Sodium carbonate pre-extraction of bamboo prior to soda-anthraquinone pulping

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Abstract

A part of hemicelluloses is dissolved in black liquor during pulping process, which can be extracted prior to pulping, that would be used for producing biofuels, biopolymers, paper additives and other chemicals in addition to pulp. However, the amount of hemicelluloses extraction must be limited to maintain pulp yield and pulp quality. In this investigation hemicelluloses were pre-extracted from bamboo with weak alkali (Na_2CO_3) and compare with hot water and dilute H_2SO_4 extraction prior to soda and soda- AQ pulping. For control experiment bamboo chips were also pulped by kraft process without pre-extraction. The biomass dissolution in weak alkaline pre-extraction was 11.1%, which was 1.2 and 7.9% lower than the hot water and dilute H_2SO_4 pre-extraction, respectively. In the alkaline pre-extraction process, 1.3% lignin, 1.7% acetic acid and about 6.4% sugars (on od bamboo) were extracted. Soda-AQ process responded well in term of pulp yield and pulp quality. Pulp yield after alkaline pre-extraction was higher and kappa number was lower than the hot water and dilute H_2SO_4 pre-extraction. Alkaline pre-extraction prior to soda-AQ pulping produced pulp with lower kappa number than non-extracted kraft pulp at almost similar pulp yield. This pulp showed better results in prebleaching with peroxyformic acid. Alkaline pre-extracted soda-AQ pulp had better tear index and slightly inferior tensile index than the non-extracted kraft pulp after $D_0E_pD_1$ bleaching. Final brightness was 1.1% higher than the control pulp. Alkaline pre-extraction is better option to integrate paper grade pulp mill in a biorefinery.

Keywords: Pre-extraction, Sugars, Soda-AQ pulping, Pulp yield, Papermaking properties

Introduction

Bamboo belongs to the family of grasses, which is most widely used for pulp production in Asia (Atchison, 1998). In Bangladesh, the main fibrous raw material is bamboo, is cooked by kraft process with conventional CEH bleaching sequences. World demand for paper and paperboard is likely to grow from 300 million tonnes to over 490 million tonnes by the year 2020, which is expected to raise the cost of pulp wood (Agnihotri et al., 2010). The increasing cost coupled with global warming, have increased interest on processes for more efficient utilization of whole fraction of raw material according to the “zero waste” context (Ohara, 2003; Kamm and Kamm, 2004). In this context, the implementation of the biorefinery concept in the existing chemical pulp mills is regarded as a strategy for the sustainable co-production of pulp, fuels, power and high value chemicals from diverse and heterogenous lignocellulosic materials (van Heiningen 2006).

Different pre-extraction methods have been studied. Hot water (autohydrolysis) pre-extraction is considered as one option (Leschinsky et al., 2009). The advantages of hot-water pre-extraction are no chemicals use other than water. In hot-water pre-extraction acetic acid is released from the acetylated carbohydrates in biomass, and pH is reduced to 3-4 (Wafa Al-Dajani et al., 2009). This makes autohydrolysis similar to acid hydrolysis in the way that leads to unwanted side reactions at elevated temperatures, which converts part of hemicelluloses to furfural and generates inhibitors for a potential subsequent fermentation process (Larsson et al., 1999; Palmqvist and Hahn-Hägerdal 2000). In addition, acid pre-extraction causes random chain cleavage in cellulose and hemicelluloses; thus promotes peeling reactions, ultimately losses pulp yield under alkaline pulping conditions (Yoon and van Heiningen 2008; Wafa Al-Dajani et al., 2009). By contrast, alkaline pre-extraction gave lower yields of hemicelluloses but improved subsequent kraft pulping process conditions in terms of chemical use and reaction time needed to reach a given kappa number. Pulp yield and fiber properties were comparable or slightly better than those of a control cook (Wafa Al-Dajani and Tschirner 2008). Another study (Walton et al., 2010) showed that water extraction (0% alkali addition) released the greatest amount of carbohydrates, up to 30 g/L measured as component sugars, but resulted in the greatest decrease in pulp yield, dropping from 47% to 35%. Extraction with 2% green liquor increased the pulp yield to 51% while greatly reducing the component sugars to 8 g/L.

It is known that the hemicellulosic fraction (glucomannans) in softwood is more susceptible in alkaline peeling reaction while the hemicellulosic fraction in hardwood (xylan) is more stable in alkaline solution (Fardim and Duran, 2004; Patt et al., 2006; Schild et al., 2010). Therefore, the alkaline extraction would be more suitable for partial removal of hemicelluloses from hardwoods than softwoods (Helmerius et al., 2010, Walton et al., 2010). Based on this fact, Helmerius et al. (2010) extracted xylan from birch wood chips by white liquor prior to kraft cooking without decreasing the pulp yield and paper strength properties. Alkali extraction of hemicelluloses from lignocelluloses can be considered as well-integrated with an existing alkaline process, since it will lower the alkali charge during cooking.

Vena et al (2013) extensively studied on the giant bamboo pre-extraction prior to kraft and soda-AQ pulping. Author concluded that the pulp mill biorefinery concept through hemicelluloses pre-extraction with NaOH can be achieved with modified kraft pulping or the soda-AQ pulping processes, but it depends on the type of raw material, extraction method and quality and performance requirements of a particular paper. In Bangladesh muli bamboo is used as pulping raw material. But no information is reported on the pre-extraction and its impact on pulping.

The purposes of the present work was to evaluate extraction of hemicelluloses from bamboo chips prior to soda-anthraquinone (AQ) cooking and to study on its impact on pulp yield. The hot water and dilute acid pre-extractions were also carried out for comparison. Bamboo chips are cooked by kraft process in Bangladesh. Therefore, one set of bamboo pulping without pre-extraction was carried out by kraft process.

Materials and methods

Materials

Bamboo chips were collected from the Kharnaphuli Paper Mills (KPM). All chemicals used in this study were GPR grades and received from E-Merck, Germany.

Prehydrolysis

Bamboo chips were pre-extracted by water, 0.25% H_2SO_4, 0.5% Na_2CO_3 in 5 L capacity digester. Pre-extraction was carried out at 170 ^oC for 60 min. The bamboo to liquor ratio was 1: 4. The time required to raise to maximum temperature (170^oC) was 50 min. After completing pre-extraction, pressure was released and the digester was cooled by circulating cold water. Samples were then collected from the drained off liquor for pH, solid content, lignin and sugars determination. The percentage of dissolved components was measured gravimetrically.

Lignin analysis

The dissolved lignin in the prehydrolysate was measured based on the UV/Vis spectrometric method at 205 μm wavelength (TAPPI UM 250).

Pulping

Pulping of pre-extracted bamboo was carried out by soda and soda-AQ processes in the same digester as in pre-extraction. Non-extracted bamboo chips were pulped by kraft process as control experiment. Pulping conditions were as follows:

- Active alkali: 16, 18 and 20% on oven-dry (o.d.) raw material as NaOH
- Sulphidity: 30%
- Cooking time: 120 min at maximum temperature of 170 ^{0}C. Raise to maximum temperature (170 ^{o}C) from room temperature was 50 min.
- Liquor to material ratio: 4:1.

After digestion, pulp was washed till free from residual chemicals, and screened on flat vibratory screener (Yasuda, Japan). The screened pulp yield, total pulp yield and screened reject were determined gravimetrically as percentage of o.d. raw material. The kappa number of the resulting screened pulp was determined in accordance to TAPPI Test Methods (T 236 om-99).

Peroxyformic acid pre-bleaching

Pre-extracted and non-extracted bamboo pulps were further delignified with peroxyformic acid (PFA) at 80 °C for 120 min and maintained 10% pulp consistency. The reaction was carried out in a thermostatic water bath. The peroxyformic was prepared by adding 90 % formic acid with 4% H_2O_2. After completing pre-treatment, a pulp was filtered off and washed with 80 % fresh formic acid, and finally with hot water. The kappa number and viscosity of the resulting pulp was determined in accordance with TAPPI (Technical Association of Pulp and Paper Industries) Test Methods (T 236 om-99) and (T 230 om-99), respectively.

Bleaching

Peroxyformic acid (PFA) pre-bleached pulps were bleached by $D_0E_pD_1$ bleaching sequence. The ClO_2 charge was 2% and temperature was 70 °C for 60 min in the D_0 stage. The pH was adjusted to 2.5 by adding dilute H_2SO_4. In alkaline extraction stage, temperature was 70 ^{o}C for 60 min and NaOH and H_2O_2 charge 2% and 0.5%, respectively. In the final D_1 stage, ClO_2 charge was 0.5, pH was adjusted to 4.5 by adding dilute alkali.

Strength properties

The bleached pulps were beaten in a PFI mill to about 40 °SR and handsheets were made for determine tear index (T414 om-98), tensile index (T404 cm-92), and burst index (T403 om-97). All properties were determined according to TAPPI Standard Methods given in the parenthesis.

Results and discussion

Pre-extraction

In order to integrate a paper grade pulp mill into a forest biorefinery, pre-extraction of hemicelluloses prior to pulping without sacrificing pulp yield and properties is a crucial step. Bamboo is the main fibrous source for pulp production in Bangladesh, Therefore, in this investigation, different hemicelluloses pre-extraction processes namely- hot water extraction, H_2SO_4 reinforced extraction and Na_2CO_3 reinforced extraction of bamboo without degrading cellulose have been evaluated for producing paper grade pulp. As shown in Table 1 the solid biomass dissolution was dependent on the pH of the pre-extracted liquor. The solid residue of bamboo after water pre-extraction was 88%, which was decreased to 81% when 0.25% (v/v) H_2SO_4 was added in pre-extraction process. The pH value of H_2SO_4 pre-extracted liquor was 2.2. This condition is too harsh to hemicelluloses extraction. Therefore, pre-extraction with dilute acid is not suitable for paper grade pulp. It is a common practice of using dilute acid extraction of hemicelluloses from hardwood prior to kraft pulping for the production of dissolving pulps (Koukios and Valkanas, 1982; Saeed et al., 2012). As shown in Table 1, the solid residue after alkaline pre-extraction was 89%. This lower biomass dissolution was due to mild condition of extraction medium (Walton et al., 2010). Helmerius et al., (2010) showed that pre-extraction with 3% EA (ethyl acetate) resulted in solid residue yields of between 89% and 96% depending on time and temperature. The yield was decreased with increasing EA concentration of pre-extracted liquor. Helmerius et al., (2010) also showed that hot water pre-extraction yielded lowest solid residue.

The maximum concentration sugar in the pre-hydrolysis liquor (PHL) was obtained from dilute acid hydrolysis, which was 9.6%. Hot water pre-extraction released acetic acid from acetyl group attached to hemicelluloses, which reduced pH value to 4.4, dissolved 6.8% sugars from the bamboo chips. Helmerius et al., (2010) also showed that hot water pre-extraction at 160 ^{o}C for 90 min extraction time resulted in 29.1 g/L of xylose.

On the other hand alkaline pre-extraction neutralize the generated acid (pH value 6.8) and mildly dissolved hemicelluloses by 6.4% only. Based on the liquor/bamboo ratio used (4:1), the concentration of sugar in the alkaline pre-extracted liquor would be 1.6%. As the theoretical conversion rate of pentoses to ethanol is approximately 50% (Lee et al., 2001), the resulting ethanol concentration after fermenting the hydrolysate would be 0.8%. It is known that one of the most energy-intensive steps in the ethanol production process is the recovery of ethanol from the fermentation broth by distillation (Zacchi and Axelsson 1989). The cost of distillation decreases as ethanol concentration increases, a starting concentration of around 5% of ethanol is generally considered the minimum. Therefore, an additional process step for increasing sugar concentrations after pre-extraction will be required. It was observed that nanofiltration increased sugar concentration significantly (Ahsan et al., 2014).

The sums of solid residue and solid content of pre-extracted liquor were 97.34, 93.74, 103.45 for water, dilute acid and alkaline pre-extraction, respectively. The mass balance in alkaline pre-extraction was more than 100%, which was due to the presence of Na_2CO_3 in the liquor (Table 1). The lower mass balance in acid prehydrolyis can be attributed by the formation of carbohydrate degradation products (Saeed et al., 2012).

Pulp yield and Kappa number

Alkaline pre-extraction prior to pulping solubilise hemicelluloses and lignin through saponification of intermolecular ester bonds crosslinking hemicelluloses and lignin (Sun et al., 1995). Kraft pulping of autohydrolysis residues resulted in a much lower total pulping yield as compared to a control cook (Yoon and Van Heiningen 2008; Wafa Al-Dajani et al., 2009). By contrast, pre-extraction of wood chips with alkaline media gave lower yields of hemicelluloses extracts but improved subsequent kraft pulp yield. An ideal pre-extraction stage to be tailored would remove selectively the target biomass components (mainly hemicelluloses) without affecting overall pulp yield and pulp quality.

Pulping of solid residues after pre-extraction were carried out in soda and soda-AQ processes with varying alkali charge and compared with non-extracted kraft process. As shown in Table 2, bamboo chips could be pulped to kappa number 24.7 in kraft process where pulp yield was 41.7%. This result is similar to mill produced pulp from bamboo. Therefore, kappa number and pulp yield of this range are considered as target values in this investigation. Soda process showed lower screened yield and higher kappa number. It is well known that AQ accelerate delignification and reduce alkaline peeling reaction through oxidation-reduction mechanism (Fleming et al., 1978), consequently increases pulp yield and decreases kappa number (Jahan et al., 2001). H_2SO_4 extracted bamboo chips produced dark and low yield pulps with high rejection levels (Table 2). This was due to the higher xylan removal together with the decrease in molecular mass of xylan under acidic conditions. Thus, the increased content of reducing end groups and the xylan becomes easily soluble in the subsequent alkali medium. Consequently, cellulose became more susceptible to the peeling reaction because the xylan layer on the cellulose fibrils was partially removed, hence the decrease in pulp yield (Wafa Al-Dajani et al., 2009). As shown in Table 2, delignification was difficult for acid pre-extracted bamboo chips. In soda process, kappa number was reached to 34.2 with 20% alkali charge where overall pulp yield was only 29.6%. The hydrophobic nature of lignin might have affected the ability of pulping liquor to penetrate and diffuse through the cell wall structure of the plant material, hence high rejection levels (Dutt, D., & Tyagi, C. H., 2011). Another possible reason of lower delignification is residual lignin condensation of acid pre-extracted chips (Salmela et al., 2008). In a previous study, H_2SO_4 extraction of hemicelluloses from aspen hardwood chips prior to kraft pulping produced dark and brittle wood chips undesirable for subsequent pulp production (Wafa Al-Dajani et al., 2009).

Figure 1 shows delignification of alkali pre-extracted bamboo chips. It is clearly seen that pre-extraction increased delignification. At the alkali charge of 18% as NaOH, kappa number of bamboo kraft pulp without pre-extraction was 31.9 while the alkali pre-extracted soda pulp was 25.7, which was further reduced to 18.5 in soda-AQ process. This means that pre-extracted bamboo chips had 42% lower kappa number than the non-extracted kraft pulp. This is potentially due to the structure of the pre-extracted wood chips is more loosen than the raw wood, allowing for the easier access of the cooking liquor into the extracted wood chips (Wafa Al-Dajani and Tschirner, 2008).

The overall pulp yield of alkali pre-extracted bamboo chips was 39% with kappa number 25.7 at the alkali charge of 18% in soda process. With the introduction of AQ, pulp yield was increased to 39.7% with kappa number 18.5 at the same alkali charge (Table 2). Still overall pulp yield was 2% lower. The advantage was pre-extraction was 8 points lower kappa number, which can reduce bleaching cost.

Figure 2 shows overall pulp yield kappa relationship of different pre-extracted bamboo chips in soda-AQ process and compares with non-extracted bamboo chips in kraft process. It is observed that non-extracted chips showed superior pulp yield but inferior delignification as compared to pre-extracted bamboo chips. At kappa number 25, pulp yield of alkaline pre-extracted bamboo chips was 39%, which was slightly higher than hot water pre-extracted bamboo chips. But acid pre-extracted bamboo chips yielded only 34% pulp.

Prebleaching

In order to reduce the bleaching cost, pulps were pre-bleached by peroxyformic acid and results are shown in Table 3. Peroxyformic acid converts lignin to soluble fragments by two different mechanisms. First, peroxy acid reduces the molecular weight of lignin polymers by cleaving β-aryl ether bonds and both carbon–carbon and carbon–oxygen bonds linked to the aromatic rings. (Lawrence et al., 1980). Second, peroxy acid causes other reactions that increase the water solubility of lignin: dealkylation of O-methyl groups, introduction of hydroxyl groups to aromatic rings and cleavage of the aromatic rings into muconic acids (Lawrence et al., 1980). These reactions increase the polarity of lignin and create water-soluble lignin fragments that wash away from the lignocellulosic biomass. Due to selective delignification nature of peroxy is also gaining interest in pre-bleaching of pulp. Pre-extracted and non-extracted bamboo chips cooked with 18% alkali charge as NaOH were prebleached by peroxyformic acid. Peroxyformic acid treatment reduced kappa number by 9% to 54% and increased brightness (Table 3). Hot-water pre-extracted soda-AQ process showed the lowest reduction of kappa number due to initial kappa number (18.0) was much lower. But alkaline pre-extracted pulp responded well in peroxyformic acid treatment. After peroxyformic acid treatment kappa number of alkaline pre-extracted pulp reached to 11.7 and 9.7 for soda and soda-AQ pulp, respectively. Non-extracted kraft pulp reduced kappa number by 35.5%. In our previous study peroxyformic acid treatment reduced kappa number by 30% for hardwood pulp and 37% of bamboo pulp, while brightness increased 12.2% and 8.6%, respectively (Jahan et al., 2013). Another study of organic solvent-based delignification has shown that peroxyacetic acid is a good delignifier as well as a good brightening agent (Mire et al., 2005). During the peroxyformic acid treatment electrophilic HO^+ ions are formed ($HCOOOH + H^+ = HCOOH + HO^+$). According to model compound studies the main reactions for the HO^+ ions with lignin are expected to be ring hydroxylation, oxidative ring opening, substitution of side chains, cleavage of β-aryl ether bonds, and expoxidation (Gierer et al., 1982; Hortling et al., 1991; Strumila and Rapson 1975; Yuan et al., 1998).

Bleaching

As it was observed in earlier sections that alkaline pre-extraction produced better pulp yield as compared to hot-water and acid pre-extracted bamboo chips. Therefore, alkaline pre-extracted pulp was selected for bleaching and strength properties evaluation. Table 4 shows that improved tear index and worse tensile and burst indices were observed for pre-extracted pulp. In soda pulp had 27% lower tensile index value than the non-extracted kraft pulp. But tensile index value was improved by 22% in soda-AQ pulp. It is known that pulps with high cellulose/hemicelluloses ratio have low tensile index and high tear index, fracture toughness and folding endurance (Molin and Teder 2002). It was also shown the removal of xylan reduced fiber swelling properties (Moss and Pere 2006). The final brightness of alkaline pre-extracted soda-AQ reached to 77.4% in $D_0E_pD_1$ bleaching sequences, which was 1.1% higher than the non-extracted kraft pulp.

Conclusions

Alkaline pre-extraction prior to pulping yielded 6.4% sugars and increased delignification degree in subsequent pulping. But acid pre-extraction yielded 9.6% sugars but retarded delignification due to condensation of residual lignin. Overall pulp yield after alkaline pre-extraction was slightly lower but kappa number was also lower, which help in bleaching stage. Lower pulp yield and quality in alkaline pre-extracted bamboo chips were compensated in soda-AQ process. Alkaline pre-extracted bamboo chips pulp showed slightly lower tensile index and higher tear index than the non-extracted bamboo kraft pulp. At the same bleaching chemical consumption, soda-AQ pulp had 1.1% higher final brightness than the control kraft pulp.

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Table 1. Pre-extraction of bamboo chips

Pre-extraction process	Solid residue (%)	Pre- extracted liquor				
		Solid content (%)	pH	Lignin (%)	Sugar (%)	Ash content (%)
Hot H_2O pre-extraction	87.7	9.64	4.4	1.0	6.8	0.76
H_2SO_4 (0.25%v/v)-pre- extraction	81.0	12.74	2.2	1.2	9.6	1.68
Na_2CO_3 (0.5%w/v as NaOH)-pre-extraction	88.9	14.55	6.7	1.3	6.4	5.4

Table 2. Pulping of bamboo chips

		Alkali charge (% as NaOH)	Screened yield (%)	Reject (%)	Total yield (%)	Overall yield (%)	Kappa number
Without pre-extraction	Kraft*	14	42.5	2.7	45.2	45.2	31.9
		16	44.0	0	44.0	44.0	28.1
		18	41.7	0	41.7	41.7	24.7
Hot-water pre-extraction	Soda	16	44.5	4.1	48.6	42.6	38.5
		18	44.1	0	44.1	38.7	30.1
		20	41.2	0	41.2	36.1	24.3
	Soda-AQ	16	44.6	0.5	45.1	39.6	22.0
		18	41.2	0	41.2	36.1	18.0
		20	40.4	0	40.4	35.4	16.7
H_2SO_4 pre-extraction	Soda	16	32.6	15.3	47.9	38.8	50.3
		18	38.4	1.8	40.2	32.6	44.6
		20	34.8	1.7	36.5	29.6	34.2
	Soda-AQ	16	33.0	11.6	44.6	36.1	30.0
		18	33.6	9.9	43.5	35.2	28.3
		20	37.0	0	37.0	30.0	20.0
Alkaline pre-extraction	Soda	16	46.4	0	46.4	41.2	36.7
		18	43.9	0	43.9	39.0	25.7
		20	42.0	0	42.0	37.3	22.1
	Soda-AQ	16	46.0	0	46.0	40.9	29.1
		18	44.7	0	44.7	39.7	18.5
		20	44.2	0	44.2	39.3	15.6

*Alkali charge as Na_2O

Pre-extraction conditions: Material to Liquor ratio-1:4, Time- 60 min at 170^oC, 0.25% H_2SO_4, in H_2SO_4 pre-extraction and 0.5% Na_2CO_3 in alkaline pre-extraction

Cooking conditions: Material to Liquor ratio-1:4, Time- 120 min at 170^{o}C

Table 3. Peroxyformic acid treatment of different bamboo pulps

	Kraft	Hot-water pre-extraction		Acid pre-extraction		Alkaline pre-extraction	
		Soda	Soda-AQ	Soda	Soda-AQ	Soda	Soda-AQ
Kappa number	18.4	18.6	16.4	27.2	18.1	11.7	9.7
kappa reduction (%)	35.5	38.2	8.9	39.0	36.0	54.4	47.6
Brightness (%)	25.9	24.8	26.6	23.6	30.6	32.6	34.2
Viscosity (mPa.s)	6.8	5.1	4.92	5.1	4.82	6.7	6.6

Table 4. Physical properties of bleached pulps of different pre-extracted bamboo chips

Properties	Kraft	Alkaline pre-extraction	
		Soda	Soda-AQ
Drainage resistance (^{o}SR)	41	38	40
Tensile index (N.m/g)	34.7	25.3	30.8
Tear index (mN.m^2/g)	6.8	7.9	7.2
Burst index (kPa.m^2/g)	2.8	1.9	1.7
Brightness (%)	76.3	76.3	77.4

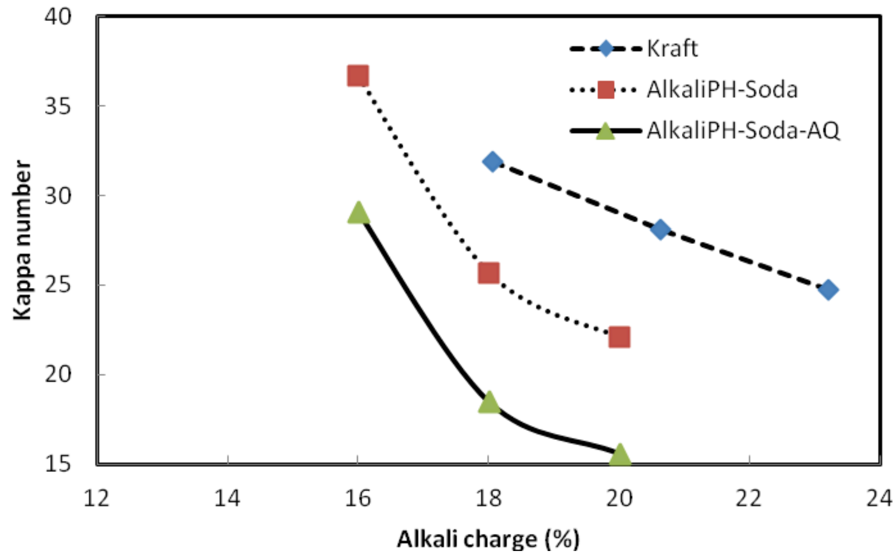

Fig. 1. Effect of alkaline pre-extraction on the delignification of bamboo chips

Pre-extraction conditions: Material to Liquor ratio-1:4, Time- 60 min at 170°C, 0.25% H_2SO_4, in H_2SO_4 pre-extraction and 0.5% Na_2CO_3 in alkaline pre-extraction

Cooking conditions: Material to Liquor ratio-1:4, Time- 120 min at 170°C

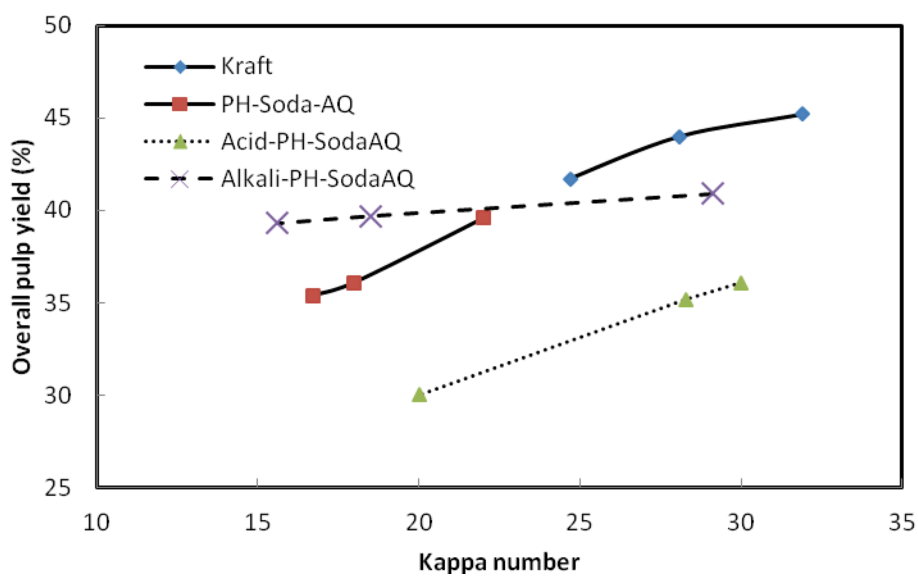

Fig. 2. Effect pre-extraction processes on the pulp yield of bamboo chips

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