

Preparation of Dissolving Pulp Made from Poplar Residual Slabs and Effect of Xylanase Post-treatment on Its Reactivity

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The production of dissolving pulp from poplar wood residual slabs was investigated. The residual slab chips were initially prehydrolyzed and subsequently pulped by the kraft process; the resulting brownstock was bleached using a totally chlorine-free (TCF) sequence to full brightness. The pulp contained low pentosans and high α -cellulose content, and the pulp had high reactivity. Its hemicellulose content, reactivity, and degree of polymerization were within acceptable levels for a rayon-grade dissolving pulp. Thus, the residual slabs from poplar can be regarded as a viable raw material for dissolving pulp production. The reactivity of this dissolving pulp was drastically decreased after the xylanase post-treatment, which can slightly lower the pentosans levels. Simultaneously, the crystallinity index of the resulting pulp obviously decreased after xylanase post-treatment.

Keywords: Pre-hydrolysis; Kraft cooking; TCF bleaching; Poplar residual slab; Dissolving pulp; Xylanase

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INTRODUCTION

Dissolving pulps have high purity with high α -cellulose content, high brightness, low hemicelluloses, and low lignin content. They are used as the raw materials for producing cellophane and rayon, cellulose esters, cellulose ethers, and grafted and cross-linked cellulose derivatives.

In view of making dissolving pulp, researchers have embarked on the development of new technologies for the utilization of raw materials, such as cotton linter, aspen wood, jute stem, bagasse, Masson pine, bamboo (Helmy and State 1991; Abad *et al.* 2000; Xu and Jiang 2005; Jahan 2009; Li and Ma 2009; Chen and Yang 2011). With the increasing cost of these raw materials, new alternatives for the production of dissolving pulp are being investigated. Wood residuals, which total about sixty million tons per year in China, is one such alternative raw material that is available in developing countries. Among the wood residuals, the morphology and chemical composition of pulps made from slab residuals of poplar wood display suitable properties; this raw material has been used to produce high-yield pulp or bleached chemical pulp (Gong 2007; Deng *et al.* 2010).

Dissolving pulp quality mainly depends on the pulping process, in addition to the properties of the raw material. Traditionally, there are two methods for producing dissolving pulp. These are extended-time acidic bisulfite cooking (Sixta *et al.* 2004) and prehydrolysis-sulfate (kraft) cooking. The basic idea behind both processes is to remove as much hemicellulose as possible from cellulose fibers, while simultaneously delignifying them, to obtain a high content of alpha-cellulose. In order to maximize the

removal of hemicelluloses and lignin, it is critical to apply a modified pulping process. Prehydrolysis of the wood chips prior to kraft pulping helps to produce high cellulose content and low hemicellulose content in the dissolving pulp. The prehydrolysis kraft process, due to the high lignin removal, is mostly used to produce dissolving pulp from hardwoods (Saeed *et al.* 2012) in comparison to the prehydrolysis soda/AQ process.

The reactivity of dissolving pulps towards derivatization or dissolution is a crucial quality parameter and is mainly determined by the accessibility of the hydroxyl groups. Reactivity is the most significant quality parameter of dissolving pulp and has a large effect on the xanthation reaction during viscose preparation. Generally, high reactivity is difficult to obtain by just increasing the alpha cellulose content of the pulp. Therefore, attention has shifted to enzymatic modification of the pulp to facilitate the accessibility of chemicals to the cellulose. Kvarnlof *et al.* (2006) tested the potential of five commercial cellulases to increase the reactivity of a dissolving pulp for viscose preparation. The addition of an endoglucanase can further increase both the amount of soluble xylan and mannan from softwood sulfite dissolving pulps (Gubitz *et al.* 1997). Endoglucanase activity might lead to a swelling of the fiber wall, leading to increased exposure to solvents and reagents used during dissolving pulp derivatization (Henriksson *et al.* 2005; Engstrom *et al.* 2006). Xylanase treatment can lower the pentosans level in dissolving pulp, and thus increase the reactivity of the dissolving pulp (Christov and Prior 1993, 1995; Christov *et al.* 1999). Gehmayr *et al.* (2011) studied the feasibility of enzyme treatments of *Eucalyptus globules* dissolving pulp for viscose application and showed that the xylanase pretreatment can increase reactivity of the dissolving pulp towards xanthation.

The specific objective of this research was to evaluate the potential of slab residuals from poplar wood for the production of high-grade dissolving pulps. Some selected properties, such as kappa number, pentosan content, α -cellulose content, and degree of polymerization (DP) were investigated to evaluate every step in the prehydrolysis kraft and totally-chlorine free (TCF) bleaching process. In addition, the effect of xylanase post-treatment on the pentosan content and reactivity of the dissolving pulps made from residual slabs from poplar is also discussed in this work. It is expected that this study can provide some fundamental knowledge that will further assist the exploration of new materials for the production of high-grade dissolving pulps.

EXPERIMENTAL

Materials

Residual slabs of poplar wood were provided by a pulp mill in Shandong Province. Chipped residual slabs were screened to obtain chips with a size of $20 \times 20 \times 2$ mm. The chips were air-dried and homogenized in a single lot to avoid differences in overall composition. Holocellulose content in the chips was measured using the acidified sodium chlorite method (Leopold 1961). The α -cellulose was then separated from the holocellulose using 17.5% sodium hydroxide solution. The xylanase (AU-PE89), which had an activity of 80,000 IU/g, was provided by Sukahan Biochemical industry Co. Ltd.

Analytical Methods

Standard Methods of the Technical Association of the Pulp and Paper Industry (TAPPI, Atlanta, GA) were used to determine the chemical composition of the samples.

Acid-insoluble lignin (T222om-06), pentosans (T223cm-01), kappa number (T236-om-99), α -cellulose (T203cm-99), and ash (T211om-12) were determined. The brightness of the pulp was also determined according to TAPPI Standards (T525om-12). The average degree of polymerization (DP) of pulp was determined and calculated by the formula ($DP^{0.905} = 0.75[\eta]$) from the intrinsic viscosity $[\eta]$, which was measured by dissolving the pulp into a cupriethylenediamine solution according to Chinese National Standard FZ/T 50010.3-2011. X-ray diffraction (XRD) patterns of the pulp were recorded with an X'Pert diffraction instrument (D8-ADVANCE; Bruker Corp.) using Cu radiation ($\lambda = 0.15406$ nm). Data were collected in the 5 to 50° range of 2θ with a 0.05° step every 1.5 s. The crystallinity index of the pulp was calculated using the method introduced by Segal *et al.* (1959).

Reactivity Measurements

The reactivity values of the dissolving pulps made from poplar residual slabs were determined *via* a two-step process, according to the Chinese National Standard FZ/T 50010.13-2011, which was based on the work by Wu *et al.* (2014). The only deviation from the standard was that the constant volume addition of CS₂ was 8 mL instead of 11 mL during the viscose preparation process. The less time that was taken, the greater was the reactivity of the dissolving pulp.

RESULTS AND DISCUSSION

The average chemical compositions of the poplar slab residuals were determined as follows: pentosan, 24.1%; holocellulose, 82.3%; α -cellulose, 43.5%; acid-insoluble lignin content, 16.7%; and ash, 0.7%. The analytical results indicated that the chemical compositions of poplar slab residuals are similar to that of poplar wood (Liu *et al.* 1995).

Prehydrolysis

The water prehydrolysis was carried out in an electrically heated stainless steel digester (15 L) that rotated at a rate of 1 rpm. After the prehydrolysis stage, the hydrolyzed slab chips were thoroughly washed with hot water; afterward the hydrolyzed chips were air-dried and the mass loss was determined gravimetrically.

The prehydrolysis was carried out at a constant cooking temperature of 160 °C, which was heated from room temperature at a heating rate of 8 °C/10 min; the time at cooking temperature was of 120 min (Zhou *et al.* 2012). To determine the optimum liquor-to-wood ratio for the maximum removal of hemicelluloses during prehydrolysis, the ratio was varied from 5:1 to 8:1 (Table 1).

Increasing the prehydrolysis liquor-to-wood ratio from 5:1 to 6:1 caused the final yield to decrease from 79.8% to 78.8% (Table 1). However, the pentosan content in the hydrolyzed chips decreased from 15.3% to 13.5%. As the liquor-to-wood ratio increased from 6:1 to 8:1, the final yield was nearly invariant while the pentosans increased slightly. Typically, the pentosan content was the most important criterion in determining the prehydrolysis process. Ultimately, a liquor-to-wood ratio of 6:1 was selected as a suitable condition for the water prehydrolysis process.

Table 1. Effect of Different Liquor-to-Wood Ratios Used during Chip Prehydrolysis

Liquor ratio	Pentosan content (%)	Final yield (%)
5:1	15.3	79.8
6:1	13.5	78.8
7:1	13.9	77.6
8:1	14.9	78.0

Under mild water prehydrolysis conditions, the pentosans in the treated chips were still more than 10%, which is relatively high for making dissolving pulp. To further decrease the pentosans in the hydrolyzed chips, sulfuric acid was added in a mild water prehydrolysis process (acid prehydrolysis). The conditions of acid prehydrolysis were: cooking temperature of 160 °C; liquor-to-wood ratio of 6:1; sulfuric acid dosage range of 0.5% to 2.0% on wood; heating rate of 8 °C/10 min from room temperature; and a cooking time of 120 min at cooking temperature.

As the H₂SO₄ dosage was increased from 0.5% to 2.0%, the pentosans in the acid-hydrolyzed chips decreased, as well as the final yield (Table 2). Consideration of the final yield and relatively high hemicellulose removal action, a sulfuric acid dosage of 1.0% was selected as a suitable condition for the acid prehydrolysis process.

Table 2. Effect of Different H₂SO₄ Dosages during Acid Prehydrolysis on Chip Properties

H ₂ SO ₄ dosage (%)	Pentosan content (%)	Final yield (%)
0.5	11.9	74.2
1.0	7.9	72.8
1.5	6.0	71.3
2.0	5.0	71.6

Kraft Pulping

The acid-prehydrolyzed chips from the slab residuals of poplar were subjected to kraft pulping under the following conditions: a cooking temperature of 165 °C; a liquor-to-wood ratio of 4:1; a sulfidity of 5%; a heating rate of 6 °C/10 min to cooking temperature; a cooking time of 120 min; and for various active alkali (as NaOH) charges of 18%, 21%, 24%, and 27% (based on oven-dry prehydrolyzed chips). Kraft cooks were performed in the same digester that was used in the prehydrolysis stage. The pulps were washed thoroughly with tap water and screened by laboratorial screener equipped with 0.2 mm slotted plate to remove the rejects. The screened yields of the kraft pulps were determined gravimetrically.

Table 3. Effects of Alkali Dosage on Kraft Pulp Properties

Alkali dosage (%)	Pentosan content (%)	Kappa number	Degree of polymerization (DP)	Brightness (%ISO)	Final screened yield (%)
18	4.5	22.8	812	27.7	31.5
21	4.3	17.9	822	37.6	27.7
24	4.2	13.6	937	36.5	27.2
27	4.2	13.5	1018	34.7	26.8

The effects of alkali dosage used in the kraft pulping process on the characteristics of the screened pulp are shown in Table 3. It can be seen that the kappa number decreased from 22.8 to 13.6 as the alkali dosage was increased from 18% to 24% while the other cooking parameters remained constant. However, the kappa number remained constant as the alkali dosage was increased further from 24% to 27%. In addition, the pentosans in the cooked pulp decreased very slightly as the alkali dosage increased. The final screened pulp yield decreased when the alkali dose increased. The degree of polymerization went down from 1018 to 812 as the alkali dosage went from 18% to 27%. The brightness of pulp was the lowest at the alkali dosage of 18% was used; this was due to the lower delignification (higher kappa number) at 18% alkali charge when compared to the other kraft cooks at higher alkali charge. Therefore, a 24% alkali charge was selected as optimum for kraft pulping for the production of a bleachable pulp; this brownstock had a lower kappa number with similar final screened yields and pentosans when compared to the other pulps made at lower alkali charges.

Oxygen Delignification Enhanced with Peroxide ((OP)-Stage)

The prehydrolyzed kraft pulps were subjected to oxygen delignification enhanced with peroxide ((OP)-stage). Optimization of the (OP), was carried out at: oxygen pressure of 0.6 MPa; reaction temperature of 105 °C; 10% consistency; 1.0% H₂O₂ on pulp; 3.0% NaOH on pulp (Bing *et al.* 2014); reaction time of 60 min; and MgSO₄ charge of 0.2%, 0.4%, 0.6%, or 0.8% on pulp. At the end of the (OP) treatment, the pulps were fully washed with tap water.

The effects of MgSO₄ dosage in the (OP)-stage on the characteristics of the pulp are shown in Table 4. It is clear that additional delignification was obtained with the (OP) process with or without the addition of MgSO₄. In addition, the brightness, pentosans and yield of the (OP)-delignified pulp slightly changed when the MgSO₄ dosage was increased from 0% to 0.8%. However, the DP increased as the MgSO₄ dosage increased. This indicated that the cellulose degradation was controlled by the addition of MgSO₄ in the (OP) process (Gilbert *et al.* 1973). Therefore, a 0.6% MgSO₄ dosage was selected as the optimum level since this (OP) pulp had the highest DP and lowest kappa number with similar pulp yield and brightness to the other (OP) pulps.

Table 4. Effect of MgSO₄ Dosage on (OP)-Stage Delignification

MgSO ₄ dosage (%)	Pentosans content (%)	Kappa Number	Degree of polymerization (DP)	Brightness (%ISO)	Final yield (%)
0	4.5	2.5	500	64.5	25.3
0.2	4.3	2.6	499	63.4	25.5
0.4	4.2	2.5	518	63.8	25.2
0.6	4.0	2.2	533	64.1	25.1
0.8	4.4	2.5	538	63.9	25.1

QP Bleaching

A TCF bleaching system was implemented using a QP brightening sequence. The chelation (Q) stage was done at the following conditions: 0.2% ethylenediaminetetraacetic acid (EDTA); temperature of 90 °C; 10% consistency; and 60 min reaction time. Peroxide bleaching (P) was carried out using: 0.5% NaOH; 0.5% MgSO₄; 0.2% diethylenetriaminepentaacetic acid (DTPA); and 2.0% H₂O₂; 10% consistency; and 95 °C reaction temperature for 120 min (Bing *et al.* 2014).

Table 5. Result of Chelation (Q) and Peroxide (P) Bleaching of (OP) Pulps

	Q-treated pulp	P-bleached pulp
Final yield (%)	24.6	23.5
Pentosans content (%)	3.9	3.7
α -cellulose content (%)	-	95.1
Kappa number	2.5	1.6
Ash (%)	-	0.08
Reactivity (s)	-	7.7
Brightness (%ISO)	70.2	86.4
Degree of polymerization (DP)	520	489

A TCF QP sequence was used for bleaching the dissolving pulps. The brightness gains of the pulp were substantial during the (P) stage, during which most of the lignin was removed (Table 5). The DP decreased from 533 to 489 during the QP bleaching sequence. As a result, the bleached dissolving pulp had a pentosans content of 3.7%, an α -cellulose content of 95.1%, a DP of 489, a brightness of 86.4% ISO, and a reactivity of 7.7 s.

Effect of Xylanase Post-Treatment on Reactivity of the Dissolving Pulp

Xylanase post-treatment of the dissolving pulp made from residual slabs of poplar was carried out under the following conditions: temperature of 50 °C; 60 min reaction time; 10% consistency; and reaction pH of 5 (using a sodium acetate buffer). The effects of different xylanase dosages (IU/g on oven-dried pulp) on the reactivity, pentosans, and α -cellulose of the dissolving pulp are shown in Table 6.

It was apparent that the reactivity of the dissolving pulp drastically decreased when the xylanase dosage was more than 0.5 IU/g (Table 6). However, the pentosans in the dissolving pulp slightly decreased with increase xylanase dosages while the α -cellulose content remained invariant. Therefore, a xylanase dosage of 1.0 IU/g on the enzyme-treated dissolving pulp was selected to compare to the original dissolving pulp in order to investigate further the reason why the reactivity did not improve after xylanase post-treatment.

Table 6. Changes in Composition and Reactivity of the Dissolving Pulp

Xylanase dosages (IU/g)	Pentosans content (%)	α -cellulose content (%)	Degree of polymerization (DP)	Reactivity (s)
0	3.7	95.1	489	7.7
0.5	3.4	95.2	492	15.3
1.0	3.2	95.5	495	120.8
1.5	3.2	95.4	490	130.4
2.0	3.1	95.2	493	200.5
2.5	3.1	95.1	491	248.6

Effect of Xylanase Post-Treatment on the Morphology and Crystallinity Index of the Pulp Fibers

SEM images of the original dissolving pulp and the xylanase (1.0 IU/g) post-treated pulp are shown in Fig. 1. The images revealed that after xylanase post-treatment, the fiber surface structure became loose and appeared to have cracks and gaps due to hemicellulose dissolution, which created a more porous structure to allow for rapid chemical penetration. This leads to the rapid formation of viscose at outer layer of the

cellulose, which will possibly hamper the further chemical penetration. Additionally, the xylanase post-treatment resulted in serious damage to the crystallinity of the cellulose in the fiber wall. The crystallinity index of the dissolving pulp was drastically lowered from 69.2% to 59.1% (Fig. 2). The crystal form of cellulose I (*i.e.*, 2θ values between 22° and 23°) did not change after the xylanase post-treatment. These results showed that the xylanase post-treatment affected the morphology and crystallinity index of the resulting dissolving pulp. The rapid drop in the crystallinity index after xylanase post-treatment, which will greatly enhance the chemical penetration in viscose preparation process, was probably a key factor for causing the lower reactivity of the resulting pulp.

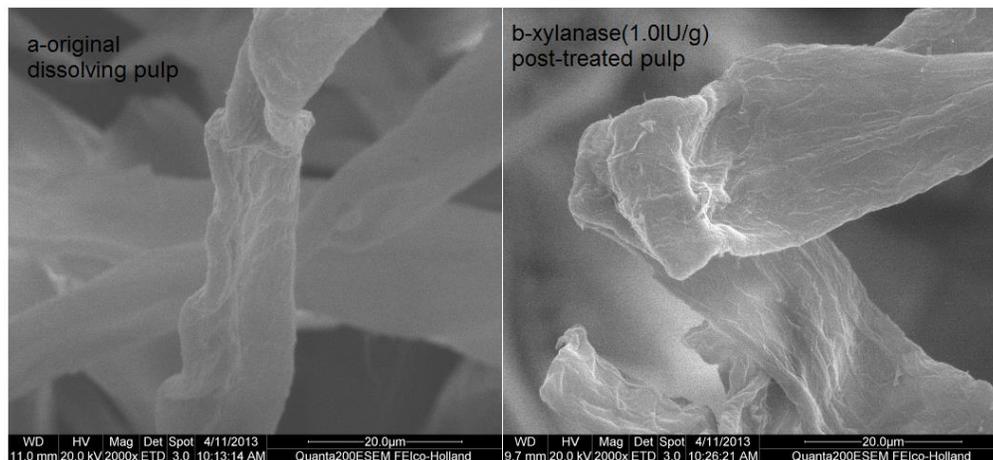


Fig. 1. SEM images of the dissolving pulp

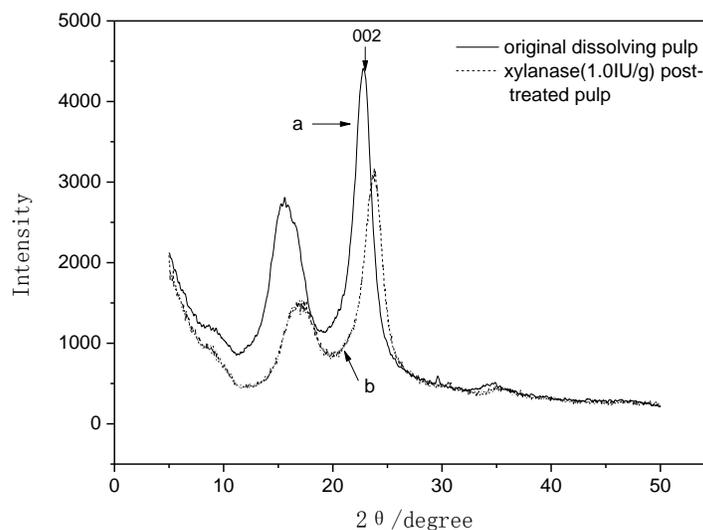


Fig. 2. X-ray diffractograms of the dissolving pulp

CONCLUSIONS

1. The dissolving pulp made from slab residuals of poplar had a pentosans content of 3.7%, an α -cellulose content of 95.1%, a degree of polymerization of 489, a

brightness of 86.4% ISO, and a reactivity of 7.7 s. This dissolving pulp was produced using an acid prehydrolysis kraft process and an (OP) QP bleach sequence.

2. The reactivity of the dissolving pulp was found to be drastically decreased after the xylanase post-treatment. The xylanase-treated dissolving pulp had a slightly lower pentosans level. Simultaneously, the crystallinity index of this pulp obviously decreased when compared to the original dissolving pulp (*i.e.*, untreated).

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