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Dissolving pulp from jute stick

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ABSTRACT

Jute stick is woody portion of jute plant, which remain as leftover after extracting bast fibre. Presently, it is being used for fencing in the rural area. In this investigation, biorefinery concept was initiated in producing dissolving pulp from jute stick by pre-hydrolysis kraft process. At 170 °C for 1 h of pre-hydrolysis, 70% of hemicelluloses was dissolved with negligible loss of α-cellulose. At this condition, 75% of dissolved sugars in the pre-hydrolysis liquor were in the oligomeric form. The pre-hydrolysed jute stick was subsequently pulped by kraft process with the variation of active alkali. The pulp yield was 36.2% with kappa number 18.5 at the conditions of 16% active alkali for 2 h of cooking at 170 °C. Final pulp was produced with 92% α-cellulose and 89% brightness after $D_0E_pD_1EpD_1$ bleaching. The produced dissolving pulp can be used in rayon production.

1. Introduction

Jute stick is the woody portion of jute plants remain as leftover after extracting fibre (Pandey, Ghosh, & Dey, 1995). It constitutes about 2.5 times of extracted fibre. Jute sticks are used as fuel wood, fencing in the rural area. Jute stick contains considerable amount of cellulose with lignin and hemicelluloses. Therefore, it is a potential source for a biorefinery.

There has been a scarcity of lignocellulosic materials for production of pulp and paper products in Bangladesh. Because of depletion of forest resources interest is growing on alternative lignocelluloses. Jute stick falls under the category of hardwood and the annual production of jute sticks in the country is around 3.0 million tonnes (IJSG, 2014, http://jute.org/statistics_search.php).

Many studies have been carried out on utilization of jute stick. Its lignin, hemicelluloses and cellulose were used in different biobased products. Roy, Sardar & Sen (1989) studied on lignin isolated from spent soda liquor of jute stick pulping process, which was used for the preparation of lignin phenol formaldehyde resins. It was observed that phenol in phenol formaldehyde resin could be replaced up to a maximum of 50% by jute stick soda lignin. The feasibility of utilizing jute sticks in terms of quality, availability and economics of paper manufacture in the paper industry was studied by Das (1980) and showed economically viable for Indian paper pulp plant. Das, Day & Pandey (1987) prepared a variety of boards from jute stick, usable purpose such as in furniture, table tops, building materials, partitioning materials, false ceilings, etc. Day, Chattopadhay, Ghosh & Bhaduri (2007) used jute stick for producing high purity cellulose pulp by pre-hydrolysis followed by Kraft process and obtained 93% cellulose. Subsequently, this CMC and MCC were produced from this pulp. At suitable condition jute stick was carbonized to give, in good yield, a light-weight, smokeless, charcoal in chip form with high fixed-carbon content (Banerjee & Mathew, 1985).

Considering the depleting of petroleum resources and global warming importance have been increased towards efficient biorefinery technology to convert lignocelluloses to biofuels, biochemicals (Cheng & Zhu, 2009). Dissolving pulp production process well fit with the biorefinery, where lignocelluloses are fractionates to cellulose pulp and hemicelluloses (Saeed et al., 2012). Dissolving pulp is of also critical importance as a sustainable feedstock for manufacturing various materials such as, cellulose derivatives, nano-/micro-cellulose, which are alternatives to many petroleum-based materials. In recent years, the production of dissolving pulp experienced a significant increase owing to the strong growth of market demand. Dissolving pulp has special properties, such as a high level of purity, uniform molecular-weight distribution and the reactivity and accessibility of cellulose to chemicals (Krässig, 1993). To achieve maximum purity of pulp single or multiple pretreatment of auto hydrolysis, acid hydrolysis, mechanical and swelling treatments, enzyme treatment etc. need to be done (Engström,

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Monica, & Henriksson, 2006). The pre-hydrolysis kraft process is a well-known process for dissolving pulp production (Sixta, 2006). The auto-hydrolysis of wood prior to pulping is carried out in order to dissolve hemicelluloses. During auto-hydrolysis of wood, chips acetic acid is formed directly from the cleavage of acetyl groups of the xylan backbone, which easily impregnates and hydrolysis uniformly. The fact that hemicelluloses are considerably easier to hydrolyse than cellulose, which means selectively removing hemicelluloses. The presence of sulphuric acid in the pre-hydrolysis process enhances the removal of hemicelluloses from the lignocellulosic materials (Jahan, 2009; Liu et al., 2009). Acid pre-hydrolysis is also carried for total hydrolysis of lignocellulosic biomass to sugar monomers, which is a novel source of chemicals (Grethlein & Converse, 1991). In mechanical pulping acid prehydrolysis is applied in order to soften the wood structure and therefore save refining energy (Kenealy, Horn & Houtman, 2007). But it was observed that the acid pre-hydrolysis attacked cellulose in the dissolving pulp production process (Jahan, 2009). Considering the above discussion, hot water pre-hydrolysis is chosen for dissolving pulp production. In the pre-hydrolysis process most of the hemicelluloses are dissolved, which can be used in biofuel and biochemicals. The α-cellulose, hemicelluloses and lignin content in jute stick are similar to hardwood. Therefore, it can be a good source for dissolving pulp-based biorefinery.

Jute stick is a very important lignocellulosic raw material in Bangladesh. But no study has been carried out on jute stick for dissolving pulp using biorefinery concept. Therefore, the objective of this study was to pre-hydrolyse jute stick by varying time and temperature with respect to maximum dissolution of hemicelluloses and minimum attack on cellulose. Subsequently, optimized pre-hydrolysed jute stick was cooked and bleached for producing dissolving pulp. The pre-hydrolysis liquor (PHL) of jute stick was also assessed to produce biofuels and biochemicals.

2. Materials and methods

2.1. Raw materials

Jute stick was collected from the Jute Research Institute, Dhaka and cut to 2–3 cm in length. After determination of the moisture content, air-dried raw material equivalent to 200 g o.d. (oven dried) material was weighed separately in polyethylene bags for subsequent pre-hydrolysis and cooking experiments. Jute stick contains about 39% α-cellulose, 18% hemicelluloses and 25% lignin with other minor ingredients.

2.2. Pre-hydrolysis

Pre-hydrolysis was carried out in an electrically heated digester of 5 L capacity. The digester was rotated at 1 rpm. Water pre-hydrolysis was carried out at 150, 160 and 170 °C for 60, 90 and 120 min. The raw material to liquor ratio was 1:5 (g/mL). The time required to raise max temperature from room temperature (30 °C) was 35 min. After completing pre-hydrolysis, pressure was released by venting the valve and the liquor was separated from the solid mass by filtration and kept in refrigerator for analysis.

2.3. Solid contents

The total solid content in the PHL was determined gravimetrically by drying 10 ml sample at 105 °C till to constant weight.

2.4. Lignin analysis

The dissolved lignin in the pre-hydrolysate was measured based on the UV/vis spectrometric method at wavelength 205 nm (TAPPI UM 250).

2.5. Sugar analysis

PHL was filtered to remove any solid particle with filter paper. A vial containing 1 mL of the PHL and 4 mL 4 N sulfuric acid was sealed and autoclaved at 121 °C for 60 min. Component sugars and organic acids were analyzed by high-performance liquid chromatography (HPLC) equipped with refractive index and UV detection (Shimadzu, Columbia, MD), using the Aminex HPX-87H column (Bio-Rad, Hercules, CA). The column was operated with a 5 mM sulfuric acid mobile phase at a flow rate of 0.6 mL/min and oven temperature of 60 °C. Samples were filtered through 0.22 μm syringe filters prior to injection. The sugar contents in oligomeric form in the pre-hydrolysis liquor were calculated from the difference of the monomeric sugar contents between pre and post hydrolysis PHL.

2.6. Pulping

Pulping of pre-hydrolysed jute stick (at 160 °C for 90 min) by the kraft process was done in the same digester as in pre-hydrolysis under following identical cooking conditions. These were:

- Fibre to liquor ratio: 1:5 g/mL.
- Active alkali charge: 14, 16, 18% on pre-hydrolysed residue.
- Sulphidity: 27%
- Temperature: 170 °C.
- Cooking time: 120 min at maximum temperature.

2.7. $D_0EpD_1EpD_2$ bleaching

Pulps were bleached by D_0EpD_1 bleaching sequences in plastic bag. In the first stage (D_0) of $D_0EpD_1EpD_2$ bleaching sequences ClO_2 charge was 2%. The initial pH was adjusted to 2.5 by adding dilute H_2SO_4. In the alkaline extraction stage, NaOH and H_2O_2 charge were 2% and 0.5% (on o.d. pulp), respectively. The temperature was 70 °C for 60 min and pulp consistency was 10%. In the D_1 stage, the end pH was 4. The ClO_2 charge in the D_1 was 1.0%. In the D_2 stage, ClO_2 charge was 0.5% and end pH was maintained to 6.0 by adding weak alkali.

2.8. Evaluation of pulps

Pulp tests were performed according to the Standard Methods of the Technical Association of the Pulp and Paper Industry (TAPPI, Atlanta, GA): kappa number (T 236 cm-85); brightness (T 452 om-92); viscosity (T 230 om-89), α-cellulose (T 203 om-88) and alkali solubility S_{10} and S_{18} (T 235 cm-85).

3. Results and discussion

3.1. Pre-hydrolysis

In this study jute stick was pre-hydrolysed with varying time and temperature to get maximum dissolution of hemicelluloses without affecting cellulose. Effect of pre-hydrolysis conditions on solid yield and the liquor composition from jute stick have been listed in Table 1. Solid yield was in the range of 72–89% depending of time and temperature. A sharp decrease in solid yield was observed with rising temperature, which was attributed to both extractive removal and hemicelluloses solubilization (Yánez, Romaníi,

Table 1
Effect of pre-hydrolysis of jute stick on solid residue and PHL composition.

Temperature (°C)	Time (min)	Yield (%)	Pre-hydrolysis liquor				
			pH	Solid content (%)	Lignin (%)	Total sugar (%)	HAc (%)
150	60	88.6	4.8	8.2	0.500	5.78	0.82
150	90	88.1	4.6	9.2	0.706	6.85	0.78
150	120	88.0	4.5	10.0	0.765	7.21	0.87
160	60	83.6	4.5	11.3	0.614	8.00	1.05
160	90	81.3	4.4	12.5	0.745	8.95	1.23
160	120	80.2	4.3	12.9	0.823	9.03	1.22
170	60	76.9	4.4	15.7	1.000	11.32	1.51
170	90	74.8	4.3	16.3	1.283	11.65	1.39
170	120	72.3	4.3	17.1	1.399	12.47	1.67

HAc, acetic acid.

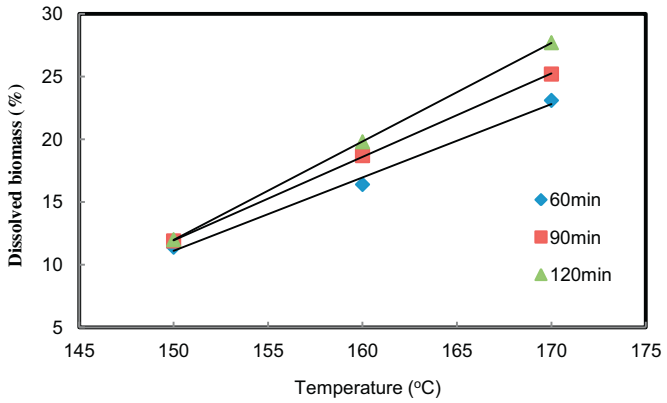

Fig. 1. Effect of temperature and time on the dissolution of biomass from the jute stick.

Garrote, Alonso, & Parajó, 2009). As shown in Fig. 1, the dissolution of biomass was increased linearly with temperature and its rate became slightly higher at longer time.

The dissolved biomass in the pre-hydrolysis at 150 °C was about 11–12% while the dissolved biomass increased to 23–27% at 170 °C (Table 1). As shown in Table 1, the sum of dissolved biomass and solid content in the pre-hydrolysis liquor was close to 96.8–98% at 150 °C. But the mass balance was decreased with increasing temperature. At 170 °C, the mass balance decreased to 89.4–92.6%, which was due to the formation furfural or other degraded products at higher temperature (Liu, Hu, Jahan, & Ni, 2013). Egüés, Sanchez, Mondragon, & Labidi (2012) showed that the liquors obtained at severity of 4–4.2, of 4.3–4.5 and 4.6–4.8, corresponding to 45, 60 and 75 min autohydrolysis time, contained less sugars compared with those obtained at severity of 3.8, 4.1 and 4.4 corresponding with 30 min of the hydrolysis time. The longer reaction time of the autohydrolysis process caused more degraded compounds. In dissolving pulp production process, pre-hydrolysis prior to pulping is carried out to remove hemicelluloses from the raw material. Therefore, pre-hydrolysis must be carried out at 160–170 °C to get dissolving pulp.

It is seen from Table 1 that hemicelluloses, acetic acid and lignin were present in the PHL. Total dissolved sugars in the PHL were 6–12% in our applied conditions. The major ingredient of hemicelluloses was xylose/xylan (C-5 sugars). Oligo-sugars was 75% of total sugar in the PHL (data are not shown) as observed in other study (Saeed et al., 2012). Commercial exploitation of these by-products will open a new window for dissolving pulp mill and will help to move towards biobased society. Furfural can be one of the possible products from the dissolved hemicelluloses in the PHL (Liu et al., 2013). The dissolved hemicellulose can also be used in producing lactic acid, propionic acid, succinic acid by saccharification followed by fermentation (Bustos, De la Torre, Moldes, Cruz, & Domínguez, 2007; Hughes, 2008). In the pre-hydrolysis process generated acetic acid from the acetyl group of hemicelluloses was 0.8–1.7%. As more hemicelluloses dissolved at higher temperature and time consequently generated more acetic acid. A typical industrial pre-hydrolysis liquor (PHL) contains about 1% acetic acid (Saeed et al., 2012), which can be separated and concentrated by reactive extraction with tri-n-octylamine using octanol as a diluents followed by sodium hydroxide back extraction (Ahsan, Jahan, & Ni, 2013). A considerable amount of lignin (0.5–1.3%) was dissolved in the pre-hydrolysis process. PHL lignin is higher in phenolic hydroxyl group (Jahan, Liu, Wang, Saeed, & Ni, 2012), therefore, it can be a good source for phenolic resin. The PHL lignin can be modified and produced carbon fibre (Sudo & Shimizu, 1992).

Table 2 also includes the composition of pre-hydroysed jute stick. The partial solubilization of the hemicellulose led to increased contents of cellulose. The cellulose content increased to 43.9% at 150 °C for 2 h pre-hydrolysis from 38.5% in the original raw materials, which was further increased to 51.1% at 170 °C for 2 h pre-hydrolysis. Yánez et al. (2009) showed that the glucan content increased with temperature up to reach a maximum value of 75.5 g/100 g oven dried pre-hydrolysis solids at 230 °C. The experimental data confirmed the selectivity of hemicelluloses solubilization with respect to cellulose degradation. The Klason lignin

Table 2
The composition of pre-hydrolysed jute stick.

Temperature (°C)	Time (min)	α-cellulose yield (%)	Pentosan yield (%)	Lignin yield (%)
150	60	38.9 (43.91)	17.9 (20.2)	19.5 (22.0)
150	90	39.1 (44.28)	17.5 (19.9)	20.4 (23.2)
150	120	38.7 (43.98)	17.4 (19.8)	20.0 (22.7)
160	60	38.0 (45.45)	13.8 (16.5)	19.5 (23.3)
160	90	37.5 (46.12)	12.5 (15.4)	19.3 (23.7)
160	120	37.4 (46.63)	11.6 (14.5)	18.7 (23.3)
170	60	37.5 (48.76)	8.2 (10.7)	18.3 (23.8)
170	90	37.2 (49.73)	6.5 (8.7)	17.5 (23.4)
170	120	37.3 (51.59)	5.2 (7.2)	17.0 (23.5)

Parenthesis indicates cellulose or pentosan or lignin content in pre-hydrolysis solid.
Yield = content in pre-hydrolysis solid × pre-hydrolysis yield/100.

Table 3
Effect of active alkali on the pulping of pre-hydrolysed jute stick.

Active alkali (%)	Pulp yield (%)	Kappa no.	α-Cellulose (%)	Pentosan (%)
14	36.4	21.0	86.0	6.90
16	35.2	18.5	86.6	7.24
18	33.2	15.2	86.4	7.57

Table 4
Effect of active alkali on the properties of dissolving pulp from jute stick.

Pulp obtained at alkali charge (%)	S_{18} (%)	S_{10} (%)	Brightness (%)	α-Cellulose (%)	Pentosan (%)	Viscosity (mPas)
14	7.52	10.8	88.60	91.0	6.7	3.54
16	7.08	11.19	88.33	92.3	6.9	3.24
18	7.12	13.00	89.53	92.4	6.5	3.05

S_{10}, solubility is 10% alkali; S_{18}, solubility in 18% alkali.

content of jute stick was 23.9%, which remain almost similar after pre-hydrolysis at 160–170 °C. The lignin yield from the starting raw material after pre-hydrolysis was 17–20% (Table 2). This result indicates that a part of lignin was dissolved in the pre-hydrolysis process. The hydrolytic lignin degradation occurred at higher temperature. The pentosan content in the starting jute stick was 18.3%, which was decreased continuously with increasing temperature. The pentosan yield was 5.2% at 170 °C for 2 h pre-hydrolysis, which indicated that 72% hemicelluloses was dissolved.

3.2. Pulping

Table 3 shows the effect of active alkali on the pulping of pre-hydrolysed jute stick. Pre-hydrolysed jute stick (at 160 °C for 90 min) was subsequently cooked by kraft process at varying alkali. The pulp yield was decreased with increasing alkali charge as expected. Pulp yield on over dried starting jute stick was 33–36%, which was close to dissolving pulp yield from bamboo by pre-hydrolysis kraft process (Ribas et al., 2012).

The kappa number was 21.0 at 14% alkali charge, which was decreased to 15.2 with increasing alkali charge to 18%. Required amount of alkali was less to get bleachable grade pulp in pre-hydrolysed jute stick. This can be explained by expose of lignin during pre-hydrolysis process. Ma, Huang, Chen & Chen (2011) showed that bamboo structure became loose in the pre-hydrolysis process due to the removal of hemicelluloses, which creates favourable environment for chemical penetration in the subsequent pulping process. It was reported in our previous study that drastic pre-hydrolysis conditions improved delignification significantly in subsequent cooking process (Jahan, Ahsan, Noori, & Quaiyyum, 2008, Jahan, Sultana, Rahman, & Quaiyyum, 2013). Therefore, alkali can penetrate easily inside the chips. The kappa number was reached to 18.5 at 16% alkali charge only. As shown in Table 3, the α-cellulose and pentosan content in the unbleached pulp was slightly increased with alkali charge due to the removal of lignin.

3.3. Bleaching

It has been shown in Table 4, the resulting bleached pulp had an α-cellulose content of about 91–92% and a pentosan content of 7%. But higher purity dissolving pulp was produced from other non-wood like corn stalks by Behin and Zeyghami (2009). Under optimum conditions, the pre-hydrolysis/Kraft pulping with 20% active alkali, 25% sulfidity, and HEHP bleaching resulted in dissolving pulp with α-cellulose content 94.8%, degree of polymerization 279, and ash content 0.75%. The xylose content in jute stick was lower as compared to hardwood (He, Cui, & Wang, 2008). But the produced dissolving pulp from jute stick is not as pure as wood. This is may be due to the entrapped of some part of hemicelluloses within the cellulose matrix and some part was associated with lignin–carbohydrate complex (Gübitz, Stebbing, Johansson, & Saddler, 1998). In addition, degraded carbohydrate content (S_{10}/S_{18}) was increased with increasing active alkali. S_{10}/S_{18} and improved pulp brightness were also observed with active alkali charge. Brightness of the produced dissolving pulp was 88–89% after $D_0EpD_1EpD_2$ bleaching. The low purity of dissolving grade pulp is not suitable for cellulose acetate production. The jute stick dissolving pulp shows low viscosity. The low viscosity of bleached jute stick pulp (3 mPas) can be used to produce CMC derivatives, rayon etc.

4. Conclusions

The dissolved biomass from jute stick and lignin, acetic acid and sugars content in the pre-hydrolysis liquor were increased with increasing pre-hydrolysis severity (time and temperature). Pre-hydrolysed jute stick easily cooked to lower kappa number and acceptable pulp yield. The α-cellulose content and viscosity of the produced dissolving pulp were 92% and 3 mPas, respectively, whereas pulp brightness was 89%. Therefore, the low viscosity and high brightness of bleached pulp from jute stick can be used for CMC derivatives, rayon etc. production.

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