Integration of hemicellulose pre-extraction in the bleach-grade pulp production process

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ABSTRACT: Loblolly pine woodchips were first pre-extracted with 6 wt% (percent on original nonextracted o.d. wood weight) sodium hydroxide at room temperature overnight, then treated at 90°C for 90 min, following another 4 h extraction with the addition of 5 wt% boric acid to partially remove hemicellulose. During the subsequent bleach-grade kraft pulping process, the cooking intensity was alleviated, either by decreasing the cooking time (reduced H-factor by 35%) or decreasing the chemical charge by 30%, with the objective of obtaining similar pulp quality as the control cook. After elemental chlorine free bleaching and PFI refining, the results indicated that the pre-extracted pulp could maintain equal or similar brightness and physical strength as the control pulp through this optimization of the pulping process.

Application: High-quality hemicellulose can be pre-extracted from loblolly pine woodchips using borate-assisted alkaline extraction before kraft pulping. With careful optimization of the subsequent pulping process for the extracted chips, the same pulp yield and pulp quality could be achieved as for nonextracted chips, and additional value-added chemicals could be generated from a kraft paper mill.

Increased demand for energy and concerns over greenhouse gas emissions have placed lignocellulosic biomass in the forefront of the search for a sustainable energy supply [1]. The conversion of lignocellulosic materials for producing fuel and chemicals (biorefinery) has received increasing attention during recent decades [2,3]. In the biorefinery concept, the three main chemical constituents in lignocellulosic materials (i.e., cellulose, hemicellulose, and lignin) are to be converted to the building blocks for biofuels, biochemicals, and biomaterials [4].

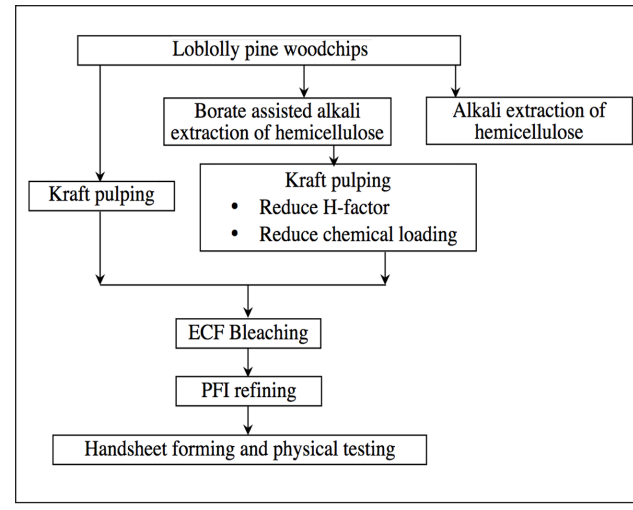
During kraft pulping, some of the hemicelluloses are degraded into low-molecular oligomers, monomers, and saccharinic acids, which end up in the black liquor. This cooking liquor is then concentrated and burned to reduce environmental impact, produce energy, and recover chemicals [5]. Thus, the hemicellulose, which is mainly made of sugars, is not fully utilized. Recently, van Heiningen proposed a concept of "value prior to pulping" (VPP) [6], in which the hemicelluloses are either partially or completely extracted before pulping to generate high-value products. The extracted hemicellulose can be used directly in polymeric form for novel industrial applications [7-9], or they can serve as a sugar source for the bio-ethanol production [10].

During the past decade, numerous studies have been conducted to introduce hemicelluloses extraction as a separate stage before the kraft cooking process. The common procedures for extracting hemicelluloses are hot-water extraction (autohydrolysis), near-neutral extraction, and dilute acid hydrolysis with small amounts of mineral acid (i.e., sulfuric acid or hydrochloric acid) [11-13]. Autohydrolysis was usually performed at temperatures above 150°C for 10-90 min, and the hemicellulose yields (based on total hemicellulose in the wood) were 20%-30% [14]. The near-neutral extraction applied small amounts of sodium hydroxide (NaOH) or green liquor to pretreat the woodchips at 140°C-160°C for 60-110 min. This process could remove 15%-30% of hemicelluloses before kraft pulping [12]. Compared with hot-water extraction, dilute acid extraction could remove more hemicellulose because of the acidity in the pretreatment medium [13]. Under the VPP concept, although high-value hemicelluloses can be pre-extracted before kraft pulping, the subsequent pulp yield and quality of the resulting kraft pulp could be degraded. To maintain the pulp yield and quality, the kraft pulping process needs to be carefully optimized for the hemicellulose pre-extracted biomass [15,16].

Our previous research applied 5 wt % boric acid together with 6 wt% NaOH at 90°C for 4 h to extract the hemicellulose from loblolly pine (*Pinus taeda*) woodchips [17]. The addition of borate in the alkali solution predisposes the extraction of glucomannans (a major component in softwood hemicelluloses) by complexing with the gluco and/or mannan units [18]. The introduction of borate in the extraction system is also beneficial for the caustic recovery process because it could eliminate or decrease the lime demand through borate autocausticizing [19]. A prior report by Hoddenbagh et al. indicated that charge of borates needed for autocausticization

was about 10% of the caustic needed in a year, which would have a favorable reduction in the variable costs of pulp production [20]. This borate-assisted, pre-extraction process could recover 35.5% of the total hemicellulose in the woodchips with only a 6.9% decrease in the cellulose degree of polymerization (DP). During the subsequent linerboard-grade kraft pulping process, the pre-extracted woodchips maintained similar pulp yield and strength as the control pulp by either decreasing the cooking time (reduced H-factor by 30%) or decreasing the chemical charge by 25%.

As far as we know, this is the first reported effort that shows the borate-assisted pre-extraction of hemicellulose from woodchips does not detrimentally affect the strength of pulp. In the present research, the effect of hemicellulose pre-extraction was extensively studied on bleach-grade kraft pulping and elemental chlorine free (ECF) bleaching. The objective of this study was to obtain equal or similar brightness and pulp quality as the control pulp through the optimization of the bleach-grade kraft pulping process on the hemicellulose-extracted woodchips. The pulping optimization process was mainly performed by modifying the H-factor and chemical loading (**Fig. 1**).



1. Optimization methodology of kraft pulping for the pre-extracted woodchips.

EXPERIMENTAL

Loblolly pine woodchips were obtained from a plantation near Macon, GA, USA. Upon receipt, the chips were screened to 2-8 mm in thickness and then stored at 4°C before use. The chips were analyzed for moisture content (46.5%) by drying samples in an oven at 105°C.

Borate-assisted alkaline extraction

During the alkaline extraction, loblolly pine woodchips were soaked overnight with 6% (w/w on the dried weight of woodchip) NaOH and treated at 90°C for 90 min. Boric acid (5 wt %w/w on the dried weight of woodchip) was then added to the medium to assist hemicellulose extraction for another 4 h. A 100 g (by dry weight) sample of woodchips was treated with a solid-to-liquid ratio of 1:8. After pretreatment, the sample was filtered to separate the solid pretreated wood and effluent. To study the effect of borate on alkaline extraction of hemicellulose, a control extraction with just NaOH was performed under the same conditions, including overnight extraction at room temperature and 5.5 h extraction at 90°C. The chemical component analysis was performed for the control woodchips, the control alkali-extracted woodchips, and the borate-assisted, alkali-extracted woodchips. This analysis included cellulose, Klason lignin, and sugar profile (arabinose, galactose, glucose, xylose, and mannose) determinations, which were based on published methods [21].

Kraft pulping

Kraft pulping was conducted for the control woodchips and borate-assisted, alkaline-extracted woodchips. The cooks were performed in a 1 L pressure reactor with a temperature controller. The chip charge for the control cook was 100 g on an o.d. basis. The effective alkali (EA) for this cook was 16.0% (18.8 % active alkali [AA], as sodium oxide on o.d. wood). The initial sulfidity charge was 30% and the liquor-to-wood ratio was 5:1. The process temperature was 170°C. Upon reaching the process temperature, the cooks were targeted for an H-factor of 2000. The cooking conditions for the borate-assisted, alkaline-extracted woodchips were alleviated by either decreasing the H-factor (by 35%) or reducing the EA/AA (by 30%), as indicated in **Table I**. After cooking, the chips were disintegrated in a heavy-duty blender and then screened on a 0.15 mm flat screen. Accepts were collected in a screen box, washed, and thickened in a centrifuge. The screened rejects were collected, dried, and weighed to determine the percent rejects based on the initial chip charge. The accept pulp was weighed and the consistency determined to calculate the screened yield. Kappa number of the pulps was determined based on TAPPI method T 236 om-06 "Kappa number of pulp."

The EA and AA in the residual black liquors were analyzed using an "ABC" titration procedure [17]. This "ABC" test procedure is conducted using a series of acid titrations (hydrochloric acid) to determine the concentrations of hydroxide and hydrogen sulfide anions (OH^- and HS^-).

Fiber charge

The fiber charge property of the accept pulp was measured by the carboxyl content determination using a conductometric titration methodology [22]. Air-dried fibers, equivalent to 1.50 g of o.d. fibers, were added to 300 mL of 0.10 N hydrogen chloride and stirred for 1 h. The pulp was then filtered and washed with 2000 mL of deionized water until the effluent water conductivity was <5 μV. The washed pulp was treated with a 0.001 N sodium chloride (250 mL) and 0.10 N hydrogen chloride solution (1.5 mL), stirred, and conductrimetrically titrated with 0.05 N NaOH under a nitrogen atmosphere. The conductivity changes were recorded and the carboxylic acid content determined from the titration curve.

	Control Cook	H-factor Reduced Cook for Pre-extracted Woodchips	Alkali Reduced Cook for Pre-extracted Woodchips
Effective alkali (EA), % Na_2O	16.0	16.0	11.2
Active alkali (AA), % Na_2O	18.8	18.8	13.2
Sulfidity, %	30	30	30
Maximum pulping temperature, °C	170	170	170
H-factor	2000	1300	2000
Liquid:wood (L/W) ratio	1:5	1:5	1:5

I. Cooking conditions for different wood furnishes.

Chemical composition analysis for the kraft pulp

The cellulose and Klason lignin were determined based on a published method [21]. The sugar profiles in the accept pulps were measured by high-performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD) according to a two-step acidic hydrolysis [3].

Pulp bleaching

The same bleaching conditions were applied for the bleach grade pulps from the control cook and the hemicellulose pre-extracted cook (H-factor reduced and chemical loading reduced cooks). These three pulps were treated with a $OD_0(EPO)D_1$ sequence. The D_0 and D_1 stages were performed in a water bath. During these two stages, the pulps were sealed in a plastic bag and mixed manually every 15 min. The pulps were thoroughly washed with deionized water to neutral after each bleaching stage. The O and (EPO) stages were performed in a Parr reactor. The pulps were stirred by the motor-driven stir bar during the experiment.

The experimental conditions were as follows:

- O stage: 10% pulp consistency, 4% NaOH loading, 0.05% magnesium sulfate loading, 550 KPa O_2 pressure, 95°C temperature, and 80 min duration.
- D_0 stage: 10% pulp consistency, 0.22 kappa factor, 2.5% chlorine dioxide loading, 75°C temperature, 60 min duration, and 2.0 pH.
- (EPO) stage: 10% pulp consistency, 3.6% NaOH loading, 0.05% magnesium sulfate loading, 1.0% hydrogen peroxide loading, 420 KPa oxygen pressure, 70°C temperature, and 60 min duration.
- D_1 stage: 10% pulp consistency, 1.2% chlorine dioxide loading, 75°C temperature, 120 min duration, and 4.0 pH.

After the bleaching, the bleached pulps were weighed. The pulp consistency was determined by drying the sample in an oven at 105°C. The bleaching yield was obtained from the weight differences of o.d. unbleached and bleached pulps. Kappa number of the pulps after D_1 was determined based on TAPPI method T 236 om-06.

Characterization of the bleached pulp

The chemical compositions (cellulose, Klason lignin, and sugar profiles) and fiber charge also were conducted for the bleached pulps. The analysis methods were the same as the chemical analysis for the pulps after the kraft pulping process, as discussed previously.

Intrinsic viscosity

The intrinsic viscosity [η] of the bleached pulp was determined following the ISO standard method (ISO 5351:2010). The viscosity-based average of pulp degree of polymerization (DP_v) was determined from the intrinsic viscosity measurement [23].

PFI refining

The accept pulp was PFI refined at 1000, 2000, and 3000 revolutions, as described by TAPPI method T 248 sp-08 "Laboratory beating of pulp (PFI mill method)." The resulting pulps were tested for the Canadian standard freeness (CSF) according to the TAPPI method T 227 om-09 "Freeness of pulp (Canadian standard method)."

Handsheet forming and physical strength testing

Handsheets (grammage of 60 g/m^2) were prepared following TAPPI method T 205 sp-06 "Forming handsheets for physical tests of pulp." The handsheets were then stored in a preconditioned chamber to control the humidity and temperature before physical analysis, in accordance with TAPPI method T 410 om-08 "Grammage of paper and paperboard (weight per unit area)" and T 411 om-10 "Thickness (caliper) of paper, paperboard, and combined board." The handsheet brightness was determined based on the TAPPI method T 452 om-08 "Brightness of pulp, paper, and paperboard (directional reflectance at 456 nm)." Tensile, burst, short span compression, and tear strength of the handsheets were obtained following TAPPI methods T 494-om 06 "Tensile properties of paper and paperboard (using constant rate of elongation apparatus)," T 403 om-10 "Bursting strength of paper," T 826 om-08 "Short span compressive strength of containerboard," and T 414 om-04 "Internal tearing resistance of paper (Elmendorf-type method)," respectively.

	Original Woodchip	Control Alkaline Extraction	Borate Assisted Alkaline Extraction
Solid sample			
Klason lignin	28.0	27.7	27.6
Cellulose	45.5	45.4	45.5
Hemicellulose	23.1	17.8	12.7
Arabinose	1.4	1.0	0.8
Galactose	2.1	1.2	1.0
Glucose	2.5	1.3	1.0
Xylose	7.3	5.8	5.7
Mannose	9.8	8.5	4.2

II. Compositional analysis of the post-pretreated samples (% original o.d. wood weight).

	Control Cook	H-factor Reduced Cook for Pre-extracted Woodchips	Chemical Reduced Cook for Pre-extracted Woodchips
Black liquor			
Effective alkali (EA), % Na_2O	10.6	10.9	9.9
Active alkali (AA), % Na_2O	13.5	13.9	12.7
Sulfidity, %	42.2	43.6	45.2
Rejects, %	0.3	0.4	0.4
Pulp yield, %	47.0	46.5	46.7
Kappa number	30.5	27.9	28.4
Chemical composition			
Klason lignin, % (o.d. pulp)	4.3	4.0	4.2
Arabinose, % (o.d. pulp)	0.4	0.3	0.3
Galactose, % (o.d. pulp)	0.3	0.2	0.2
Glucose, % (o.d. pulp)	84.2	84.1	83.7
Xylose,% (o.d. pulp)	5.2	4.8	5.0
Mannose, % (o.d. pulp)	4.6	4.2	4.4
Fiber charge (mmol/100g o.d. pulp)	8.0	7.2	7.6

III. Pulp chemical analysis for different cooks.

Error analysis

Multiple measurements (3 to 10 runs) were conducted for all characterization techniques, and a mean value was reported. The error margin associated with the chemical composition analysis was ±5.0%.

RESULTS AND DISCUSSION

Chemical composition analysis of extracted woodchips

Table II shows the basic chemical compositions of control and hemicellulose-extracted woodchips. After extraction, no significant change of lignin content was observed in the borate-assisted and the control alkaline-extracted samples. The cellulose contents were maintained approximately at the same level (45.5%) in the borate-assisted and the control alkaline-extracted (45.4%) samples as in the original woodchips (45.5%). In comparison with the borate-assisted and the control alkaline-extractions, borate can enhance the mannose removal by 43.9% in the extracted solid samples, which is in accordance with previous studies: the vicinal *cis*-hydroxyl groups of the mannose unit could form complexes with borate and these complexes are soluble in alkali medium [24]. Besides the significant removal of mannose, the other hemicellulose contents (arabinose + galactose + glucose + xylose) in the borate-assisted, alkaline-extracted woodchips (8.5%) were slightly lower than control alkaline extracted woodchips (9.3%), which means the borate could be generally helpful for the removal of hemicellulose during the extraction.

	Control Cook	H-factor Reduced Cook for Pre-extracted Woodchips	Chemical Reduced Cook for Pre-extracted Woodchips
Kappa number	2.6	2.7	2.5
Bleaching yield, % (based on o.d. unbleached pulp)	92.9	92.8	92.7
ISO brightness, %	85.3	85.9	85.1
Chemical composition			
Klason lignin, % (o.d. pulp)	0.4	0.5	0.4
Arabinose, % (o.d. pulp)	0.3	0.3	0.3
Galactose, % (o.d. pulp)	0.4	0.4	0.4
Glucose, % (o.d. pulp)	88.5	88.3	88.6
Xylose,% (o.d. pulp)	5.5	5.4	5.4
Mannose, % (o.d. pulp)	4.9	4.6	4.7
Fiber charge (mmol/100g o.d. pulp)	4.2	3.8	3.9

IV. Pulp property analysis for different bleached pulps (on o.d. pulp).

Kraft pulping analysis

Table III lists the EA, AA, and sulfidity in the cooking black liquor. Enough alkali was maintained throughout the three cooks to avoid lignin precipitation. The sulfidity levels were close in the three cooks (~43%). The pulps obtained from the three cooks were very similar in terms of rejects rates, pulp yield, and kappa numbers. The pulp chemical composition analysis also showed that the Klason lignin (~4.3%) and glucose content (~84.0%) were close in these three pulps. The hemicellulose content (arabinose + galactose + xylose + mannose) in the H-factor reduced cook (9.5%) and chemical reduced cook (9.9%) were slightly lower than the control cook (10.3%). The fiber charges of these three pulps were also close (~7.5%), which signified that the carboxylate content in the fiber polysaccharides and lignin were in the same level.

Bleached pulp analysis

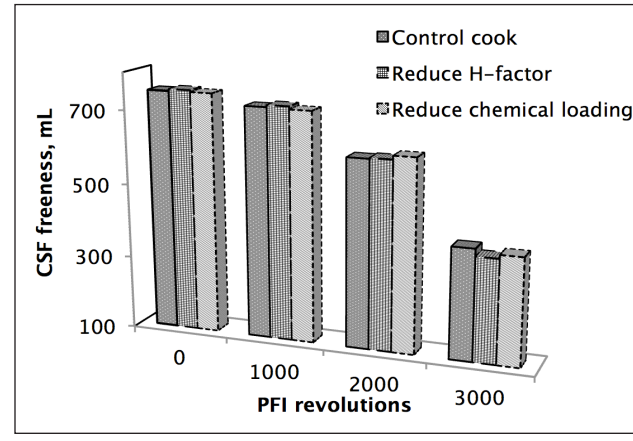
Table IV lists the kappa number, Klason lignin content, bleaching yield, sugar profiles, and fiber charge of the bleached pulp. The pulp chemical composition analysis showed that the three pulps contained very low Klason lignin (~0.4%) after bleaching. The glucose content (~88.5%) was close in these three pulps. The hemicellulose content (arabinose + galactose + xylose + mannose) in the bleached pulp from the H-factor reduced cook (10.7%) and the bleached pulp from the chemical reduced cook (10.8%) were close to the control cook (11.1%). The fiber charges of these three pulps were also comparable (~4.0 %), which signified that the carboxylate content in the fiber polysaccharides and lignin were in the same level [25]. In addition, the ISO brightness of these three bleached pulps was virtually the same (~85%).

Pulp Sample	$[\eta]$ cm³/g	DP_v
Control cook	769	1124
H-factor reduced cook for pre-extracted woodchips	763	1115
Chemical reduced cook for pre-extracted woodchips	766	1120

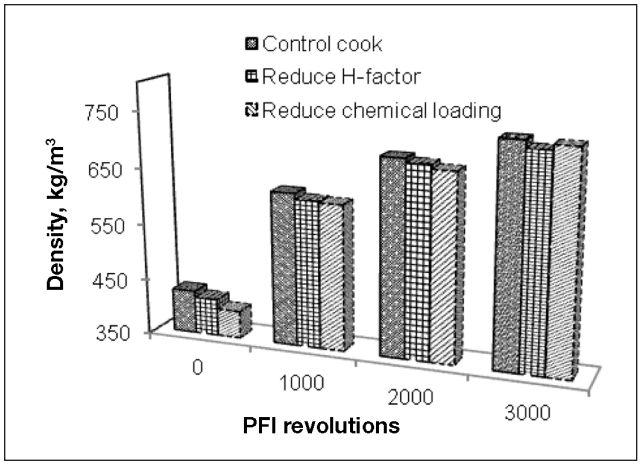
V. The intrinsic viscosity [η] and viscosity average degree of polymerization (DP_v) of different bleached pulps.

Intrinsic viscosity analysis

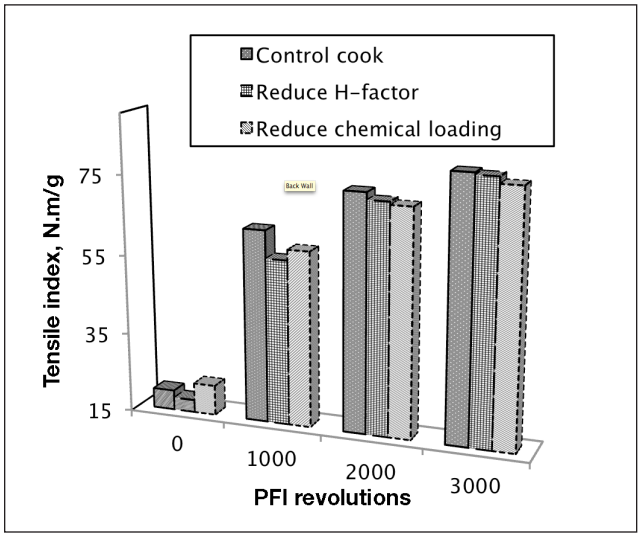
Table V lists the results of molecular weight analysis of the extracted samples. After kraft pulping, no significant difference in cellulose DP_v was observed between the control cook pulps and hemicellulose pre-extracted pulps. The DP_v of these three pulp (~1100) are in agreement with published values [26,27]. In addition, the pulp DP_v from H-factor reduced cook is similar to that from the chemical reduced cook, which signifies that optimization of the kraft pulping process is



2. Plot of pulp freeness vs. PFI mill revolutions.



3. Plot of density vs. PFI mill revolutions.



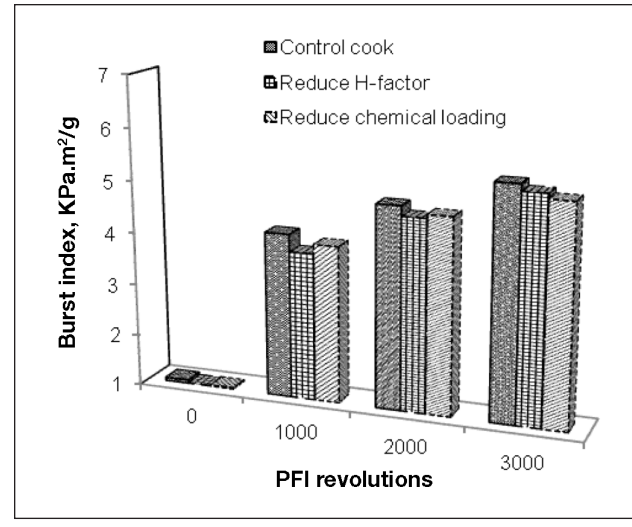
4. Plot of tensile index vs. PFI mill revolutions.

efficient for the hemicellulose pre-extracted woodchips. However, the results were obtained in the Parr reactor, which was different from the continuous cooking in the paper mill. These studies need to be further explored with continuous cooking technologies.

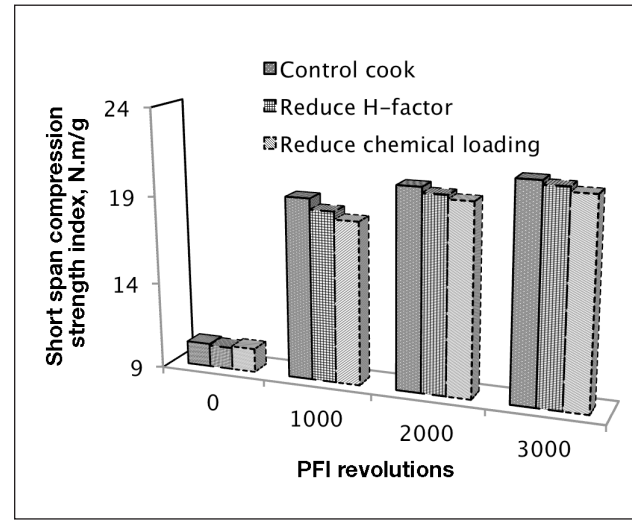
Pulp properties

Figure 2 shows the effects on freeness of PFI mill refining of the pulps from the different cooks. Under mechanical action in refining, the pulp fibers can be shortened and fibrillated internally and externally. As a result, the pulp freeness decreases. The beating behaviors are very similar among the control pulp and the pre-extracted kraft pulps from the H-factor and chemical loading reduced cook: under the same refining revolutions, these three pulps show similar freeness. This is reasonable because these three pulps had no significant variations in chemical compositions (Table IV).

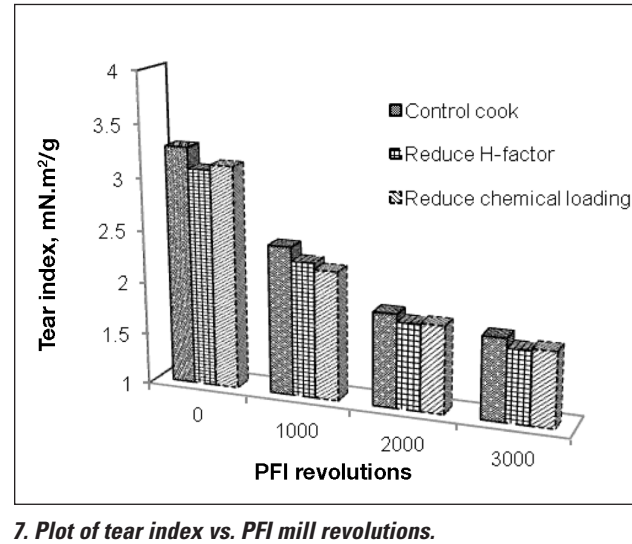
Figures 3-7 show the pulp physical properties. The sheet density and physical strengths (tensile, burst, short span compression, and tear strength) are very close between the control



5. Plot of burst index vs. PFI mill revolutions.



6. Plot of short span compression strength index vs. PFI mill revolutions.



7. Plot of tear index vs. PFI mill revolutions.

pulp and the pre-extracted kraft pulps from the H-factor and chemical-loading reduced cook. These findings indicated that the optimization of kraft pulping of the hemicellulose extracted woodchips could yield comparable physical/optical properties as the control woodchips.

CONCLUSIONS

A mild borate-assisted alkaline pretreatment was applied to loblolly pine chips before kraft pulping to extract hemicellulose for use as biofuels. Loblolly pine woodchips were first pre-extracted with 6 wt% (percent on original nonextracted o.d. wood weight) NaOH at room temperature overnight, then treated at 90°C for 90 min following another 4 h extraction with the addition of 5 wt% boric acid to partially remove hemicellulose. During the subsequent bleach-grade kraft pulping process, the cooking intensity was alleviated either by decreasing the cooking time (reduced H-factor by 35%) or decreasing the chemical charge by 30%, aiming to obtain similar pulp quality as the control cook. After ECF bleaching and PFI refining, the results indicated that the pre-extracted pulp could maintain equal or similar brightness and physical strength as the control pulp through this optimization of the pulping process. **TJ**

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We found that high-quality hemicellulose can be pre-extracted from loblolly pine woodchips using borate-assisted alkaline extraction before kraft pulping. It is possible to obtain the same pulp yield and maintain pulp quality as the nonextracted chips by either reducing the cooking time or reducing the chemical loading.

Future experiments will be focused on the optimization of enzymatic hydrolysis of extracted hemicellulose and the subsequent fermentation to convert the hydrolyzed monosaccharides to bioethanol. The inhabitation effect of borate on the fermentation will be also involved.

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