Journal of Tropical Forest Research 3 (2) : 29-43 (2019)

Original article

Biomass Cane Bagasse: An Alternative Raw Material for Dissolving Pulp Production

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Received: Jun 17, 2019 Revised: Jul 25, 2019 Accepted: Jul 26, 2019

ABSTRACT

In this study, the potential of biomass cane bagasse in the production of dissolving pulp was evaluated. The chemical composition of whole bagasse was analyzed, and then prehydrolyzed with different acid pretreatments and subsequently pulped via the soda process. The unbleached soda pulps were fully bleached using elemental chlorine-free bleaching with the D_0EpD_1 sequence and their dissolving pulp properties were evaluated. The biomass cane bagasse contained 49.0% cellulose, 26.4% pentosan, 17.7% acid-insoluble lignin, 1.9% acid-soluble lignin, 4.5% acetone extractives, and 1.8% ash. The acid pretreatment at 0.2–0.3% (w/v) acid and at a temperature of 150 °C for 30 min was sufficient for the removal of pentosan from the biomass cane bagasse and achieved a low pentosan content and kappa number for the unbleached soda pulps. The results showed that biomass cane bagasse produced high-purity bleached pulps containing a high alpha-cellulose content (96.0–96.3%), high pulp viscosity (615–723 mL/g), high brightness (91.8–92.9% ISO), high R10 content (93.3–95.0%), and high R18 content (95.8–98.9%) but low pentosan (2.0–3.3%), acetone extractives (0.08–0.10%), and ash (0.12–0.14%) content. Hence, biomass cane bagasse can be used as a raw material for preparing standard and high-grade dissolving pulp, and other bio-products.

Keywords: Acid pretreatment, Biomass cane bagasse, Dissolving pulp, Elemental chlorine-free bleaching, Soda pulping

INTRODUCTION

Dissolving pulp, also known as cellulosic fiber, is gaining worldwide attention as they are typically used in the production of textile fibers (viscose and lyocell) or other high value-added cellulose esters and ethers (He *et al*., 2017). In addition, dissolving pulp can also be used as a basic raw material in the production of nano- and microcrystalline cellulose (Chen *et al*., 2016). Dissolving pulp is characterized by its high-purity cellulose content (90–99%) with a low amount of hemicellulose (<4%) and traces of other components (Sixta, 2006; He *et al*., 2017; Kumar and Christopher, 2017). This pulp has distinct properties such as high brightness, low degree of polymerization, and uniform molecular-weight distribution (Bajpai, 2012; Dien *et al*., 2015).

The production of dissolving pulp has substantially increased worldwide, from 5.6 million tonnes in 2013 to 7.5 million tonnes in 2015. More than 70% of the produced dissolving pulp is used to manufacture viscose staple (Kumar and Christopher, 2017; Yang *et al*., 2018). Generally, about 85% of the dissolving pulp is produced from softwood and hardwood, whereas 10% is produced from cotton linter (Chen *et al*., 2016). In recent years, non-wood materials have received increased attention for use in pulp and paper industry and as feedstocks for a bio-based economy, owing to diminishing forest resources and an increase in the consumption of lignocellulosic products (Yuan *et al*., 2016). Non-wood plants offer several advantages such as moderate irrigation and fertilization requirements during a short growth cycle and reduced energy and chemical consumption during pulping, owing to their low lignin content (Dien *et al*., 2015). Several studies focusing on the use of non-wood raw materials, including corn stalk, bamboo, sugar cane bagasse, and jute, for the production of dissolving pulp, have been reported previously (Behin *et al*., 2008; Luo *et al*., 2014; Andrade and Colodette, 2014; Maryanar, 2017; Matin *et al*., 2015). In Thailand, new cane species have been hybridized for specific purposes, e.g., sugar cane, energy cane, and biomass cane (BC), through a collaboration between the National Science and Technology Development Agency and the Department of Agronomy, Faculty of Agriculture, Kasetsart University (Kamphaeng Saen campus), Thailand. BC, a hybrid of sugar cane (*Saccharum officinarum*) and wild sugar cane (*Saccharum spontaneum*), has been developed with a high fiber content and can be used both as an animal feed and as a raw material in the energy and fiber industry (National Science and Technology Development Agency, 2012; 2016). BC is a perennial grass that can grow up to five meters in height, with spreading rhizomatous roots (CAB International, 2018). Compared to sugar cane, BC has a smaller stalk with a higher percentage of fiber content (15-20% in BC compared to 10-14% in sugar cane), and lower

sugar content (brix percentage of cane juice is 8-10% for BC compared to 23-25% for sugar cane). It has a relatively higher tolerance to drought and flooding than sugar cane and can also be planted in various areas (National Science and Technology Development Agency, 2012).

The aim of this work was to characterize and evaluate the potential of biomass cane bagasse (BCB) in the production of dissolving-grade pulps.

MATERIALS AND METHODS

Preparation of BCB sample

Samples of 10-month-old BC were collected from experimental plots maintained by the Department of Agronomy, Kasetsart University (Kamphaeng Saen campus), Nakhon Prathom province, Thailand. The stalk height and diameter of the BC was approximately 1.9 m and 1.5 cm, respectively. After harvesting, the BC leaves and tops were removed from the stalks, which were then crushed to extract juice from the fibrous matter known as bagasse. The BCB was chopped into pieces which were 3–4 cm long. The chopped BCB was washed with tap water to remove its sugar and then centrifuged and stored in a refrigerator at 4 °C. A 200-g chopped BCB sample was randomly selected, air-dried, ground in a Wiley Mill (Thomas Scientific, USA), and screened in a vibratory sieving apparatus to obtain 40–60 mesh fractions for further analysis of its chemical composition.

Analysis of chemical composition

The procedure outlined by the Technical Association of the Pulp and Paper Industry (2012) was used for the chemical composition analysis: preparation of sample for chemical analysis (TAPPI T 264 cm-07), pentosan (TAPPI T 223 cm-10), acid-insoluble lignin (TAPPI T 222 om-11), acid-soluble lignin (TAPPI UM 250), acetone extractives (TAPPI T 280 pm-99), and ash (TAPPI T 211 om-07). The cellulose content was determined according to Updegraff method (Updegraff, 1969).

Acid pretreatment

Prior to soda pulping, the BCB was treated with 0–0.3% (w/v) sulfuric acid in a ratio of liquor:BCB of 5:1 (v/w) in an electrical digester with a capacity of 5 L, at a temperature of 150 °C for 30 min. After pretreatment, the pH of the prehydrolysate was measured. The pretreated BCB was washed with deionized water to remove the residual acid and dissolved substances. The yield of pretreated BCB was determined and expressed as a percentage of pretreated BCB obtained from the original BCB (based on oven-dried weight). The pentosan content of pretreated BCB was determined according to TAPPI T 223 cm-10 (Technical Association of the Pulp and Paper Industry, 2012).

Soda pulping

The soda pulping of untreated (as control) and pretreated BCB was carried out

in an electrical digester with a capacity of 5 L under the following conditions: 16% alkali charge (as Na_2O), maximum temperature of 165 °C, time to maximum temperature of 60 min, H-factor of 1,300, and a ratio of liquor:pretreated BCB of 5:1 (v/w). After cooking, the pulp was washed with water and disintegrated using a pulp disintegrator (Lorentzen & Wettre, Sweden). The pulp was screened in a pulp screener (Kumagai Riki Kogyo, Japan) with a 0.15-mm slot plate, and then centrifuged and stored in a refrigerator at 4 °C. The pulp yield was determined and expressed as a percentage of unbleached pulp obtained from the original BCB (based on oven-dried weight). The kappa number and brightness of the unbleached soda pulp was determined according to the procedures of the International Organization for Standardization (2011), ISO 302:2004 and ISO 3688:1999, respectively. The pentosan content was determined according to TAPPI T 223 cm-10 (Technical Association of the Pulp and Paper Industry, 2012).

Elemental chlorine-free bleaching

The unbleached soda pulp was fully bleached in an elemental chlorine-free (ECF) bleaching sequence to obtain a target brightness of more than 90% ISO, including chlorine dioxide delignification (D_0), alkaline extraction with addition of hydrogen peroxide (Ep) and chlorine dioxide bleaching (D_1). The bleaching conditions are shown in Table 1. After each bleaching stage, the pulp samples were washed with deionized water. The properties of bleached pulp was determined according to the procedures of the International Organization for Standardization (2011) for pulp viscosity (ISO 5351:2010), alkali resistance (ISO 699:1982), and brightness (ISO 3688:1999), and according to the procedures of the Technical Association of the Pulp and Paper Industry (2012) for pentosan (TAPPI T 223 cm-10), alpha cellulose (TAPPI T 203 cm-09), acetone extractives (TAPPI T 280 pm-99), and ash (TAPPI T 211 om-07).

Table 1 Bleaching conditions of unbleached soda pulp.

Parameter/Stage	D_0	Ep	D_1
ClO_2 (%)	1.5	–	0.5
H_2O_2 (%)	–	1.5	–
NaOH (%)	–	1.5	–
Consistency (%)	10	10	10
Temperature (°C)	70	70	70
Time (min)	60	90	120
pH	2.5–3.0	10.5–11.0	3.5–4.0

Remarks: D_0 = chlorine dioxide delignification; Ep = alkaline extraction with addition of hydrogen peroxide; D_1 = chlorine dioxide bleaching; – = not applicable.

RESULTS AND DISCUSSION

Chemical composition of BCB

The chemical composition of BCB and sugar cane bagasse is indicated in Table 2. The results show that BCB contained a significantly higher percentage of cellulose and pentosan when compared to the whole bagasse derived from sugar cane (Andrade and Colodette, 2014). However, the total lignin, extractives, and ash content was lower. These data illustrate both the advantage and challenge of using BCB as an alternative raw material in dissolving-pulp applications. With respect to the challenges, for example, a high pentosan content is undesirable in a dissolving pulp because the presence of pentosan causes several problems in the downstream conversion process and affects the final quality of cellulose derivatives (Zhao *et al*., 2017). On the other hand, a higher cellulose content should result in a higher chemical pulp yield. However, the chemical composition of bagasse is dependent on the soil, climate, and length of the growing season, among other factors (Hamilton and Leopold, 1987).

Table 2 The chemical composition of BCB and sugar cane bagasse.

Composition	BCB	Sugar cane bagasse (Andrade and Colodette, 2014)
Cellulose (%)	49.0±0.9	41.6
Pentosan (%)	26.4±1.0	18.9
Total lignin (%)	19.6±0.1	22.5
- Acid insoluble lignin (%)	17.7±0.1	21.3
- Acid soluble lignin (%)	1.9±0.0	1.2
Extractives (%)	4.5±0.1 (acetone extractives)	8.5 (total extractives)
Ash (%)	1.8±0.1	4.0

Acid pretreatment of BCB

The pH of the acid prehydrolysate decreased from 3.8 to 1.2 after the addition of acid and an increase in the dosage. Correspondingly, the resulting yield of pretreated BCB decreased with an increase in the dosages of acid, and substantially fell from 84.9% to 62.5% after a 0.3%-acid hydrolysis (Figure 1). It was speculated that this decrease in the pretreated-BCB yield at higher acid concentrations was mainly caused by a chemical reaction of the acid, leading to the complete removal of the hemicellulose fraction in the BCB. This result is similar to several previous research studies that reported on the effects of acid hydrolysis on the chemical composition of various lignocellulosic materials (Ibrahim *et al*., 1996; Li *et al*., 2010; Dien *et al*., 2015; Li *et al*., 2015).

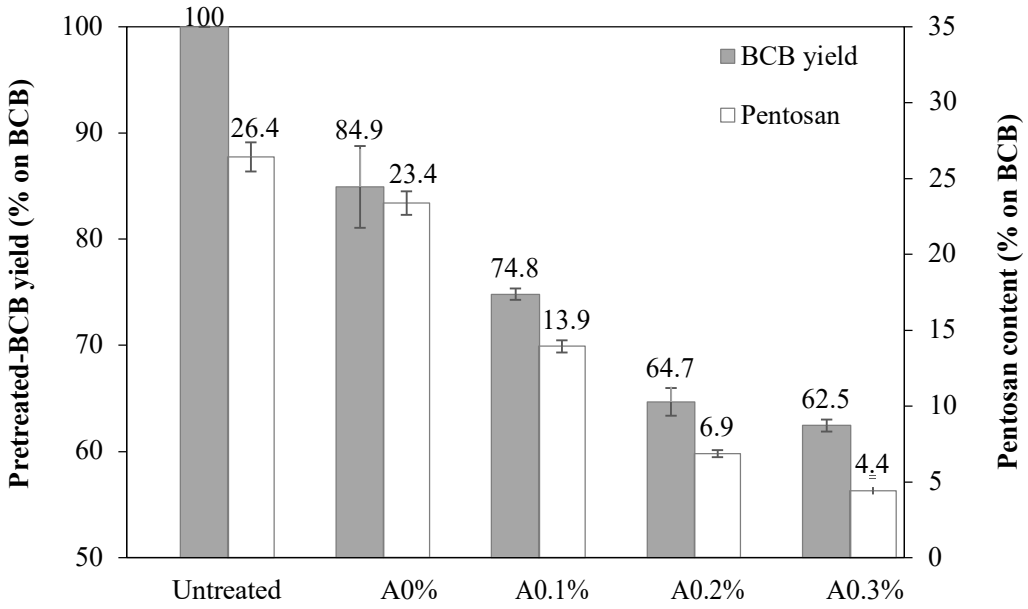

Figure 1 Effect of acid pretreatment on the yield and pentosan content of pretreated biomass cane bagasse (untreated = no pretreatment; A0% = no acid; A0.1%, A0.2%, and A0.3% = pretreatment with 0.1%, 0.2%, and 0.3% acid, respectively).

The effect of acid pretreatment on the pentosan content in the pretreated BCB is illustrated in Figure 1. The pentosan fraction was mostly removed after the acid pretreatment, as indicated by a significant reduction in the pentosan content of the 0.3%-acid-pretreated sample (approximately 82%) compared to that of the untreated control. Low pentosan removal was observed when prehydrolysis was conducted without the addition of acid (A0%), whereas the pentosan content dropped dramatically from 26.4% to 6.9% and 4.4% after pretreatment with 0.2% and 0.3% acid. Most of the pentosan fraction was removed during the acid hydrolysis at minimum values of 0.2% acid or higher.

Lehto and Alén (2014) reported that hydronium ions originating from the organic acids are formed in the acid prehydrolysis and they catalyze the hydrolysis of the linkages between hemicellulose and lignin as well as the hydrolysis of the glycosidic bonds of the carbohydrate chains, resulting in a chain degradation and reduction in the molar masses. The hydrolysis process affects the physical structures and conformation of the carbohydrate components of plant biomass. Owing to the amorphous nature, different ring structures and carbon atom configurations of hemicellulose degrade faster under acidic conditions than those of cellulose. Hence, the solid residue from the dilute acid hydrolysis mainly contains cellulose and lignin.

Soda pulping

In this study, the acid-pretreated BCB was subsequently subjected to the soda pulping

step under operationally constant conditions. The results plotted in Figure 2 show that the pulp yield of the unbleached soda pulp decreased progressively with an acid pretreatment ranging from 0.1% to 0.3% acid, due to the removal of pulp components, mainly hemicellulose and lignin. Pulp yield obtained from 0.1–0.3% acid pretreatments was between 31–39%, whereas around 46% and 51% pulp was recovered from the untreated control and sample treated without acid hydrolysis, respectively. The hemicellulose (mainly xylan) removal by an acid pretreatment prior to soda pulping reduced the pulping yield significantly due to a very low molecular weight and high solubility of xylan after acid pretreatment and the more sensitive to alkaline pulping of cellulose after partially removing of fibrils and opening the cellulose to alkali attack with a subsequent reduction in the molecular weight (Colodette *et al*., 2011; Cordeiro *et al*., 2013; Li *et al*., 2015).

Figure 2 also demonstrates that the total yield from the pulp samples was greater for the pulp with a high kappa number. Furthermore, the acid pretreatment step prior to the soda pulping process reduced up to 52% more total lignin in the pulp samples compared to that of the untreated control, as indicated by a decrease in the kappa number of the acid-pretreated pulps. This significant decrease in the lignin fraction in the soda-pulping samples was caused by partial depolymerization under the acidic conditions, resulting in an enhanced delignification reaction and an improvement in the permeability of the cell wall by the alkali (Sixta, 2006). Lehto and Alén (2014) found that removing hemicellulose and the associated degradation products during prehydrolysis prior to alkali cooking resulted in reducing the cost of the pulping and bleaching chemicals, a reduced cooking time, and a lower energy demand.

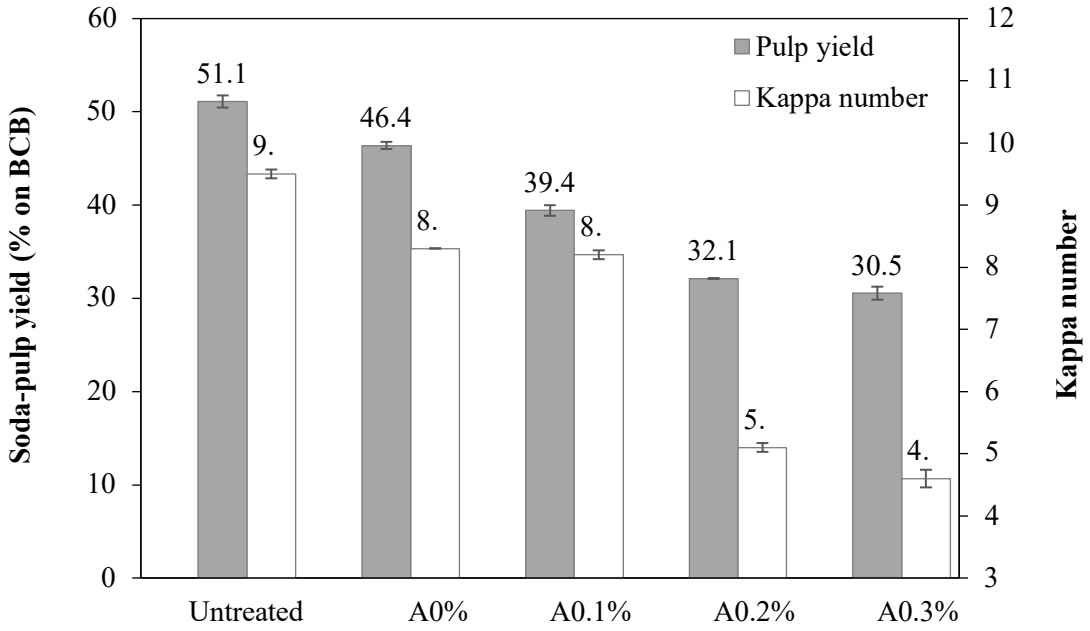

Figure 2 Effect of acid pretreatment on pulp yield and kappa number of unbleached soda pulp (untreated = no pretreatment; A0% = no acid; A0.1%, A0.2%, and A0.3% = pretreatment with 0.1%, 0.2%, and 0.3% acid, respectively).

The effect of acid concentration on the residual hemicellulose content of the unbleached soda pulp obtained from pretreated BCB is shown in Table 3. The residual pentosan content decreased from 32.7% to 2.0% of the pulp with an increase in the acid dosage. The content of pentosan decreased rapidly to 10.0% upon the addition of only 0.1% acid, which was able to remove up to 69% of the pentosan. Increasing the acid concentration to 0.2% and 0.3% resulted in a slow drop in the pentosan content to 3.2% and 2.0 %, respectively, indicating that the pentosan removal was enhanced by up to 90% and 94%, respectively. Considering only the residual hemicellulose content, the acid hydrolysis pretreatment was more efficient due to a greater extent of cleavage of the hydrogen and covalent bonds of the lignocellulosic structure (Lehto and Alén, 2014).

Table 3 Pentosan content of the unbleached soda pulp from BCB.

Parameter	Untreated	A0%	A0.1%	A0.2%	A0.3%
Pentosan (%)	32.7 ± 0.5	26.9 ± 0.0	10.0 ± 0.2	3.2 ± 0.0	2.0 ± 0.0

Remarks: untreated = no pretreatment; A0% = no acid; A0.1%, A0.2%, and A0.3% = pretreatment with 0.1%, 0.2%, and 0.3% acid, respectively.

Properties of dissolving pulp

The properties of the bleached soda pulp (so called "dissolving pulp") are shown in Table 4.

Brightness

The commercially available dissolving pulps used in the manufacturing of cellulose derivatives such as viscose, lyocell, and acetate pulp require high and acceptable levels of brightness (>90% ISO), whereas the technical-grade cellulose derivatives used as raw material in the textiles, paper, drilling muds, and ceramics industry do not (Sixta, 2006). As expected, the acid-pretreated pulps were easily bleached by short sequences of bleaching, as evidenced by a higher brightness compared to that of the untreated control and pulp sample treated without acid hydrolysis. In addition, after the bleaching process, the pulp brightness increased in conformity with the amount of acid used in the prehydrolysis step. The resulting pulp obtained from the bleaching step showed a higher degree of brightness of up to 92.9% ISO, which is acceptable for further use in dissolving applications. The bleachability of the xylan-depleted material was improved by the hydrolysis process, as indicated by the lower pulp kappa numbers of the pulp entering the bleaching plant. Furthermore, the lower kappa numbers resulted in a decrease in the total active chlorine demand (less chlorine dioxide), which in turn led to a reduction in the bleaching chemical cost and the bleaching effluents (Colodette *et al*., 2011; Yao *et al*., 2017).

Table 4 Properties of bleached soda pulp from BCB.

Parameter	Untreated	A0%	A0.1%	A0.2%	A0.3%
Brightness (% ISO)	89.9 ± 0.1	90.0 ± 0.2	91.8 ± 0.1	92.9± 0.1	92.7± 0.1
Pentosan (%)	21.7 ± 0.4	20.6 ± 0.4	11.9 ± 0.4	3.3 ± 0.1	2.0 ± 0.0
Alpha cellulose (%)	84.9 ± 0.1	85.9 ± 0.4	92.8 ± 0.0	96.0 ± 0.0	96.3 ± 0.0
Pulp viscosity (mL/g)	1002 ± 4	965 ± 2	1054 ± 11	723 ± 1	615 ± 1
R10 (%)	80.1 ± 1.2	85.0 ± 0.1	91.5 ± 0.0	93.3 ± 0.0	95.0 ± 0.0
R18 (%)	85.6 ± 0.0	88.5 ± 0.0	93.8 ± 0.1	95.8 ± 0.1	98.9 ± 0.1
Acetone extractives (%)	0.08 ± 0.01	0.10 ± 0.00	0.10 ± 0.00	0.10 ± 0.01	0.08 ± 0.01
Ash (%)	0.17±0.00	0.15±0.01	0.12±0.00	0.14±0.00	0.12±0.01

Remarks: untreated = no pretreatment; A0% = no acid; A0.1%, A0.2%, and A0.3% = pretreatment with 0.1%, 0.2%, and 0.3% acid, respectively.

Pentosan content

The efficiency of cellulose conversion into specific derivatives depends on the hemicellulose content of the dissolving pulp (Andrade and Colodette, 2014). A typical viscose-grade hardwood pulp comprises of up to 5% residual xylan; however, the residual xylan content in acetate-grade pulp should not

exceed 2% (Sixta, 2006). After bleaching, the pentosan content of the bleached pulp from untreated sample and the sample treated without acid was lower than their unbleached pulp counterparts; the residual pentosan content decreased from 32.7% and 26.9% (in the unbleached pulp) to 21.7% and 20.6%, respectively. On the other hand, the residual pentosan content of the bleached pulp of the acid-pretreated samples was below 10%. This result is in agreement with that of Luo *et al.* (2014), who reported that under constant alkali pulping and bleaching conditions, the xylan content in the bleached pulp could be successfully reduced by substantially increasing the intensity of hot water extraction or by adding a small amount of mineral acid as a hydrolysis catalyst. However, the result from Table 4 shows that the ECF bleaching had a minor positive effect on the pentosan removal from the unbleached pulp containing a small amount of pentosan.

Alpha-cellulose content

The results in Table 3 show that the alpha-cellulose content of the bleached pulp increases when a higher dosage of acid is used in the pretreatment stage. A higher pulp purity was obtained using conditions of 0.2% acid and 0.3% acid, which produced an alpha-cellulose content of 96.0% and 96.3%, respectively. These results are similar to those obtained for rayon-grade dissolving pulp (94–96%) from prehydrolysis kraft pulp (Chen *et al*., 2014). In addition, the values obtained in this study were higher than those for the laboratory dissolving-pulp produced from other lignocellulosic samples, e.g., sugar cane bagasse, jute stick, birch wood, and bamboo willow (Andrade and Colodette, 2014; Luo *et al*., 2014; Wu *et al*., 2018).

Pulp viscosity

As the level of alpha cellulose increases, the pulp viscosity decreases, as shown in Table 3. The pulp viscosity decreased with an increase in the prehydrolysis intensity. This decrease was due to the cleavage of the glycosidic bonds in the cellulose chains and the consequent reduction in the degree of polymerization of cellulose. This resulted in the removal of a large amount of hemicellulose from the fiber cell walls, enhancing the exposure of the cellulose chains and promoting their hydrolytic degradation (Borrega *et al*., 2017). However, a decrease in the viscosity of the bleached pulp was still higher than that of a commercial viscose-grade pulp that has a viscosity in the range of 400–500 mL/g (Borrega *et al*., 2018). At the highest prehydrolysis intensity (0.3% acid), the bleached pulp had a higher viscosity than the commercially used dissolving-grade pulp produced from eucalypts (Vehma *et al*., 2013). Furthermore, the pulp produced in this study is comparable to the special dissolving-pulp produced from eucalypts (pulp viscosity 500–700 mL/g), which can be used to make cellulose acetate, cellulose ether, and nitrocellulose (Manhaes *et al*., 2001).

Alkali resistance

The alkali resistance is a measure of the cellulose content (total cellulose: R18, long-chain cellulose: R10) and is generally used to characterize the degree of purity of dissolving pulp as an indicator of the residual xylan and degraded cellulose content in the pulp (Sixta, 2006; Testova *et al*., 2014). Table 3 shows the effect of acid pretreatment on the alkali resistance of the bleached pulp. Both the R10 and R18 alkali resistances showed an increase with the intensity of prehydrolysis. The bleached pulp of untreated BCB and of that pretreated without acid dosage had a lower alkali resistance; this may be due to the higher pentosan content and degraded cellulose. Intense prehydrolysis (0.3% acid) produced higher values of R10 and R18 (95.0% and 98.9%, respectively), which were greater than those of commercial dissolving pulp (Vehma *et al*., 2013).

Acetone extractives and ash content

Table 3 shows that the content of acetone extractives in the bleached pulp was in the range of 0.08–0.10%, based on the oven-dried pulp. Compared to commercial dissolving-pulp produced from eucalypts, the bleached pulp from BCB had lower levels of extractives than the standard and special grades, and was comparable to the high grade (Vehma *et al*., 2013). The extractive content of a fully bleached pulp depends on many parameters, with the wood species and the pulping and bleaching processes being the most important influencing factors. A large amount of extractives in the pulp can cause severe problems along the process chain, e.g., precipitation (especially due to abrupt changes from alkaline to acidic pH), haze in viscose, clogging of the spinnerets, and yellowing of the yarn (Sixta, 2006).

For the production of dissolving pulp from annual plants, it is important to remove the inorganic compounds as the presence of certain inorganic compounds such as silicates, Ca salts, and metal ions (Fe, Mn, Co, etc.) may impair the filterability and spinnability of a cellulose spinning dope (viscose- or lyocell-type fibers) (Sixta, 2006). In this study, it was found that the ash content of the bleached pulp showed a decreasing trend (from 0.17% to 0.12%) when the prehydrolysis was intensified (Table 3). The ash content of the bleached BCB pulp was lower than that of laboratory dissolving-pulp produced from sugar cane bagasse, corn stalk, aspen wood, and green bamboo, but slightly higher than that of the pulp produced from bamboo willow (Andrade and Colodette, 2014; Luo *et al*., 2014; Wu *et al*., 2018).

CONCLUSIONS

Dissolving-grade pulp can be produced using whole bagasse from BC, via acid prehydrolysis, soda pulping, and subsequent bleaching with the D_0EpD_1 sequence.

The bleached pulps produced using acid pretreatment conditions of 0.2% and 0.3%

acid were considered satisfactory and were produced in sufficient qualities for the next steps (preparation of cellulose derivatives) because these pulps had high levels of desirable properties (brightness, pulp viscosity, alpha cellulose, and alkali resistance) and low content of undesirable contaminants (pentosan, acetone extractives, and ash).

The bleached pulp with acid pretreatment conditions of 0.2% acid was comparable to the standard and special grades of commercial eucalypt dissolving-pulp. It is therefore a suitable specification for a viscose grade.

The bleached pulp with acid pretreatment conditions of 0.3% acid had the highest purity and was comparable to high-grade commercial eucalypt dissolving-pulp, except for a trace residual ash content that slightly exceeded the acceptable range. Nonetheless, it complies with the requirements for a typical acetate-grade pulp.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge Assoc. Prof. Dr. Prasert Chatwachirawong, Department of Agronomy, Kasetsart University (Kamphaengsaen campus) for providing biomass cane, and thanks are also given to Environment Pulp and Paper Co., Ltd., Nakhon Sawan province for providing bleaching agent (chlorine dioxide).

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