching vibrations of C-OH side groups. The peak at 1100 cm^{-1} is indicative of associated OH groups from cellulose. A strong signal at 1050 cm^{-1} is indicative of C-O at C-C stretching.

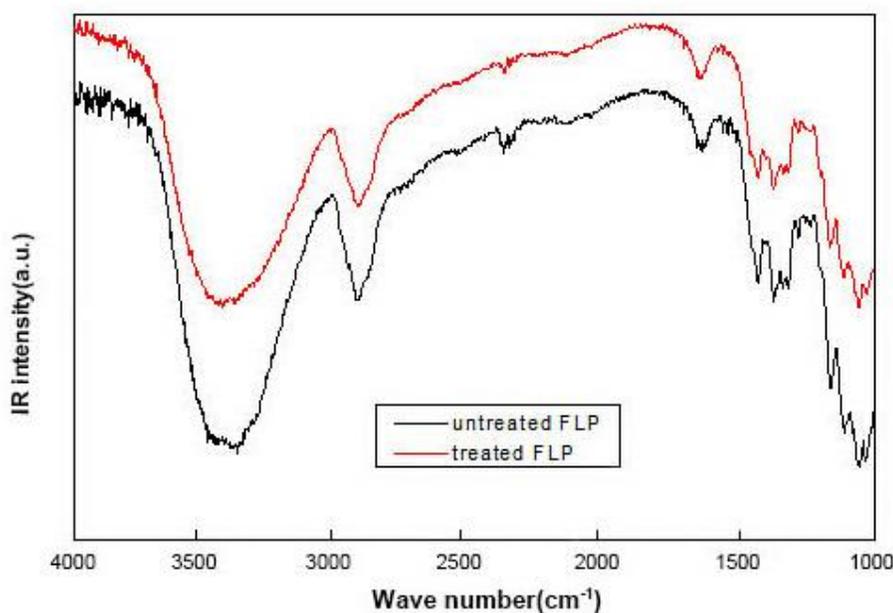


Fig. 1. FT-IR spectra of untreated and treated FLP

XRD Analysis

The XRD spectra of the treated and untreated FLP are shown in Fig. 2. The variation of the spectra was not obvious. Through calculation, the crystallinity of the treated FLP was found to be 0.69, about 0.05 higher than that of the untreated FLP. This indicated that the crystalline structure could not be altered substantially by NaOH-thiourea-urea aqueous solution under the optimal conditions in the present work. The jellification of the solution played the main role in the improvement of paper wet strength.

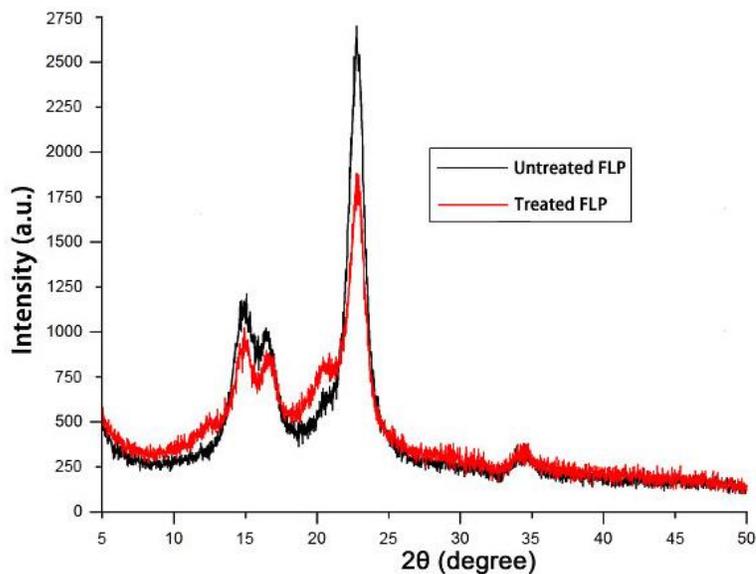


Fig. 2. XRD spectra of untreated and treated FLP

SEM and Some Other Paper properties Analysis

Figure 3 shows the surface morphology of the paper investigated by SEM. Figure 3a displays the morphology of untreated FLP, and Fig. 3b displays that of treated FLP. There were fewer single fibers in the treated FLP after the treatment of NaOH-thiourea-urea aqueous solution. Fibers could be jellified in the solution under the optimal condition detailed by the present work. Therefore, much of the cellulose would become cellulose gel and the single fibers would gradually disappear (Zhang *et al.* 2001, 2002).

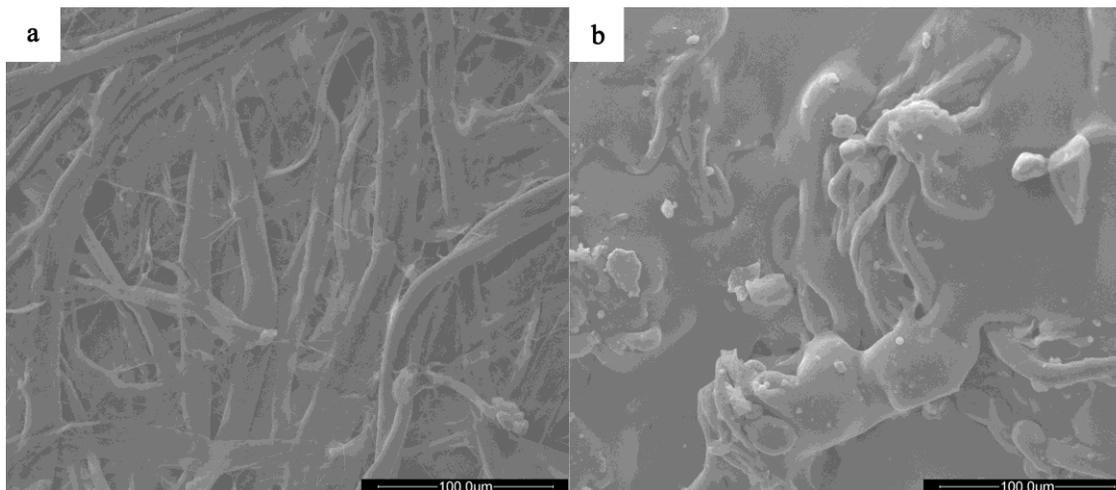


Fig. 3. SEM image of untreated and treated FLP

The treating process in the present work was similar to the procedure of parchment paper or vulcanized paper. The body paper was treated with H_2SO_4 (72%, mass fraction) aqueous solution or ZnCl_2 (65%, mass fraction) aqueous solution (this process was called gelling), then the paper would be transformed to gel paper, and finally, the gel paper was washed and plasticized to prepare the product paper (Cuissinat and

Navard 2008; Zhang *et al.* 2010). Although the surface morphologies was similar to that of membrane, parchment paper or vulcanized paper have also been called paper or wet-strength paper by scholars. Just like the fact that the wet-strength can be increased with wet-strength reagents (*e.g.* urea resin, PAE), paper gelling is also a procedure to prepare wet-strength paper, and usually the product could be used as functional paper in paper-making industries.

During our tests, the tightness, the water absorption, and porosity of the dry body paper and dry treated paper were also measured.

The variations of properties mentioned above are shown in Table 6. The tightness or porosity was modified significantly, which could be helpful for paper strength. Higher tightness meant stronger bonding of the fibers or gels, and lower porosity indicates that it was much more difficult for water to soak into the interior of the paper (Xu *et al.* 1999; He 2010). Therefore, the water absorption would also become more difficult for the treated paper, and the wet strength could be increased significantly and in the present work.

Table 6. Comparison of Some Other Paper Properties

Samples	Tightness (g/cm ³)	Water absorption ratio (%)	Porosity (%)
Body paper	0.68	135%	72%
Treated paper	1.12	50%	45%

Proposed Mechanism

As discussed in the introduction, the amorphous region of fibers could be swelled by NaOH first and then the crystal region could be swelled by urea or thiourea (Wang *et al.* 2008). With higher cellulose concentration, more fibers could be jellified by the solution, and the cellulose semigels would be solidified to gels during the washing process. The resultant cellulose gels could resist the soaking of water. Ungelled fibers might be wrapped by gelled fibers, so it would be much more difficult for water to soak into the interior of the fibers, and there would be little effect on the hydrogen bonding of internal ungelled fibers. Therefore, the wet strength could be increased significantly.

However, the wet strength of paper might decrease substantially under conditions of high alkali concentration or long soaking and freezing time. More cellulose would be dissolved in the solution under these conditions. Therefore, some components, particularly cellulose, in the body paper would be removed. The body paper was made from cotton fibers, softwood fibers, and hardwood fibers (Yang 2008; He 2010). There was almost no lignin or hemicellulose in cotton fibers, and fibers from softwood or hardwood had been cooked with chemical pulping methods, indicating that almost all the lignin, as well as much of the hemicellulose and extractives would have been removed (Zhai and Lee 1989; Zhan 2010). According to the present tests, the content of cellulose in the body paper was about 98%. NaOH-thiourea-urea aqueous solution has been shown to be able to dissolve cellulose at low temperature and cellulose concentration (Jin *et al.* 2007). In the present work, the body paper was treated in such a solution, so some of the cellulose component in the body paper might be dissolved by the solution and could not retain in the filter paper (it would exist in the solution). During the subsequent washing process, the dissolved cellulose would be eliminated with the removal of treatment solution. In the present work, the ratio of dissolved cellulose was much lower under

suitable conditions (high cellulose concentration, low absorption of the NaOH-thiourea-urea aqueous solution or short freezing time, yield of 98% under the optimal conditions in the present work) than that under the drastic conditions (low cellulose concentration, high absorption of the NaOH-thiourea-urea aqueous solution or longer freezing time). The whole filter paper sheet might be dissolved in solution with alkali concentration of 30% and freezing time of 40 min.), which would lead to the decrease of paper strength.

When the NaOH-thiourea-urea aqueous solution was used in the cellulose dissolving process, a low cellulose concentration and a high alkali dosage were required (e.g. cellulose concentration of 5% and alkali dosage of 160%). Also, after the freezing dosage, high speed stirring was applied (Jin *et al.* 2007); thus the cellulose could be dissolved gradually. During these processes, the amorphous region of cellulose could be swelled by NaOH. Then the urea or thiourea might soak into the crystalline region, resulting in the gradual reduction of the fibers bonding. Therefore, the cellulose could be dissolved. The treating or stirring were applied at low temperature, for the dissolving was an exothermic process (Wang *et al.* 2008).

When the NaOH-Thiourea-Urea aqueous solution was used to prepare the wet-strength paper in the present work, high cellulose concentration and low alkali dosage were required (according to our tests or calculation in this work, the cellulose concentration was about 20%, and the alkali dosage was about 18%). Under these conditions, the cellulose could not be dissolved, because the soaking was much more difficult than that under the condition of low cellulose concentration and high alkali dosage. However, the gelling process might play a pivotal role at this time, since some of the fibers would be transferred to the state of semi-gel fibers and become solid gel fibers during the washing process (Zhang *et al.* 2001, 2002; Cai and Zhang 2006). As we know, the bonding of gels was much stronger than that of fibers. Also, the solid gels could resist the soaking of water, and the ungelled fibers were wrapped by the gel fibers. Then it would be much more difficult for water to soak into the fibers interior or treated paper when the treated paper. Therefore, the wet strength might be increased significantly.

CONCLUSIONS

1. The optimal conditions of an NaOH-thiourea-urea aqueous solution treatment were determined to be alkali concentration of 8%, a soaking time of 2 s, a freezing time of 15 min, and a washing time of 10 min.
2. Many fibers could be jellified in the solution and then become cellulose gels. The ungelled fibers might be wrapped by gelled fibers, so the fiber bonding and the water-resistance property could be increased significantly.
3. The structure of the functional group or the crystalline region of FLP could not be altered by NaOH-thiourea-urea solution and the jellification process played the main role under the optimal conditions.

ACKNOWLEDGEMENTS

The authors are grateful for the support of the National Science Foundation of China (Grant No. 31270629) and the Doctorate Fellowship Foundation of Nanjing Forestry University (Grant No. 163105019).

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Article submitted: September 22, 2014; Peer review completed: November 12, 2014;
Revised version received and accepted: December 3, 2014; Published: December 11, 2014.