

## EFFECT OF SCREW EXTRUSION PRETREATMENT ON PULPS FROM CHEMICAL PULPING

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The effect of compressive pretreatment before chemical pulping on the properties of poplar kraft and soda-AQ pulp was evaluated. Compressive pretreatment not only resulted in the dissolution of hemicellulose, but also leached extractives. Pulps made from compressive pretreated wood chips required lower beating energy than the untreated pulps to achieve the same beating degree of 45°SR, and the brightness of the handsheets was improved by 2% ISO. Compressive pretreatment allowed for efficient delignification and saved about 6% alkali consumption to achieve similar pulp screen yield. Furthermore, a higher content of fines and slightly lower mechanical properties were observed after the compressive treatment.

*Keywords:* Pretreatment; Poplar; Alkali consumption; Screen yield

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### INTRODUCTION

Plant biomass exhibits recalcitrance to mechanical, chemical, and enzymatic treatments due to its complex heterogeneous structure (Cosgrove 2005; Iiyama *et al.* 1994; Himmel *et al.* 1999; Wyman *et al.* 2005). Pulping is a conventional biomass conversion process to separate fibers by means of mechanical action, chemical action, or a combination of them. To reduce the recalcitrance of biomass, different pretreatment methods have been developed, such as chemical pretreatments, enzymatic pretreatments, and mechanical pretreatments (Chang *et al.* 1998; Esteghlalian *et al.* 1997; Kim and Lee 1996). All of them accomplish the reduction of recalcitrance by weakening bonding, depolymerizing, and solubilizing components of biomass (Grous *et al.* 1986; Mosier *et al.* 2005). Screw extrusion is a short-time pretreatment technology for production of conventional mechanical pulp. During the screw extrusion process, raw materials are treated under short residence time, high pressure, and strong shear forces. Extrusion of chips could promote the penetration of chemicals into wood, and it has been applied successively in the production of alkali peroxide mechanical pulp (APMP) and chem-thermol mechanical pulp (CTMP) (Xu *et al.* 1999; Yuan *et al.* 2006). However, there has been no report about its application in chemical pulping.

In order to solve problems related to the shortage of wood materials for pulp and paper industry in China, fast-growing poplar has been introduced, and it has become an important raw material for the pulp and paper industry in recent years (Tang *et al.* 2006). Fast-growing poplar grows fast for the first 5 to 7 years, and its harvesting cycle is short.

It has many advantages compared to other hardwood, such as fast growth, better adaptability, good quality, and high economic efficiency.

Poplar wood chips can be permeated easily by pulping chemicals due to its low basic density (Pang *et al.* 2004). However, poplar chemical pulp is characterized by a high consumption of active alkali and high concentration of residual alkali. The objective of this paper was to find a feasible method to reduce the chemical charge for pulping, thus enabling a reduction in the environmental impact. Extrusion treatment was selected as a processing technology in poplar chemical pulping, since it requires no chemical input and thus can be considered as being environmentally friendly.

## EXPERIMENTAL

### Material

Freshly cut 7-year-old poplar with 16.8 m height was obtained from vocational technology college of Shandong Agricultural University and harvested in Juxian, Shandong province, China. Its basic properties were as follows: density of 0.405 g/cm<sup>3</sup>, diameter of 18.6 cm, and wood powder brightness of 62.5% ISO. The tree was debarked and chipped with a laboratory chipper. The wood chips were screened, and the accepts (size 25 mm×20 mm×3~5 mm) were collected. Finally, the screened chips were hand-sorted to remove all pieces of knots, barks, and decayed wood. The chips were then air dried and stored in a sealed polythene bag for pulping. The other chemicals were all analytical grade without further purification. The content of water extractives, ethanol-benzene extractives, holocellulose, klason lignin, acid soluble lignin, and pentose were tested according to GB/T2677.4-1993, GB/T2677.6-1994, GB/T2677.10-1995, GB/T747.6-1989, GB/T10337-1989, and GB/T2677.9-1994, respectively. The chemical compositions of poplar were expressed by weight percentage of the total dry weight and results are shown in Table 1.

**Table 1.** Main Chemical Components of Poplar (%)

Extractives	Cold water	2.15
	Hot water	3.27
	1%NaOH	21.07
	Ethanol-benzene	2.69
Holocellulose		79.52
Klason lignin		21.59
Acid soluble lignin		2.59
Pentose		24.92

### Methods

#### Chip compression

Wood chips were compressed at room temperature in a JS10 screw extrusion machine (made by Wenrui Machinery Co., Ltd.), with a compression ratio of 4:1. The chips were soaked in cold water for 48 hours, and then impregnated in water steam at 97°C for 20 minutes before compression.

Dissolution Ration (DR) in this work were calculated according to the formula,

$$\text{DR (\%)} = (C_{\text{uncom}} - Y \times C_{\text{com}}) / C_{\text{uncom}} \times 100 \quad (1)$$

where  $C_{\text{uncom}}$  is a component content before compression,  $C_{\text{com}}$  is the component content of chips after compression, and  $Y$  is yield of wood chips after compression.

#### *Chemical pulping*

Wood chips were cooked in a 15-L batch cylindrical rotatory digester. The operating conditions for kraft pulping were: active alkali charge was variable; sulfidity, 20%; and liquor/wood ratio, 5/1. The operating conditions for Soda-AQ pulping were: active alkali charge was variable; AQ, 0.05%; and liquor/wood ratio, 4/1. The curves of raising temperature for the two pulping methods were similar: temperature, 165°C; time to 165°C, 90 min; and reaction time, 120 min. The chips were discharged after cooking. The cooked fibers were soaked overnight and then washed under running water to remove the residual liquor. After washing, the materials were disintegrated and screened with 0.30 mm slotted plate.

#### *Pulp fractionation*

The pulps were fractionated using a Bauer-McNett classifier. The long fiber retained on 28-mesh screens were coded as R28, passed 28-mesh screen but retained on 50-mesh screen coded as P28/R50, and so on.

#### *Handsheet making and physical testing*

The pulp samples were beaten to  $45 \pm 1^\circ\text{SR}$  in a PFI mill and handsheets were made for determining various strength properties. The sheets were then conditioned at  $23 \pm 1^\circ\text{C}$  and  $50 \pm 1\%$  relative humidity overnight. Kappa number, screen yield, beating degree, folding endurance, tear index, and brightness were performed in accordance with relevant standards.

## RESULTS AND DISCUSSION

### **Effect of Compressive Pretreatment on Chemical Components of Poplar**

As shown in Table 1, holocellulose, lignin, and pentose contents of raw material were 79.52, 24.18, and 24.92%, respectively. The extractives contents of poplar were 2.15, 3.27, 21.07, and 2.69%, corresponding to cold water, hot water, 1% NaOH, and alcohol-benzene, respectively. Wood chips are prone to split, and bulk density decreases after extrusion, which influences not only the physical properties, but also leads to extensive chemical component changes. Effects of pretreatment on the chemical components of poplar are presented in Table 2. Pretreatment of wood chips made a major contribution to leaching of extractives, while the content of lignin changed little due to its high molecular weight and network structure. In addition, some hemicellulose oligomers dissolved, which was demonstrated by the decrease of pentose content. Extractives, especially those removed by 1% NaOH, had a key influence on alkali consumption of chemical pulping. It could be presumed that compression of wood chips would reduce alkali consumption of chemical pulping.

**Table 2.** Effect of Compressive Pretreatment on the Chemical Components of Poplar (%)

Component	Content
Dissolution ration of ethanol-benzene extractive	6.45
Dissolution ration of 1% NaOH extractive	7.42
Dissolution ration of lignin content	0
Dissolution ration of pentose content	3.74

### Effect of Compressive Pretreatment on Fiber Morphology of Poplar

Wood chips are subjected to a complex set of forces involving compression, shearing, stretching, and twisting in the course of treatment in a screw extrusion machine. As a result, the cell wall can be delaminated. Compression ratio had an important influence on these changes. In order to get detailed information about whether the compression treatment condition used could affect fiber morphology, the pulps were separated into five fractions using a set of screens ranging from 28- to 203-mesh size. Generally speaking, the fractions above 203-mesh could be considered as fibers, the fraction above 50-mesh as long fibers, and those below 203-mesh as fines.

**Table 3.** Fractional Screen Analysis Results of Chips with Different Treatment methods (%)

Treatment manner	Untreated	Compressed
R28	9.84	12.80
P28/R50	43.78	41.38
P50/R102	25.99	26.31
P102/R203	9.19	5.94
P203	11.20	13.57

As shown in Table 3, the content of long fibers that was retained on the 50-mesh screen was 54.18% after compression, which was almost equal to that of untreated chips. However, the content of fines that passed 203-mesh screens was slightly increased from 11.23% to 13.57%. This indicated that forces generated during extrusion of poplar wood chip with compression ratio of 4:1 could not result in severe fiber damage.

### Effect of Compressive Pretreatment on Properties of Chemical Pulping

Kraft and soda-AQ pulping methods are widely accepted as the dominant chemical pulping processes. Kraft pulp exhibits superiority in pulp quality, high efficiency in chemical recovery, and suitability for most types and species of feedstock. As for soda-AQ pulping, it causes odor pollution, and is only suitable for loose fibrous materials. The effect of compression on properties of kraft pulp and soda-AQ pulp was therefore investigated. As shown in Table 4, the optimum active alkali charge for conventional kraft pulping and soda-AQ pulping was 18 to 20%. The wood chips could obtain a relatively high screen yield at this condition, and kappa number could meet the requirements of manufacture.

**Table 4.** Effect of Compression on Chemical Pulp Properties

		Active alkali (%)	Screened yield (%)	Reject (%)	Kappa number
Kraft	Untreated	16	50.99	4.86	27.36
		18	52.34	1.23	21.65
		20	51.88	0.74	19.74
		22	49.16	0.46	16.31
	Compressed	12	51.36	1.85	24.68
		14	51.52	0.43	18.77
Soda-AQ	Untreated	16	55.78	3.68	30.09
		18	54.08	1.82	23.61
		20	53.80	1.42	21.78
		22	51.84	1.03	18.22
	Compressed	12	56.32	3.13	29.74
		14	54.63	0.52	21.07

The kappa number of pulp gives information regarding the performance of cooking, residual lignin content in pulp, and the delignification efficiency degree of pulp. As shown in Table 4, the kappa number of the pulp was influenced by compression of wood chips. In order to achieve similar screen yield, kappa number of kraft pulp from compressed wood chip was lower than that of untreated pulp, which implied that compression could lead to an increase of delignification efficiency. For example, the kappa number of compressed pulp was 18.77 when the screen yield was about 51.5%. However, the kappa number of untreated pulp reached 19.74. It should be stressed that the dosage of alkali of compressed wood chip was only 14% to reach similar screened yield, which was lower than 20% in the case of untreated wood chip. Moreover, compression of wood chips also reduced the rejects rate of wood pulp. That's important for conserving wood resources and improving the utilization of wood material. The trend for soda-AQ pulp was similar.

The results of the mechanical and optical tests performed with handsheets of different pulps were presented in Table 5. Beating can improve bonding between fibers and thus enhance strength properties. Similar pulp beating degrees for untreated and compressed pulp were achieved by applying different beating revolutions. It was found that, irrespective of whether the chips were subjected to kraft pulping or sodium-AQ pulping, the required beating revolutions to a target beating degree of 45°SR of untreated pulp were much higher than that of compressed pulp. The beating revolutions for kraft pulp could decrease from 19000 to 17000 as a consequence of pretreatment, and that of soda-AQ pulp could also be decreased from 24500 to 20000. Beating ability of pulp is very important for energy saving of mills and generally depends on the chemical composition of pulp. The striking difference in required beating revolutions between the untreated pulp and compressed pulp with regards to the consumed energy was most probably due to the fact that pretreatment changed the structure of wood chips. Thus, more cellulose was exposed because of the degradation of hemicelluloses, and the exposed cellulose participated directly in the swelling process.

**Table 5.** Effect of Compression on Properties of Handsheets Made from Chemical Pulp

		Active alkali (calculated by Na <sub>2</sub> O, %)	Beating revolutions	Beating degree (°SR)	Brightness (%ISO)	Folding endurance (times)	Tear index (mN·m <sup>2</sup> /g)
Kraft	Untreated	16	19000	45.2	25.3	1324	9.46
		18		45.3	27.9	1245	9.28
		20		45.1	28.2	1256	9.12
		22		44.8	29.3	1056	8.94
	Compressed	12	17000	45.4	27.6	905	8.79
		14		45.1	30.8	852	8.52
Soda-AQ	Untreated	16	24500	45.8	23.6	1256	9.68
		18		45.3	27.7	1224	9.57
		20		44.9	28.7	1189	9.39
		22		44.8	29.6	892	8.62
	Compressed	12	20000	45.2	27.5	923	8.74
		14		45.1	30.7	868	8.68

On the other hand, the compressive treatment had a slightly negative effect on mechanical properties, which could be exhibited by decreases in folding endurance and tear index. The results of Table 5 showed that the handsheets of the compressed pulp had slightly lower folding endurance and tear index than those of the corresponding untreated pulp. The decrease of hemicelluloses was identified as one of the main reason for the loss of mechanical strength of wood fiber.

The brightness of handsheets obtained from compressed pulps was found to be higher than that of untreated pulp. The brightness of handsheets obtained from compressed kraft pulp and soda-AQ pulp at 14% alkali charge pulp could reach as high as 30.8% ISO and 30.7% ISO, respectively, which were higher than that of untreated pulp cooked at all levels of alkali charge. The higher the brightness of pulp, the lower the value of kappa number would be. The results in Table 5 were in accordance with the results in Table 4, which further confirmed the conclusion that compression treatment of chips could reduce the kappa number of pulp.

## CONCLUSIONS

The effect of compressive pretreatment before chemical pulping on the properties of poplar kraft and soda-AQ pulp were evaluated, and the following conclusions could be made by the investigation:

1. The compressive pretreatment of poplar wood chips before chemical pulping has the potential of being more widely applied in chemical pulping processing.

2. The pretreatment could result in the dissolution of hemicelluloses and leaching of extractives. Energy consumption and alkali consumption were reduced by pretreatment, and the brightness of the treated pulp was improved.
3. The fiber length of poplar was not influenced, and mechanical properties were slightly decreased.

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