Hydrothermal Pretreatment of Hardwood Chips Prior to Alkaline Pulping and $D_0(E_P)D_1$ Bleaching

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In this study, mixed southern hardwood was pretreated *via* a hydrothermal process at 160 °C for 1 h. Alkaline pulping was then conducted on both the pretreated and original chips to prepare pulp with Kappa 17 and 30. For the Kappa 30 pulps, oxygen delignification was further carried out to render Kappa 17. After pulping, the different pulps with similar Kappa 17 were bleached by the $D_0(E_P)D_1$ sequence. Pulping results show that the pretreated chips are easier to cook and necessitate less alkaline charge to achieve a similar Kappa without decreasing the pulp viscosity. Under similar bleaching conditions, hydrothermal pretreatment leads to pulps with a higher brightness, viscosity, and paper sheet opacity and scattering coefficient, while also lowering the burst and tensile strength. Furthermore, the pulp fiber length, fiber width, and percentage of fines decreases, yet the coarseness, curl index, and kink index increase. However, the paper properties of bleached pulps are not improved significantly when subjected to modified continuous cooking (MCC) pulping or oxygen delignification.

Keywords: Hydrothermal pretreatment; Fuel ethanol; Hardwood; Alkaline pulping; $D_0(E_P)D_1$ *Bleaching*

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INTRODUCTION

The continual increase in global population, industrialization, and pollution has inherently caused an exponential increase in the demand for fossil fuels. Because of the depletion of fossil fuel reserves, the demand for fuels from renewable energy sources continues to increase. The dominate substrates used for the first generation of fuel ethanol production are pure sugars (*e.g.*, sucrose from sugarcane in Brazil) and easily degradable carbohydrates (*e.g.*, starch, from corn in the USA and cereals in Europe). In recent years, more interest has been focused on the use of lignocellulose for the production of second generation fuel ethanol (Sun and Cheng 2002; Mabro 2006; Cohen 2009).

As one of the most abundant renewable resources on the earth, lignocellulose is of great importance to the pulp and papermaking industry. To adapt to lignocellulose's beneficial characteristics, more emphasis should be given to developing effective utilization of the biomass, especially in woody biomass from Europe and North America. In recent years, many pulp and paper researchers, scholars, and companies have switched their attention to the forest biorefinery and the Integrated Forest Products Biorefinery (IFBR) (van Heiningen 2006; Wising and Stuart 2006; Janssen and Rutz 2007).

Hemicelluloses, which are one of the primary components of lignocelluloses, are dissolved to a considerable extent into the spent liquor during conventional chemical pulping and finally burnt together with lignin. Because of their low heating value, hemicelluloses can be fractionated during pretreatment prior to chemical pulping, allowing them to be used in the production of value-added products such as fuel ethanol, isobutanol, xylitol, acetic acid, and polylactic acid (Lawford and Rousseau 1991; Dominguez *et al.* 1997; Converti *et al.* 1999; Saha 2003; Neureiter *et al.* 2004; Chen *et al.* 2011; Erickson *et al.* 2012). Regarding the pretreatment technology, many different methods have been developed and used, such as hot water, dilute acid, alkaline, and organosolv pretreatment (Mosier *et al.* 2005; Pan *et al.* 2005; Wyman *et al.* 2005; Johnson and Blander 2008). Among all of these methods, hydrothermal pretreatment (hot water) is the simplest, as no chemicals are added.

Mixed southern U.S. hardwood chips were used in this study. The choice of such wood material is consistent with the following pathway: wood → pretreatment → pulping → (paper and fuel ethanol). In such a scenario the ethanol constitutes another potential revenue stream in addition to the usual sales of pulp and paper. Based on a previous study by the authors, the hydrothermal pretreatment may have negative effect on the pulp or paper properties, and the autohydrolysis at 160 °C for 1 h is more reasonable, considering both sugar yield and pulp properties. In the present study, just one hydrothermal pretreatment condition was applied to investigate its impact on alkaline pulping and pulp bleaching effectiveness.

EXPERIMENTAL

Materials

The mixed southern hardwood used in this experiment was provided by a paper mill in the United States. The chips were screened, with the accepted pieces ranging from 0.95 cm to 1.59 cm in terms of hole size and were used for hydrothermal pretreatment and alkaline pulping.

Methods

Hydrothermal pretreatment

The hydrothermal pretreatment process was carried out in an M&K Digester (USA) in parallel (one for chemical pulping and the other for analysis and characterization). The chips were cooked in hot water with a liquor to wood ratio of 4:1 at a maximum temperature of 160 °C for 1 h. The chip yield after autohydrolysis was 85.8%. After pretreatment, the prehydrolyzate and chips were separated immediately. The hydrolyzate liquid was cooled down and preserved for the subsequent analysis. The chips were thoroughly washed with a certain amount of cold water until the filtrate was colorless, then left for the eventual alkaline pulping and chemical component analysis. The flow sheet of the whole process is shown in Fig. 1.

Alkaline pulping

Alkaline pulping was conducted at the maximum temperature of 160 °C, with a liquor to wood ratio 4:1, sulfidity 25%, and an H-factor of 800 (time to temperature 50 min, time at temperature 60 min). Furthermore, the study used different alkaline charges (active alkaline: 12% to 20%) to target pulps of Kappa number 30 and 17, respectively. Kraft pulping was done on both the original and pretreated chips.

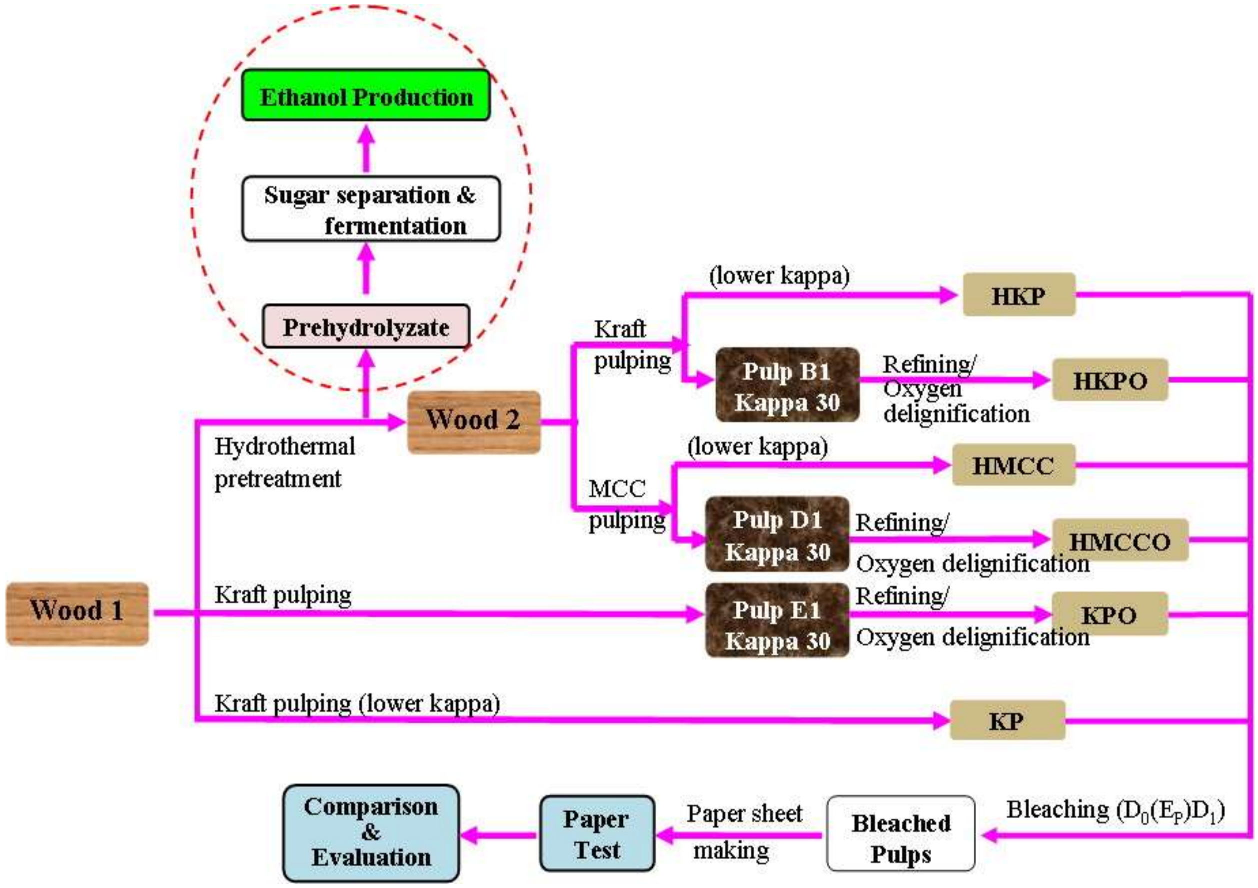

Fig. 1. Flow sheet of the hydrothermal pretreatment and alkaline pulping process. KP, Kraft pulping; HKP, hydrothermal pretreatment-Kraft pulping; HMCC, hydrothermal pretreatment-MCC; KPO, Kraft pulping-oxygen delignification; HKPO, hydrothermal pretreatment-Kraft pulping-oxygen delignification; HMCCO, hydrothermal pretreatment-MCC-oxygen delignification

For the pretreated chips, modified continuous cooking (MCC) pulping process was also carried out, in which the chemicals were added by pumping from the other vessel. The liquor profiling for the process is shown in Fig. 2. After cooking, the pulps were thoroughly washed and screened.

Oxygen delignification

For the Kappa number 30 pulps, oxygen delignification was carried out in a 2.8-L reactor in an oven heated by hot air. The NaOH dosage ranged from 1.0% to 3.5% to target Kappa 17. The conditions were as follows: 10% pulp consistency, oxygen pressure 100 psi, maximum temperature 100 °C, total reaction time 60 min (including time to temperature of 30 min), and a dosage of 0.1% for $MgSO_4$.

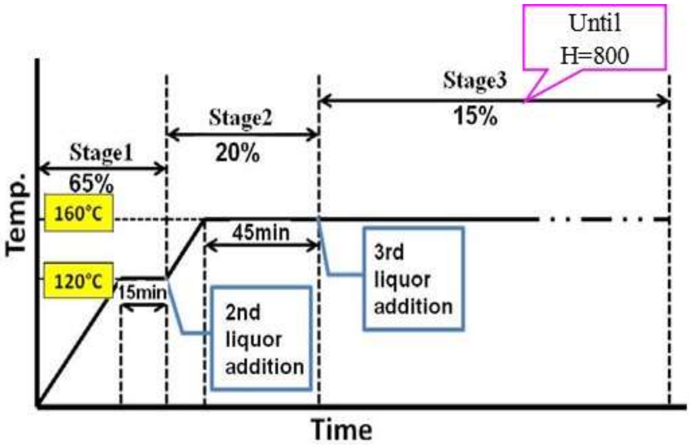

Fig. 2. Liquor profiling for MCC process

$D_0(E_P)D_1$ bleaching

The $D_0(E_P)D_1$ bleaching sequence was applied. The specific conditions were as follows: pulp consistency 10%, D_0 (50 °C, 1 h, Kappa factor 0.21), E_P (70 °C, 1 h, H_2O_2 charge 0.5%), and D_1 (70 °C, 3 h, ClO_2 charge 1.25%). The pulps were thoroughly washed with deionized water after the E_P and D_1 stages.

Evaluation of the pulp and paper

The handsheets (60 g/m^2) used in this report were prepared based on TAPPI T205 (1988). The paper properties, pulp Kappa number, viscosity, and brightness were tested in accordance with TAPPI T220 sp (2010), TAPPI T236 om (1999), TAPPI T230 om (1999), and TAPPI T452 om (1998). The pulp fibers were analyzed by fiber quality analysis (FQA), OpTest Equipment Inc. (Canada).

Analytical methods

The carbohydrates present in both the wood and prehydrolyzates were analyzed *via* Ion Chromatography (ICS-3000, Dionex; USA) using CarboPac^{TM} PA1 (2 × 250 mm) as the analytical column. For the sugars in the prehydrolyzate liquor, both monomers and oligomers were analyzed. The total sugars were obtained by analyzing liquors hydrolyzed with 3% H_2SO_4 at 121 °C for 1.5 h in addition to the monomers obtained during direct analysis. The difference between the two sugars is referred to as the oligo-sugars.

Benzene-ethanol extractive content, lignin, and ash content were determined referring to the standard methods TAPPI T204 cm (2007), TAPPI T222 om (2002), and TAPPI T211 om (2002).

RESULTS AND DISCUSSION

Composition of Wood and Prehydrolyzate

Hydrothermal pretreatment of the chips was performed, and the composition of the wood and liquor is shown in Table 1. When comparing the content of cellulose in the original (O) and hydrothermal pretreated wood (H), a certain amount of hemicelluloses was found to have dissolved into the liquor, predominantly oligo-sugars. Low degradation of cellulose is beneficial for the overall yield. As to lignin, the acid-soluble lignin content in prehydrolysed wood was greatly decreased, which was due to the lignin degradation and dissolution into the liquor during hydrothermal pretreatment. It is worth noting that the total lignin in wood and prehydrolyzate was imbalanced, which should be related to the precipitation of partial soluble lignin during cooling of the prehydrolyzate (Leschinsky *et al*. 2009).

Table 1. Composition of Wood and Prehydrolyzate (% of Original Wood)

Samples	O	H	Prehydrolyzate
[a]Extractive	2.58	4.06	-
Klason	19.68	16.67	0.35
[b]ASL	4.27	2.27	1.90
Cellulose	45.14	43.59	-
Hemicellulose	24.36	15.15	-
[c]Mono-/oligo-	-	-	0.76/5.49
Furfural/HMF	-	-	0.14/0.09
Ash	0.35	0.12	-

Notes: O, original wood; H, hydrothermal pretreated wood; HMF, 5-hydroxymethylfurfural; [a]benzene-ethanol (2:1 v/v) extractive; [b]acid soluble lignin; [c]monomers and oligomers of sugars

Alkaline Pulping

Table 2 lists the pulping results for the original and hydrothermal pretreated chips. For higher kappa pulping, similar alkalinity was needed to reach similar kappa levels for both the original and hydrothermal pretreated wood. However, at higher alkaline charges, the hydrothermal process leads to lower kappa without decreasing the pulp viscosity.

Table 2. Pulping Results under Different Conditions

	AA%	Kappa No.	Viscosity (cP)
KP	14	27.5	65.1
	16	20.0	63.1
	18	17.2	44.8
	20	13.9	37.3
HKP	12	64.2	116
	14	28.4	89.0
	16	16.1	68.0
	18	13.0	60.2
HMCC	14	34.7	-
	16	18.7	102.5
	18	13.4	71.8

Note: KP, HKP, and HMCC are Kraft pulping, hydrothermal pretreatment-Kraft pulping, and hydrothermal pretreatment-MCC, respectively. AA%, the active alkaline dosage in pulping

In the case of Kappa 17 pulps, the alkaline charges for KP (Kraft pulping), HKP (hydrothermal pretreatment-Kraft pulping), and HMCC (hydrothermal pretreatment-

MCC) are 18%, 16%, and 16%, respectively, which indicates that the autohydrolysis process can decrease the amount of chemicals required for cooking. Obviously, pulp yield is crucial for the pulp and papermaking industry, with higher screened pulp yield being preferred. The relationship between pulp yield and active alkaline charge or kappa number, is shown in Figs. 3 and 4.

As can be seen in Fig. 3, the total pulp yield decreased as the alkaline charge (12 to 20%) increased for all the pulps. In regards to the screened pulp yield, an increasing trend was shown with higher alkaline charge (12% to 18%). However, when the alkaline charge for the original wood was further increased to 20%, the overall screened pulp yield decreased. As a whole, pulp yield typically decreased after hydrothermal pretreatment, which was attributed to the loss of hemicelluloses into the prehydrolyzate (Table 2).

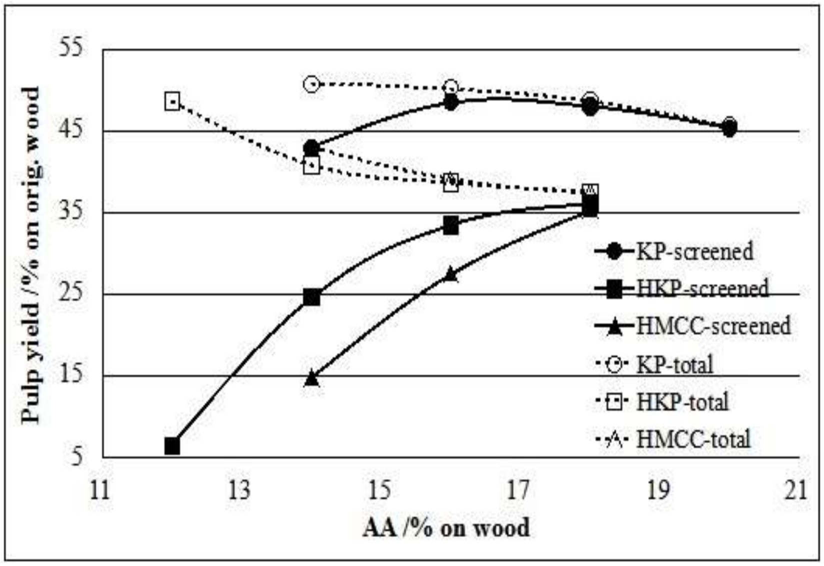

Fig. 3. Effect of alkaline charge on pulp yield

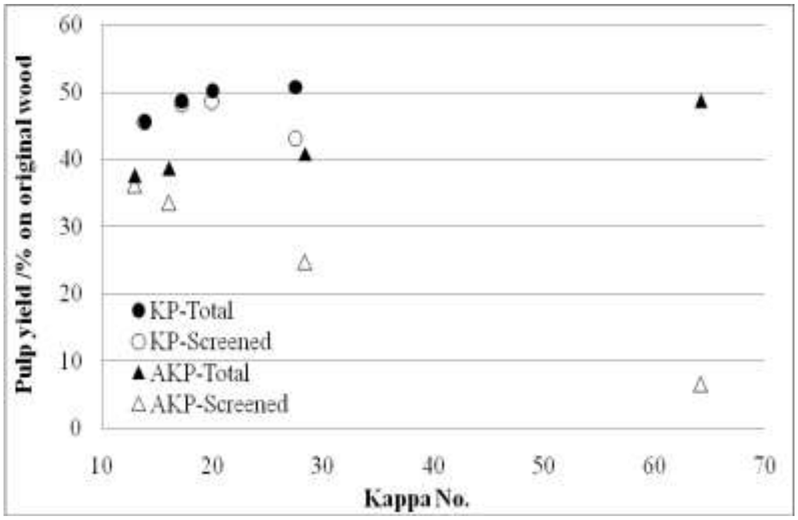

Fig. 4. Relationship between Kappa No. and pulp yield

As shown in Fig. 4, the total pulp yield was related to the kappa number for both the original and hydrothermal pretreated chips. The pulp yield was lower for the autohydrolysis case, as some hemicellulose sugars were dissolved into the prehydrolyzate. It should be noted that more rejects were produced after autohydrolysis. This phenomenon may be explained by the lignin condensation and possibly precipitation onto the chip surface during hydrothermal pretreatment (Chua and Wayman 1979).

Effect of Hydrothermal Pretreatment on Pulp Bleachability

Six pulp samples of similar kappa were chosen for bleaching, three of which were subjected to oxygen delignification. Table 3 shows the details of the unbleached pulps, and Table 4 provides the results for the bleached pulps.

Table 3. Results of Unbleached Pulps

Sample No.	Unbleached pulp			
	Kappa No.	Viscosity /cP	Pulp yield /%	Brightness / %ISO
KP	17.2	44.8	48.1	33.5
KPO	(27.5)15.5	(65.1)60.6	(44.2)41.2	(28.8)46.0
HKP	15.5	95.6	39.3	35.2
HKPO	(28.4)16.5	(89.0)55.7	(41.4)38.6	(29.5)40.9
HMCC	17.1	101	39.1	31.7
HMCCO	(34.7)17.3	(-)56.1	(42.7)39.7	(24.5)42.0

Notes: O indicates oxygen delignification; the data in the parentheses are from the pulps before oxygen delignification.

Table 4. Results of Bleached Pulps

Sample No.	(E_P)			D_1		
	Resi. H_2O_2 /%	P No.	Brightness / %ISO	Resi. ClO_2 /%	Brightness / %ISO	Viscosity (cP)
KP	12.2	1.5	72.9	6.9	88.9	28.3
KPO	26.9	1.4	75.2	5.6	90.6	27.2
HKP	17.1	0.5	77.6	19.9	90.4	31.5
HKPO	39.2	0.6	78.1	18.1	90.7	25.1
HMCC	17.8	0.6	77.6	15.4	89.3	38.8
HMCCO	33.1	0.4	78.2	19.8	90.8	23.1

Notes: O indicates oxygen delignification

When compared to the original wood, unbleached pulps subjected to hydrothermal pretreatment led to the following: (1) higher viscosity, which can be attributed to lower hemicellulose content; and (2) a greater decrease in pulp viscosity by oxygen delignification, which may be due to the high charge of sodium hydroxide to attain similar kappa in the O_2 stage.

In the case of pulp bleachability, hydrothermal pretreatment resulted in higher residual peroxide and chlorine dioxide, higher brightness after both the E_P and D_1 stages, and a much lower P number after the E_P stage. Furthermore, the higher bleachability should be related to the decreased content of hemicelluloses, hexuronic acid, and transition metal ions in pulps (Liu *et al.* 2008). Also during pulp bleaching, oxygen delignification results were much higher in regards to residual peroxide in the E_P stage (Reitberger 2001). Similar results were obtained for brightness. The MCC process made it possible to reach higher pulp viscosity with less reagent consumption;

Effect of Hydrothermal Pretreatment on Paper Properties

Bleached pulps were refined using PFI milling, with a target CSF (Canadian Standard Freeness) of 300 mL for handsheet making and testing. As can be seen from the curves in Fig. 5, lower slopes for the pulps from pretreated wood indicated more difficulty in the refining process.

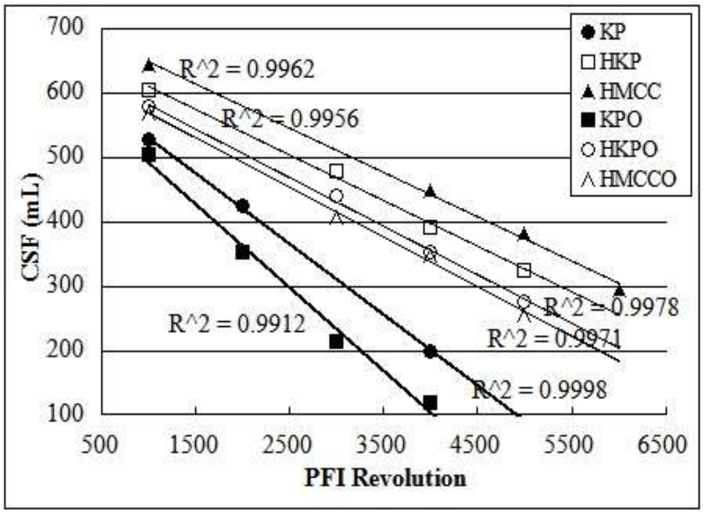

Fig. 5. Effect of autohydrolysis on PFI refining

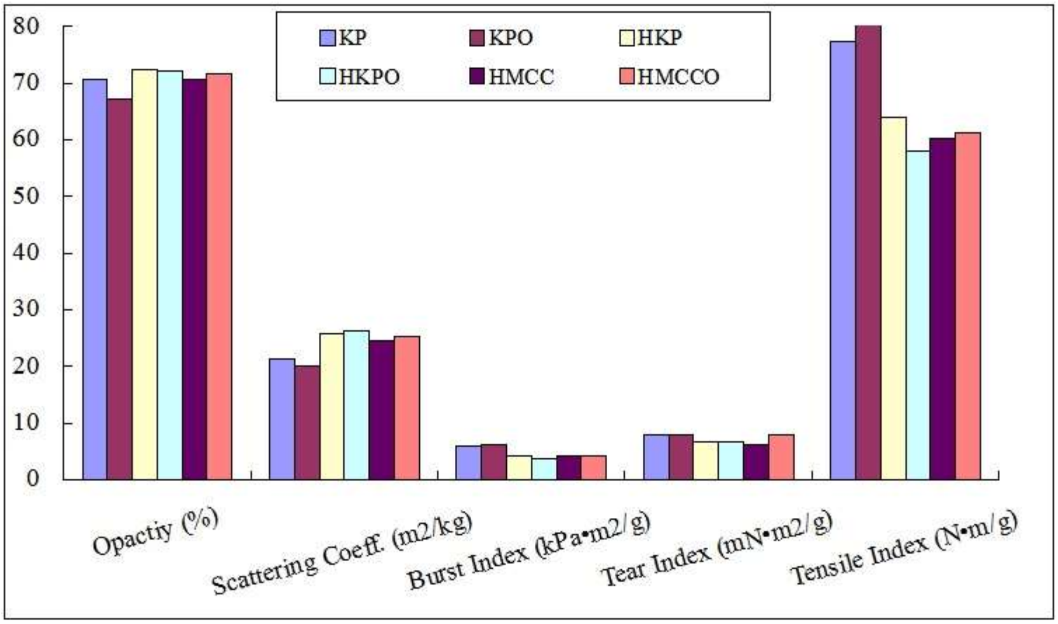

Fig. 6. Effect of hydrothermal pretreatment on paper properties (CSF 300)

Such results are caused by the removal of hemicelluloses, which can help enhance fiber swelling and fibrillation. At CSF 300 mL, the beating revolutions needed for the hydrothermal pretreated wood was nearly doubled compared to original wood. An increase in beating revolutions is not favorable for a papermaking operation, as more refining energy will be needed.

Paper testing results are shown in Fig. 6. Obviously, after hydrothermal pretreatment, the pulp had the following characteristics: (1) a higher opacity and scattering coefficient, which is favorable for printing and specialty paper; (2) similar or higher tearing strength; and (3) lower burst and tensile strength, which may be caused by the loss of hemicelluloses and some degree of cellulose degradation. These results can be further explained by the decreased length, and the increased curl and kink index shown in Table 5. Oxygen delignification had a negative effect on the pulp strength, which was manifested by the increased curl and kink index. These results are consistent with those in the literature (Mohlin and Alfredson 1990; Kontturi *et al*. 2008).

Table 5. Fiber Morphological Characteristics by FQA

Sample	Percent Fines /%	L_W /mm	Width /μm	Coarseness mg/m	Curl index 1/mm	Kink index 1/mm
KP	46.25	1.12	18.7	0.086	0.087	1.35
KPO	37.84	1.12	19.2	0.097	0.088	1.43
HKP	28.41	1.07	18.0	0.088	0.146	2.08
HKPO	37.12	0.99	18.4	0.094	0.231	2.93
HMCC	27.08	1.09	18.0	0.088	0.162	2.28
HMCCO	45.03	1.01	17.8	0.090	0.206	2.80

CONCLUSIONS

1. Hydrothermal pretreatment can reduce the amount of chemicals required for the pulping and bleaching process.
2. Because of hemicellulose loss, the pulp yield is significantly decreased without decreasing the viscosity for similar kappa pulps.
3. It can be speculated that the improved pulp bleachability is related to the decreased content of hemicellulose, hexuronic acid, and transition metal ions.
4. Under the same bleaching conditions, autohydrolysis results in higher opacity and a higher scattering coefficient, with a lower burst and tensile index. Furthermore, the fiber length, width, and percentage of fines decrease, as the curl and kink indexes increase. However, no significant improvements in paper properties were shown by introducing MCC pulping or oxygen delignification.

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