Effect of Boric Acid Addition on the Prehydrolysis of *Whangee*

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Prehydrolysis is an important step in the kraft-based dissolving process for pulp production, as it helps remove as much hemicellulose as possible from cellulose fibers before the material is subjected to the main delignification operation, *i.e.*, pulping. In this paper, a novel process concept was proposed by adding different dosages of boric acid (BA) based on the oven dry weight of *Whangee*, a genus of bamboo, in the prehydrolysis stage. The final yields of the prehydrolysis stage obviously increased and ferric ion contents in the hydrolyzed *Whangee* largely decreased with the addition of BA. Additionally, the highest α-cellulose retention occurred at a BA dosage of 0.5%. The results of acetic acid percent in the total sugars and furfural percent in xylose of the PHL showed that the addition of BA had an important impact on the structure of hemicelluloses in *Whangee*. Mass balance analysis of the PHL and *Whangee* indicated that the partly acid-insoluble lignin in *Whangee* was likely converted into acid-soluble lignin in the PHL.

Keywords: Boric acid; Prehydrolysis liquor; Whangee; Dissolving pulp

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INTRODUCTION

The prehydrolysis of wood prior to kraft cooking can be a significant part of a biorefinery process. Prehydrolysis using water or mineral acid can solubilize the majority of hemicellulose, resulting in prehydrolysis liquor (PHL) containing large quantities of hemicellulose-derived saccharides, *e.g.*, xylose and its oligomers. A part of lignin is also dissolved in the PHL (Wang *et al.* 2015a). The high quality dissolving pulp contains a high α-cellulose content (95% to 98%), but a relatively low hemicellulose (1% to 10%) and lignin content (< 0.05%) (Susi *et al.* 2015). Hence, prehydrolysis (high hemicellulose removal) is an important stage for the production of high quality dissolving pulp.

However, with increasing demand for pulpwood and a shortage of oil resources, numerous countries have attached great importance to the use of non-wood raw material for the production of high quality dissolving pulp, especially bamboo resources. Bamboo, which is an abundant and renewable resource in China, is considered a sustainable fiber resource to produce dissolving pulp (Ma *et al.* 2011; Susi *et al.* 2015). In general, the α-cellulose content in bamboo is 40% to 50%, which is comparable with the reported α-cellulose contents of softwoods (40% to 52%) and hardwoods (38% to 56%). Furthermore, ideal bamboo-dissolving pulp with high brightness (92.4% ISO) and α-cellulose content (94.9%) has been obtained (Batalha *et al.* 2012). Presently, a number of methods have been proposed for extracting hemicellulose in the prehydrolysis stage, including auto-hydrolysis (Leppänen *et al.* 2011), dilute acid (Jeong *et al.* 2010), mild alkali extraction (Huang and Ragauskas 2013), organic acid (Li *et al.* 2010; Scordia *et al.* 2011), and solid acid pretreatment (Dhepe and Sahu 2010). Among those methods, dilute acid extraction usually resulted in a lower pulp yield and physical properties, which were attributed to the acidity in the pretreatment medium (Aldajani *et al*. 2009). According to Huang and Ragauskas (2013), the addition of boric acid (BA) in alkali solution can extract more hemicellulose from softwood and have little influence on cellulose.

To date, no study has been carried out on the addition of BA in the prehydrolysis stage for producing dissolving pulp. In this work, a novel process concept was proposed by adding different dosages (based on the oven dry weight of *Whangee*) of BA in the prehydrolysis stage. The chemical compositions of hydrolyzed *Whangee* under various pretreatment conditions were compared, and some characteristics of the corresponding PHL were also determined. It is expected that this study can provide some fundamental knowledge for the exploration of prehydrolysis process of *Whangee*.

EXPERIMENTAL

Materials and Chemical Composition of *Whangee*

The raw material of *Whangee* (a genus of bamboo) was provided by one of the forestry centers in Sichuan province, China. Chipped *Whangee* was screened to obtain chips with a size of 11 × 25 × 6 mm. A portion of *Whangee* chip was taken for moisture determination for subsequent experiment. A portion of *Whangee* chip was ground into powder, and particles of 40 to 60 mesh powder were kept for chemical analysis. 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 content of ash, α-cellulose, pentosan, acid insoluble lignin, acid-soluble lignin, solvent extractives, and ferric ion were measured according to TAPPI T211om-12 (2012), TAPPI T203cm-99 (1999), TAPPI T223cm-01 (2001), TAPPI T222 om-11 (2011), TAPPI UM 250, TAPPI T204cm-07 (2007), and ISO 779 (2005), respectively. The BA was an analytically pure agent and was dissolved in distilled water of 10.0 g/L prior to being used.

Prehydrolysis Process

Prehydrolysis was carried out in an electrically heated stainless steel digester (1 L) with 0.07 kg oven dried *Whangee* chips at a constant cooking temperature of 165 °C, and it was heated from room temperature at a heating rate of 8 °C/ 5 min. The time at cooking temperature was 90 min, with a liquor-to-*Whangee* ratio of 8:1. Various dosages (based on the oven dry weight of *Whangee*) of BA were added (0%, 0.1%, 0.3%, 0.5%, and 0.7%) in the prehydrolysis stage. After the prehydrolysis stage, the hydrolyzed *Whangee* chips were directly air-dried without washing and the final yield was determined gravimetrically. The content of ash, α-cellulose, holocellulose, pentosans, acid insoluble lignin, acid-soluble lignin, alcohol-benzene extractives, and ferric ion were also measured according to the above Standards. The measurements of α-cellulose and holocellulose contents in hydrolyzed *Whangee* were not extracted with alcohol-benzene because alcohol-benzene can extract a large quantity of organic matrixes (Yan *et al.* 2010; Guo *et al.* 2013; Ma 2014). Theoretically, 0.56 L PHL can be obtained from prehydrolysis stage of 0.07 kg oven dried *Whangee* chips. In actuality, only 0.46 L PHL was separated and collected. Then, the collected PHL was filtered using two-tiers of slowly quantitative filter paper prior to subsequent analysis.

Dissolved Lignocelluloses Analysis

The sugars (oligomeric sugars and monomeric sugars) were measured using an indirect method based on quantitative acid hydrolysis of the liquid sample. To convert oligomeric sugars to monomeric sugars, the PHL was hydrolyzed with 4% (wt.) H_2SO_4 at 121 °C for 60 min in an oil bath (Shen *et al.* 2011). The hydrolyzed PHL was filtered (0.22 µm), and the precipitate was collected as acid insoluble lignin in the PHL. The supernatant was used for the determination of monomeric sugars, acid-soluble lignin, formic acid, acetic acid, and furfural (Chen 2014). The oligomeric sugar contents in the PHL were determined by considering the monomeric sugars and total sugar content of the samples before and after this additional acid hydrolysis stage. The monomeric sugars of the PHL were measured using high-performance anion-exchange chromatography coupled with a pulsed amperometric detector (HPAEC-PAD) and an HPAEC-PAD system (ICS-5000,Thermo Fisher, Sunnyvale, CA) equipped with a CarboPac PA20 analytical column. A guard column was used for MS determination (Wang *et al.* 2015b). The acid-soluble lignin content in the PHL was measured using a UV/Vis spectrometric method at a wavelength of 205 nm according to TAPPI UM 250 (Shen *et al.* 2011; Shi *et al.* 2011; Wang *et al*. 2014). Formic acid, acetic acid, and furfural were analyzed by an HPLC system equipped with a Waters C18 symmetry column (4.6 × 150 mm, 5 mm) and a UV/Vis detector at 210 nm (formic acid and acetic acid) and 275 nm (furfural) at 30 °C with 0.1% H_3PO_4 (v/v) as eluents (Wang *et al.* 2015b).

Equations

The total lignin removal was calculated as follows,

$$\text{Total lignin removal (\%)} = \frac{\textit{Total lignin}\text{ content }\textit{in raw material} - \textit{Total lignin}\text{ content }\textit{in hydrolyzed material} \times \textit{Final}\text{ yield}}{\textit{Total lignin}\text{ content }\textit{in raw material}} \times 100\% \tag{1}$$

Calculation of ash, pentosan, holocellulose and α- cellulose removals is the same as that of the total lignin removal (Zhou *et al*. 2016).

$$\text{Calculated pentosan content (g/L)} = \frac{\textit{Pentosan}\text{ content }\textit{in raw material} - \textit{Pentosan}\text{ content }\textit{in hydrolyzed material} \times \textit{Final}\text{ yield}}{\textit{Total volume of PHL collected }(L)} \times \text{Oven } \textit{dry weight of whangee }(g) \tag{2}$$

Calculations of calculated total lignin, acid-soluble lignin, and acid insoluble lignin contents in the PHL are the same as that of calculated pentosan.

$$\text{Calculated total solid content (g/L)} = \frac{(1 - \textit{Final}\text{ yield}) \times \text{Oven } \textit{dry weight of whangee }(g)}{\textit{Total volume of PHL collected }(L)} \tag{3}$$

RESULTS AND DISCUSSION

Chemical Composition of *Whangee*

The chemical composition of *Whangee* is listed in Table 1. Acid-insoluble lignin content in *Whangee* is higher than acid-soluble lignin content. Pentosan content in *Whangee* is lower than that of poplar wood (Liu *et al.* 1995). Moreover, the α-cellulose content in *Whangee* is similar with that of softwoods and hardwoods, hence, it is a potential raw material for dissolving pulp production.

Table 1. Chemical Composition of *Whangee*

Component	Amount (%)
Ash	1.08
Pentosan	19.78
Holocellulose	73.99
α-Cellulose	44.52
Ferric ion	0.005309
Total lignin	22.02
Acid-soluble lignin	1.94
Acid-insoluble lignin	20.08
Alcohol- benzene extraction	4.64

Effect of BA Dosage on Chemical Composition of Hydrolyzed *Whangee*

To investigate the effect of BA on the chemical composition of hydrolyzed *Whangee,* BA was added in the prehydrolysis stage of *Whangee*. The pre-hydrolysis process of *Whangee* was controlled with P-factor by computer, and the calculation of P-factor value was using the equation introduced by Zhang *et al*. (2011). The P-factor value of every prehydrolysis process was moderately changeable, since it is closely related with fluctuated prehydrolysis temperature and time.

Effects of the addition of various dosages of BA on chemical composition of hydrolyzed *Whangee* are shown in Table 2. The P-factor values of BA dosages of 0%, 0.1%, 0.3%, 0.5%, and 0.7% in the prehydrolysis process of *Whangee* were 2087, 1906, 1972, 2066, and 1924, respectively. It was obvious that higher P-factor value and boric acid concentration could result in the lower pentosan contents (respectively, 15.18% and 15.58%) in hydrolyzed *Whangee*. This showed that the boric acid concentration and P-factor value were direct related to the effect of the prehydrolysis of *Whangee*. However, with the addition of BA, the final yields of the prehydrolysis stage obviously increased. Lowest final yield and pentosan content in the hydrolyzed *Whangee* resulted in the highest pentosan removal at the BA dosage of 0% (Table 2 and Table 3). In addition, α-cellulose removal at the BA dosage of 0.5% was the lowest (Table 3). The lowest of α-cellulose removal meant the highest α-cellulose retention. These results indicated that the addition of BA in the prehydrolysis stage of *Whangee* could protect cellulose in the *Whangee*. Based on consideration of the final yield and relatively high hemicellulose removal action, a boric acid dosage of 0.5% was judged to be the optimum concentration. Although the total lignin content in hydrolyzed *Whangee* was the highest at the dosage of 0.5%, most lignin will be mainly removed in the subsequent cooking and bleaching stages of producing *Whangee* dissolving pulp. Generally, the ferric ion content in final wood dissolving pulp is not greater than 20 mg/kg (Zhou 2013). The results in Table 2 showed that ferric ion contents in the hydrolyzed *Whangee* were greatly decreased with the addition of BA. This indicated that addition of BA in the prehydrolysis stage of *Whangee* would have a beneficial impact on the production of *Whangee* dissolving pulp.

Table 2. Effect of BA Dosage on Chemical Composition of Hydrolyzed *Whangee*

BA dosage (%)	Ash (%)	Pentosan (%)	Holocellulose (%)	α-Cellulose (%)	Ferric ion (mg/kg)	Total lignin (%)	Acid-soluble lignin (%)	Acid-insoluble lignin (%)	Final yields (%)
0	0.38	15.18	62.66	53.17	283.77	22.77	1.33	21.44	71.13
0.1	0.28	16.26	62.62	51.80	202.68	21.45	1.14	20.31	73.30
0.3	0.36	15.78	62.64	52.22	173.26	21.96	1.13	20.83	72.32
0.5	0.34	15.82	62.30	53.77	70.19	23.73	1.19	22.54	72.34
0.7	0.40	15.58	62.68	52.32	97.95	23.27	1.22	22.05	72.61

Table 3. Effect of BA Dosage on Chemical Composition Removal of *Whangee*

BA Dosage (%)	Ash Removal (%)	Pentosan Removal (%)	Holocellulose Removal (%)	α-Cellulose Removal (%)	Total Lignin Removal (%)
0	74.97	45.41	39.76	15.05	26.45
0.1	81.00	39.74	37.96	14.71	28.60
0.3	75.89	42.30	38.77	15.17	27.88
0.5	77.23	42.14	39.09	12.63	22.04
0.7	73.11	42.81	38.49	14.67	23.27

Characteristics of PHL

There are various dissolved organic compounds in PHL due to the reactions in the prehydrolysis stage: i) depolymerization and dissolution of hemicellulose resulting in the formation of sugars; and ii) further degradation of sugars and lignin to form furan derivatives, organic acids, and phenolic compounds (Saeed *et al*. 2012; Ludwig *et al.* 2013). Separation and purification of hemicellulose can produce value-added products, *e.g.*, ethanol or xylitol production (Shen *et al.* 2013; Shi *et al.* 2012). Lignin can be utilized as a raw material for many value added products, *e.g.*, phenols and bio-fuel, and can generate additional revenues (Yang and Jahan 2013).

The effect of different BA dosages on chemical composition in the PHL of *Whangee* is shown in Table 4. It was obvious that oligosaccharide, which accounted for over 80% of the total sugars, was the main sugar in the PHL of *Whangee* with or without BA addition. Among the five kinds of sugar, xylose and glucose were the predominant sugars in the PHL. The sum of xylose and glucose percentages in the total sugars at BA dosages of 0%, 0.1%, 0.3%, 0.5%, and 0.7% were 90.69%, 90.73%, 91.41%, 91.13% and 91.14%, respectively. With the addition of BA, acetic acid percentages in the total sugars and furfural percent in xylose obviously increased, and they were the highest (11.43% and 5.28%) at the BA dosage of 0.5%. Acetic acid was mainly released during pre-hydrolysis from the labile acetyl groups present in the hemicelluloses, and furfural was mainly generated from further degradation of xylose (Saeed *et al.* 2012). These results indicated that the addition of BA had an important impact on the structure of hemicelluloses in *Whangee*. The highest values of acetic acid/total sugars in the PHL at BA dosage of 0.5% also showed higher hemicellulose removal with BA addition in the prehydrolysis stage of *Whangee*.

It was obvious that acid-soluble lignin was the major lignin in the PHL of *Whangee* with or without BA addition. Formic acid content in the PHL was all very low, which is toxic for the fermentation of hemicellulose-derived sugars to produce ethanol (Lee *et al.* 2013).

Table 4. Effect of BA Dosage on Chemical Composition in the PHL

BA Dosage (%)		0	0.1	0.3	0.5	0.7
Arabinose (A) (g/L)	Monomeric	0.74	0.62	0.7	0.73	0.74
	Oligomeric	0.31	0.41	0.27	0.30	0.27
Galactose (g/L)	Monomeric	0.22	0.16	0.20	0.21	0.21
	Oligomeric	0.54	0.55	0.50	0.52	0.50
Glucose (g/L)	Monomeric	2.12	1.70	2.02	2.08	1.99
	Oligomeric	9.96	9.67	9.58	9.62	9.38
Xylose (X) (g/L)	Monomeric	1.12	0.78	1.04	1.11	1.09
	Oligomeric	8.73	8.12	8.00	8.55	8.31
Mannose (g/L)	Monomeric	0.10	0.14	0.16	0.14	0.13
	Oligomeric	0.34	0.19	0.11	0.18	0.17
Total sugars (T) (monomeric+oligomeric) (g/L)		24.18	22.34	22.58	23.44	22.79
Acid insoluble lignin (g/L)		1.78	1.85	1.64	1.71	1.87
Acid-soluble lignin (g/L)		4.86	4.57	4.70	4.68	4.70
Total lignin (L) (g/L)		6.64	6.42	6.34	6.39	6.57
Formic acid (F) (g/L)		1.81	1.04	1.87	1.72	2.05
Acetic acid (C) (g/L)		2.20	2.22	2.12	2.68	2.41
Furfural (U) (g/L)		0.41	0.38	0.39	0.51	0.45
A+X (g/L)		10.90	9.93	10.01	10.69	10.41
T+L+F+C+U (g/L)		35.24	32.40	33.30	34.74	34.27
Acetic acid/Total sugars (%)		9.10	9.94	9.39	11.43	10.57
Glucose+Xylose/Total sugars (%)		90.69	90.73	91.41	91.13	91.14
Total oligomeric sugars/ Total sugars (%)		82.22	84.78	81.75	81.78	81.75
Furfural/ Xylose (monomeric+oligomeric) (%)		4.16	4.27	4.31	5.28	4.79
Acid-soluble lignin/Total lignin (%)		73.19	71.18	74.13	73.23	71.54

Mass Balance Analysis of the PHL and *Whangee*

The mass balance analysis was simply performed on the basis of calculated total solid, lignin, and pentosan contents in the PHL at the BA dosages of 0%, 0.1%, 0.3%, 0.5%, and 0.7%. The results in Table 5 show that calculated total solid contents were greater than the sum of measured total sugars, total lignin, formic acid, acetic acid, and furfural contents (Table 4) in the PHL. The calculated total solid also included inorganic ash in the PHL and filtered large size materials during the collection process of the PHL. Calculated total lignin and acid-insoluble lignin contents in the PHL were higher than the measured total lignin and acid-insoluble lignin contents in the PHL, while calculated acid-soluble lignin content was less than the measured acid-soluble lignin contents in the PHL (Table 4 and Table 5). These results indicated that a part of the acid-insoluble lignin in *Whangee* had likely been converted into acid-soluble lignin during the prehydrolysis process of *Whangee*. Arabinose and xylose are five-carbon sugars, and they are mainly originated from pentosan in *Whangee*. Notably, calculated pentosan content in the PHL (Table 5) was higher than the sum of measured arabinose and xylose content in the PHL (Table 4).

Table 5. Calculated Composition Contents in the PHL

BA Dosage (%)	Calculated Pentosan (g/L)	Calculated Acid-soluble Lignin (g/L)	Calculated Acid-insoluble Lignin (g/L)	Calculated Total Lignin (g/L)	Calculated Total Solid (g/L)
0	13.67	1.51	7.35	8.86	43.93
0.1	11.96	1.68	7.90	9.58	40.63
0.3	12.73	1.71	7.63	9.34	42.12
0.5	12.68	1.65	5.74	7.39	42.09
0.7	12.88	1.61	6.19	7.80	41.68

CONCLUSIONS

1. The addition of boric acid (BA) in the prehydrolysis stage of *Whangee* pulping could protect cellulose in the *Whangee*, resulting in the increase of the final yields compared with no BA addition.
2. Xylose and glucose were the predominant sugars in the PHL of *Whangee*. With the addition of BA, the percentages of acetic acid in the total sugars and furfural in xylose obviously increased. The addition of BA had an important impact on the structure of hemicelluloses in *Whangee*.
3. The calculated acid-soluble lignin content was less than the measured acid-soluble lignin contents in the PHL. This indicated that a part of the acid-insoluble lignin in *Whangee* had likely been converted into acid-soluble lignin during the prehydrolysis process.

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