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Jute as raw material for the preparation of microcrystalline cellulose

M. Sarwar Jahan · Abrar Saeed · Zhibin He · Yonghao Ni

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Abstract Cellulose was extracted at a yield of 59.8% from jute fibres based on the formic acid/peroxyformic acid process at an atmospheric pressure. The amounts of dissolved lignin and hemicelluloses were determined in the spent liquor. The results showed that the spent liquor contained 10.6% total sugars and 10.9% lignin (based on jute). Microcrystalline cellulose (MCC) was further prepared from the jute cellulose based on the acid hydrolysis technique. A very high yield, 48–52.8% (based on the jute raw material) was obtained. The acid hydrolysate of cellulose contained 2.7% glucose and 0.2% xylose. The MCC samples obtained from two different conditions, one at a low acidity and the other at a high acidity, were characterized by means of Thermo Gravimetric Analysis, Fourier Transform Infrared, X-ray detraction, Scanning Electron Micrograph, and Transmission Electron Micrograph techniques.

M. S. Jahan (✉) · A. Saeed · Z. He · Y. Ni (✉)
Limerick Pulp and Paper Centre, University of New Brunswick, Fredericton, NB E3B 5A3, Canada
e-mail: sarwar2065@yahoo.co.uk

Y. Ni
e-mail: yonghao@unb.ca

M. S. Jahan
Pulp and Paper Research Division, BCSIR Laboratories, Dhaka, Dr. Qudrat-i-Khuda Road, Dhaka 1205, Bangladesh

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Introduction

Jute has played a significant role in the socio-economic development in some Asian countries. For example, Bangladesh has earned the largest amount of foreign currency by exporting jute, and jute goods have played an important role in the country's economy and have provided considerable employment opportunities to the country's work force. The traditional uses of jute as sack, bag, jute hessian cloth/burlap, jute gunny sack, jute gunny bag, jute yarn, woven bag/sack, carpet backing, etc., have recently declined. Therefore, it is important to develop new products from jute to regain its economic importance. Jute and its subsequent conversion into value-added products may regain the lost glory of jute.

The acetic acid or formic acid process is an effective alternative method to fractionate lignocellulosic materials to produce cellulosic pulp/other products (Poppius et al. 1991; Jimenez et al. 1998; Lam et al. 2001; Kham et al. 2005; Jahan et al. 2007d; Jahan 2009). The process can be carried out at an atmospheric pressure. The acid used in the process can be easily recovered by distillation and reused in the process (Muurinen 2000). The method has been used to separate cellulose, hemicellulose and lignin from lignocellulosic materials (Jahan et al. 2007d; Pan and Sano 2005; Seisto and Poppius 1997). The lignin thus obtained is an optimal feedstock for many value-added products due to its favourable physical and chemical properties (Kubo et al. 1998; Cetin and Ozmen 2002). The sugars from hemicellulose are readily convertible to chemicals and fuels, therefore, the overall process concept fits well with the vision of biorefinery (Amidon et al. 2008; Li et al. 2010; Ragauskas et al. 2006; van Heiningen 2006; Yoon et al. 2008).

The percentage of crystallinity of jute fibre (73.4%) was considerably high than other non-woods (52–53%; Jahan et al. 2009). The higher crystallinity in jute fibre indicates its suitability in the preparation of micro-/nano-cellulose crystal (MCC/NCC). MCC/NCC can find many high value-added applications, including the production of light-weight and high-strength hybrid composites (Samir et al. 2005). It was reported that a controlled acid hydrolysis can produce cellulose nanocrystals with an elastic modulus of 150 GPa, which is higher than that of the S-glass (85 GPa) and Aramid fibres (65 GPa; Samir et al. 2004).

Different methods have been reported on the isolation of MCC/NCC from lignocellulosics. Moran et al. (2008) isolated MCC from sisal fibres by: (1) treatment with sodium chlorite, followed by NaOH; (2) acid hydrolysis. Alemdar and Sain (2008) isolated NCC from wheat straw and soy hull by: (1) soaking them in NaOH; (2) HCl treatment and peroxide bleaching; (3) cryocrushing. Wang et al. (2009) isolated MCC from jute fibre by: (1) mercerization with 12% NaOH at room temperature for 2 h; (2) acid hydrolysis; (3) finally treated with NaOH. Lee et al. (2009) prepared NCC from microcrystalline cellulose by: (1) acid hydrolysis in 2.5 N H_2SO_4; (2) followed by sonication.

The biorefinery concept has received much attention recently (Amidon et al. 2008; Chambost et al. 2008; Jahan et al. 2009; Leschinnsky et al. 2009; Yoon et al. 2008; Liu et al. 2010) with the objective of producing multiple products, including fuels, polymers, chemicals, so that the value of lignocellulosic materials can be maximized. In this project, we followed the same concept to process jute fibre. The scope of the study includes: (1) jute fibre was fractionated into cellulose, hemicelluloses and lignin based on the formic acid and peroxyformic acid treatment processes; (2) the cellulose fraction was subsequently converted into micro-crystalline cellulose (MCC) based on the weak and strong acid hydrolysis; (3) the prepared MCC samples were characterized based on the Thermo Gravimetric Analysis, Fourier Transform Infrared (FTIR), X-ray detraction, Scanning Electron Microscope (SEM), and Transmission Electron Microscope (TEM) techniques.

Experimental

Materials

Retted jute fibre was collected from the Narayangong District in Bangladesh. The chemical compositions were: 0.6% acetone extractive content, 12.7% Klason lignin, 13.9% pentosan, 15.8% hemicelluloses, 65.0% α-cellulose and 2.1% ash.

Formic acid treatment

The jute fibre was refluxed with Formic Acid (FA) in a hotplate under the following conditions:

- 90% (v/v) formic acid concentration
- 120 min reaction time at boiling temperature
- Liquor to fibre ratio: 8 (v/w).

At the completion of the reaction time, the fibre was filtered in a Büchner funnel and washed with fresh formic acid, followed by hot distilled water.

Peroxyformic acid treatment

The formic acid treated fibre was further delignified with peroxyformic acid (PFA) at 80 °C. PFA was prepared by mixing 90% formic acid with 4% H_2O_2. The reaction was carried out in a thermostatic water bath. At the completion of 120 min, the fibre was filtered off and washed using an 80% formic acid solution.

Bleaching

Bleaching experiments of unbleached pulp were carried out at a 10% pulp concentration. The pH was adjusted to 11 by adding the required amount of sodium hydroxide (NaOH). The hydrogen peroxide charge was 4% (on fibre material). Other conditions were 80 °C, and 1 h. The pulp yield was determined gravimetrically. The α-cellulose content in the bleached jute fibre was determined according to the Tappi Test Method (T 203 om-88).

Preparation of MCC

The acid hydrolysis of FA/PFA/H_2O_2-treated fibre was conducted according to the method of Batitista (1975). Fifteen grams were hydrolyzed in 375 mL of 2.0 N sulphuric acid at 100 °C for 120 min with constant agitation using a ground-joint electrical stirrer. The reaction mixture was allowed to cool to room temperature and filtered. The filtrate was collected for sugar analysis. The white residue obtained was washed repeatedly with distilled water, diluted ammonium hydroxide solution (5%), and more distilled water until becoming acid-free (the filtrate showed a near neutral pH). The residue was then dried in a vacuum oven to a constant weight and ground in a Wiley mill into fine powder (designated as MCC1).

The other MCC sample was produced by following a literature procedure (Dong et al. 1998) under the conditions of 64% w/w sulphuric acid solution for 5 h at 45 °C with constant stirring. The hydrolysis was cooled by adding a large volume of water to the reaction mixture. The resultant mixture was washed with cold water and centrifuged repeatedly. The residue was then dried in a vacuum oven to a constant weight for 48 h and ground in a Wiley mill into fine powder (designated as MCC2).

Isolation of lignin and sugar

The lignin dissolved during the FA/PFA process was precipitated by adding water to the concentrated spent liquor (five times more than the volume of formic acid). The residue was filtered in a Büchner funnel, and washed with water and vacuum dried over P_2O_5. The filtrate was subjected to the sugar analysis.

Sugar analysis

The sugar concentrations were determined by using an Ion Chromatography unit equipped with CarboPac™ PA1 column (Dionex-300, Dionex Corporation, Canada) and a pulsed amperometric detector (PAD; Li et al. 2010). The further acidic hydrolysis of the pre-hydrolysis liquor (PHL) was carried out at 121 °C and 4% sulphuric acid in an oil bath (Neslab Instruments, Inc., Portsmouth, NH, USA) to convert oligosaccharide to monosaccharide. The PAD settings were $E1 = 0.1$ V, $E2 = 0.6$ V and $E3 = -0.8$ V. De-ionized water was used as the eluant with a flow rate of 1 mL/min, 0.2 N NaOH was used as the regeneration agent with 1 mL/min flow rate and 0.5 N NaOH was used as the supporting electrolyte with 1 mL/min flow rate. The samples were filtered and diluted prior to analysis. The sugar content in the PHL before the acid hydrolysis represented the monomeric form while the oligomeric sugars were calculated from the difference with and without the acid hydrolysis (Li et al. 2010).

Scanning electron microscope and transmission electron microscope analyses

Scanning electron microscope (JEOL 6400, Tokyo, Japan) was used for the microstructural analysis of the MCC samples with gold coating. All images were taken at an accelerating voltage of 15 kV. The TEM observations were made with JOEL 2011 STEM. For TEM, a 20 μL drop of solution was pipetted onto a carbon coated copper grid.

Thermo gravimetric analysis

The thermal gravimetrical analysis was performed with about 5 mg of air-dried sample using a Perkin-Elmer set-up (TAQ 500) and a heating rate of 20 °C/min under a nitrogen atmosphere.

FTIR analysis

The dried samples were embedded in KBr pellets, and were analyzed by using a Shimadzu FTIR spectrometer (model 8201PC). The spectra were recorded in the absorption band mode in the range of 4,000–400 cm^{-1}.

X-ray diffractometry

The X-ray powder patterns for α-cellulose, MCC1 and MCC2 were scanned and recorded using the Bruker D8 Advance spectrometer with a D-5000 rotating anode X-ray generator from 10 to 40 of 2-theta (scanning angle), using Cu Kα radiation generated at 30 mA and 40 kV. The crystalline indices of samples were calculated from the X-ray diffraction patterns based on the following equation (Hermans and Weidinger 1950).

$$Xc = I_{002} - I_{am}/I_{002} \times 100$$

where, Xc is crystallinity index, I_{002} is the peak intensity from the 002 lattice plane (2-theta = 22°) and I_{am} the peak intensity of the amorphous phase.

Results and discussion

Fibre fractionation

Shown in Table 1 are the results of jute fibre fractionation after the formic acid/peroxyformic acid/hydroxide peroxide (FA/PFA/H_2O_2) process. The cellulose fraction has very high α-cellulose (97.8%) content, indicating that hemicelluloses and lignin were effectively removed in the FA/PFA/H_2O_2 process. The cellulose yield was 59.8%. This is a very high in comparison of other non-wood material; for example, Pan and Sano (2005) obtained 44.2% yield from wheat straw in the acetic acid process. The yield from reed canary grass from the formic acid treatment, followed by peroxyformic acid treatment, was 50.9% (Seisto and Poppius 1997). The FA/PFA treatment of dhaincha gave a yield of about 49.2% at a Kappa number of 20 (Jahan et al. 2007b), while the kraft process of dhaincha gave a yield of 44% at a Kappa number of 21 (Jahan et al. 2007c). The much higher yield from jute in such a process is due to: (1) very high α-cellulose content in the initial raw materials (Jahan et al. 2007d); and (2) the α-cellulose is highly protected in the FA/PFA process (Pan and Sano 2005). It was reported (Jahan et al. 2007a, b; Pan and Sano 2005) that acetic acid and formic acid can effectively remove lignin and hemicelluloses from other lignocellulosics, such as dhaincha, and wheat straw, at an atmospheric pressure.

Mass balance

The total biomass recovered in the FA/PFA/H_2O_2 process was 81.3%, which is the sum of 59.8% cellulose yield as the solid residue, 10.7% total dissolved sugars and 10.9% lignin in the spent liquor (Table 1). This was relatively lower, most likely due to: (1) some biomass such as lignin, hemicelluloses, volatile compounds (e. g. methanol) was lost during alkaline peroxide treatment. This has been well supported by literature results; for example, in the alkaline peroxide process, He et al. (2004a, b) reported that methanol is formed and a pulp yield loss of about 4–12%; (2) the formation of 4-O-methy glucuronic acid and other degradation products (He et al. 2005); (3) inorganics dissolved from jute.

The spent liquors from the FA/PFA treatments were further concentrated, followed by dilution with water, so that lignin can be precipitated. The extracted lignin was 10.9% (based on original material), which represented 85.8% of the klason lignin content of jute. The formic acid treatment (in 90% v/v formic acid solution) of rice straw at an atmospheric pressure dissolved about 90% of lignin present in the raw material (Lam et al. 2001). Seisto and Poppius (1997) found 11.3–13.0% lignin (based on original materials) from different grasses in the FA/PFA process, which accounted for 56–59% of the klason lignin. Following the biorefinery concept, Yoon et al. (2008) extracted loblolly pine in hot water prior to pulping and obtained 5.11% lignin in the prehydrolysate. The lignin content in hot-water extract of sugar maple was 3.27% (Amidon and Liu 2009). Lignin can be the starting material for high value-added applications in renewable polymeric materials development (Satheesh et al. 2009). The value added applications of lignin not only helps to boost the economic viability of the biorefinery but also serves as a source of renewable materials.

The sugar compositions (monomeric and oligomeric) in the formic acid spent liquor are shown in Table 2. Under the employed conditions, 10.6% of the sugars of the jute can be extracted. Xylose/xylan accounted for nearly 80% of the total sugars dissolved during the treatment, while the glucose/glucan content was only 8.9% of the total sugars in the spent liquor. Pan and Sano (2005) reported that 63.5% of xylose/xylan was dissolved in the spent liquor during the acetic acid treatment of wheat straw, while cellulose was highly protected. Lam et al. (2001) observed that above 80% of pentose sugars were dissolved from rice straw in the formic acid treatment at an atmospheric pressure. Xylose/xylan

Table 1 FA/PFA/H_2O_2 fractionation of jute fibre

Cellulose fraction		Spent liquor	
Yield (% on original material)	α-cellulose (% on cellulose)	Total sugar (% on original material)	Lignin (% on original material)
59.8	97.8	10.6	10.9

Table 2 Chemical compositions of spent liquor (% on original material)

	Arabinose	Galactose	Glucose	Xylose	Mannose	Ramnose	Total
Mono- sugars	0.12	0.32	0.15	7.50	0.20	0.11	8.40
Oligo- sugars	0.19	0.12	0.80	0.95	0.23	0	2.29
Total	0.31	0.44	0.95	8.45	0.43	0.11	10.69

Table 3 Dimension characteristics of MCC1 and MCC2 and sugar composition in cellulose acid hydrolysis liquor

	Diameter, μm			Acid hydrolysis liquor (% on cellulose)	
	Max	Min,	Avg (SD)	Glucose	Xylose
MCC1	10.1	5.6	8.1(1.35)	4.5	0.31
MCC2	9.8	4.4	7.5(1.82)	5.2	0.32

can be converted into xylitol, yeast, furfural and others in the chemical or enzymatic processes.

Microcrystalline cellulose preparation

Microcrystalline cellulose can be prepared from the acid hydrolysis (Yu and Atalla 1998; Dong et al. 1998). This is because acid hydrolysis is effective in dissolving the amorphous cellulose, leaving behind the micro-cellulose crystalline. The yields of MCC1 and MCC2 were 88.3 and 80.2% on cellulose (after the FA/PFA/H_2O_2 treatment), respectively, which is equivalent to 52.8 and 48.0% on jute. Their yields are exceptionally high, which can be attributed to the very high crystalline proportion in jute cellulose. In our earlier study, it was found that the cellulose crystallinity proportion of jute fibre (73%) was much higher than that from other non-wood (Jahan and Mun 2009) or wood species (Andersson et al. 2003; Fengel and Wegener 1989).

The sugar analysis of the acid hydrolysate was carried out based on the ion chromatographic method and the results are presented in Table 3. In the MCC1 and MCC2 preparation “4.5% glucose and 0.3% xylose” and “5.2% glucose and 0.32% xylose” were dissolved (based on cellulose), respectively. Some of the glucose and xylose formed may be further converted to hydroxymethyl furfural (HMF) and furfural under the present conditions because it is well known that in a high acid concentration HMF and furfural are generated from the hexoses and pentoses (Leschinnsky et al. 2009). The glucose and xylose present in the hydrolysate can be further utilized for the production of cellulosic ethanol following the biorefinery concept, as reported by many recent studies (Bothast et al. 1999; Chandrakant and Bisaria 1998; Sedlak and Ho 2004.

Microcrystalline cellulose characterization

SEM and TEM observations

MCC is known to have strong mechanical properties, therefore can be used in the production of high-strength and low-abrasive products (Eichhorn and Young 2001; Mathew et al. 2005). It is well accepted that the dimensions of the MCC are vital in yielding these mechanical properties (Sakurada et al. 1962). The dimension characteristics of the MCC samples prepared from jute cellulose based on the acid hydrolysis are shown in Table 3. It can be seen that the average diameters are 8.1 and 7.5 μm for MCC1 and MCC2, respectively, which are lower than those reported by others; Wang et al. (2009) reported that microfibrils of 40–80 nm can be produced from the mercerization/steam acid/alkali process of jute fibre; Alemdar and Sain (2008) produced MCC of 10–15 μm from rice straw and soy hull. Shown in Fig. 1 are the SEM micrograph of the MCC1 and MCC2 prepared from jute cellulose. It is evident that: (1) both MCC1 and MCC2 are well-defined fibrils; (2) more fibrillation was observed in MCC2 as compared to MCC1. The TEM results for the MCC1 and MCC2 are shown in Fig. 2. They revealed that some of MCC2

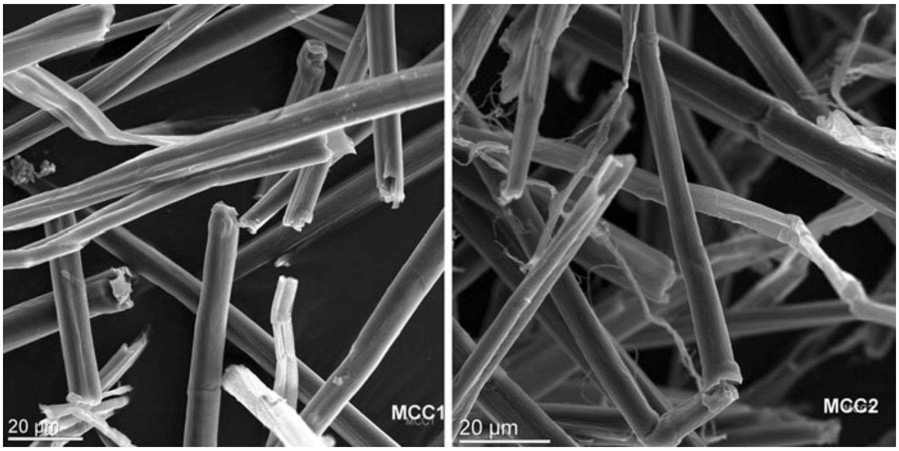

Fig. 1 SEM micrograph of MCC1 and MCC2

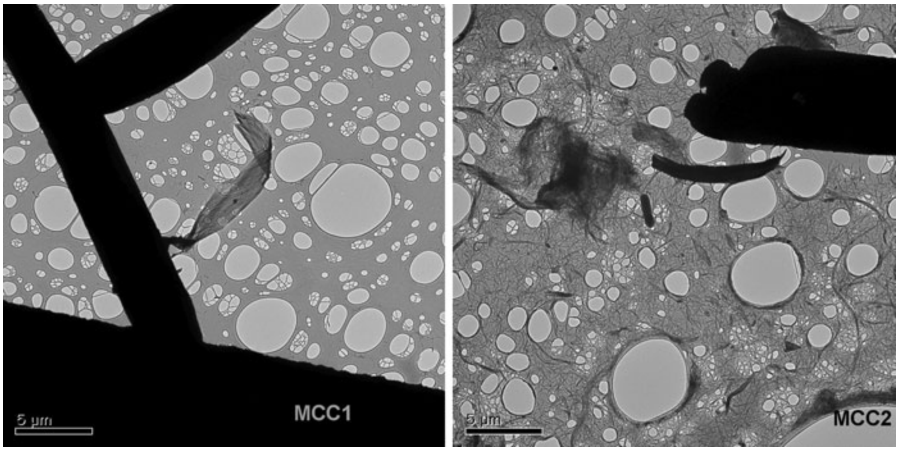

Fig. 2 TEM micrograph of MCC1 and MCC2

fibrils are defibrillated to nanofibre from the cell walls and the width were in the range of 15–40 nm.

Thermo gravimetric analysis

The thermal properties are critical for many applications, including the use of MCC for the production of biocomposites; in these applications, the processing temperature may rise above 200 °C. Many studies related to the decomposition of lignocellulosic materials can be found in specific literature. For example, Yang et al. (2007) showed that in the thermal analysis, cellulose decomposition started at 315 °C and continued until 400 °C. The maximum weight loss rate was reached at 355 °C. At 400 °C almost all cellulose was pyrolyzed, and the solid residuals were relatively small (6.5 wt%). It can be seen in Fig. 3 that there were no other degradation peaks except that of cellulose, which supported the conclusion that the MCC samples we prepared are very pure. The weight losses of these products have three stages: (1) dehydration of absorbed water (<200 °C), (2) cellulose chain scissions (200 ~ 380 °C) (Gassan and Bledzki 2001), due to the breaks of C–C and C–O bonds; (3) aromatization (>380 °C), leading to the formation of the residual char. The decomposition peaks of the maximum mass loss were at 337, 313 and 315 °C for cellulose and MCC1 and MCC2, respectively. The negatively charged sulfate groups introduced into the outer surface of the cellulose crystals decreased thermal stability (Fahma et al. 2010). It was reported (Alemdar and Sain 2008) that

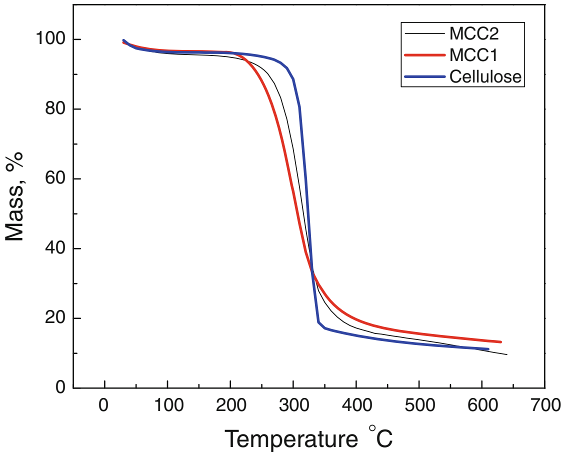

Fig. 3 Thermal degradation behaviors of cellulose, MCC1 and MCC2

the degradation temperature of nano-fibrils from wheat straw and soy hulls was 296 and 290 °C, respectively. The solid residue was 12–14% at 600 °C. Based on the above results, we can conclude that the MCC samples from jute have excellent thermal stability and will be suitable in the production of bio-composites.

FT-IR

The FT-IR spectra of jute MCC samples are shown in Fig. 4. The band at 3,340–3,420 cm^{-1} related to stretching of H-bonded OH groups and 2,900 cm^{-1} to the C–H stretching (Wang et al. 2007; Cao and Tan 2004; Sun et al. 2008). A very weak shoulder at 1,714 cm^{-1} (indicated by arrow in Fig. 3) in cellulose is indicative of either the acetyl and uronic ester groups of the trace amount of hemicelluloses (Sain and Panthapulakkal 2006; Sun et al. 2005). The absorbency at 901, 1,033, 1,114, 1,158 cm^{-1} increased weakly, while it caused no change at 1,201, 1,237, 1,281, 1,316, 1,336, 1,373, 1,431, 2,900 cm^{-1}. The acid hydrolysis removed the amorphous cellulose on the surface, therefore, more C–OH, C–O-C and C–C bonds were exposed, resulting in the increases in the stretching absorbency (Sun et al. 2008). For the two MCC samples, there is an obvious absorption at 710 cm^{-1} and weak shoulder at 750 cm^{-1}, which were assigned to I$_\alpha$ (triclinic) and I$_\beta$ (monoclinic) cellulose (Sugiama et al. 1991; Oh et al. 2005). These results indicate that these samples are rich in I$_\beta$ (indicated by arrow in Fig. 3). The I$_\beta$ proportion in the samples increased in the order of MCC2 > MCC1 > Cellulose.

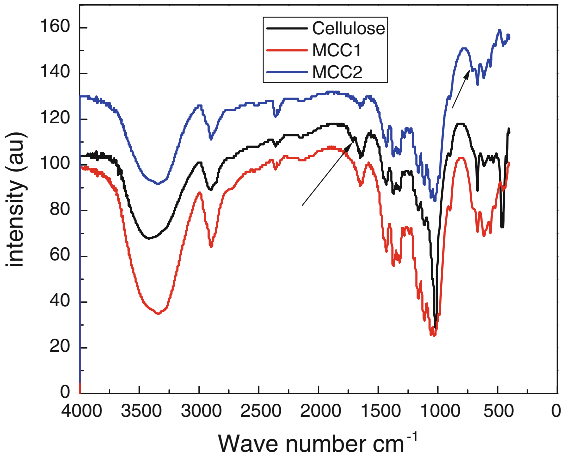

Fig. 4 FT-IR spectra of cellulose, MCC1 and MCC2

X-ray diffraction

The X-ray diffraction pattern of the MCC samples is shown in Fig. 5. The peak at 2-theta = 22° is sharper for MCC2, indicative of a higher crystallinity degree in the structure of MCC2. The crystallinity index of cellulose of 68.7% was found, which increased to 74.9% for MCC2. It was reported that the crystallinity degree increased with the transformation of micro to nano- fibrils (Lee et al. 2009). Leonard and Martin (2002) observed that the crystallinity degree of jute fibre was 71%, which is similar to that of ramie or

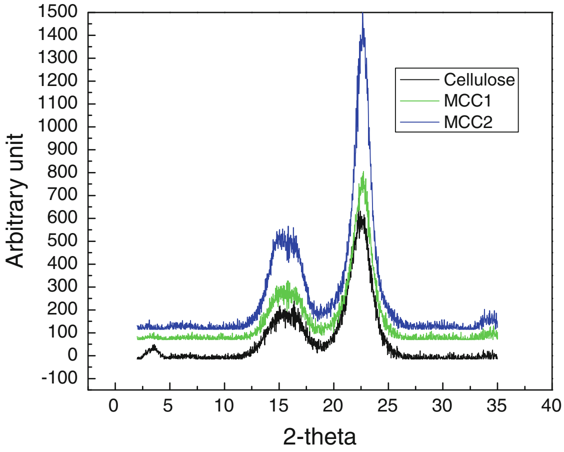

Fig. 5 X-ray diffraction patterns of cellulose, MCC1 and MCC2

cotton (Ray 1969). The crystallinity index of flax and rutabaga nanofibrils was estimated to be 59 and 64%, respectively (Bhatnagar and Sain 2005). The higher crystallinity is associated with its higher tensile strength of the MCC.

Conclusion

The formic acid/peroxyformic acid/hydrogen peroxide process can effectively remove lignin and hemicelluloses from jute fibre. The cellulose yield in this process was very high (59.8%). The dissolved lignin and hemicelluloses can be recovered by other unit operations. This study has demonstrated that a large amount of sugars and lignin (about 106 kg of sugars and 109 kg of lignin/ton jute) can be extracted, which account for 76% of hemicelluloses and 85.8% of lignin in jute. The yield of MCC was 48–52.8% on jute. The TEM results showed that the diameter of fibrillated MCC2 samples was 15–40 nm. The crystallinity of the MCC2 sample was higher than that of the MCC1 sample. The I_β proportion in the samples increased in the order of MCC2 > MCC1 > Cellulose. The MCC samples produced from jute have good thermal stability. The high acid concentration method produced MCC with better properties than the low acid concentration method.

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References

Alemdar A, Sain M (2008) Isolation and characterization of nanofibres from agricultural residues—wheat straw and soy hulls. Bioresour Technol 99(6):1664–1671

Amidon TE, Christopher DW, Shupe AM, Wang Y, Graves M, Liu S (2008) Biorefinery: conversion of woody biomass to chemicals, energy and materials. J Biobased Mater Bioenergy 2(2):100–120

Amidon TE, Liu S (2009) Water-based woody biorefinery. Biotechnol Adv 27(5):542–550

Andersson S, Serimaa R, Paakkari T, Pekka S, Pesonen E (2003) Crystallinity of wood and the size of cellulose crystallites in Norway spruce (*Picea abies*). J Wood Sci 49(6):1435

Batitista OA (1975) Microcrystal polymer science. McGraw Hill, New York, p 17

Bhatnagar A, Sain M (2005) Processing of cellulose nanofiber-reinforced composites. J Reinforced Plast Compos 24: 1259–1268

Bothast RJ, Nichols NN, Dien BS (1999) Fermentations with new recombinant organisms. Biotechnol Prog 15:867–875

Cao Y, Tan H (2004) Structural characterization of cellulose with enzymatic treatment. J Mole Struct 705(1–3): 189–193

Cetin NS, Ozmen NU (2002) Use of organosolv lignin in phenol-formaldehyde resins for particleboard production I. Organosolv lignin modified resins. Int J Adhes Adhesives 22(6):477–480

Chambost V, Mcnutt J, Stuart PR (2008) Guided tour: implementing the forest biorefinery (FBR) at existing pulp and paper mills. Pulp Paper Canada 109(7):1–9

Chandrakant P, Bisaria VS (1998) Simultaneous bioconversion of cellulose and hemicellulose to ethanol. Crit Rev Biotechnol 18:295–331

Dong XM, Revol J-F, Gray DG (1998) Effect of microcrystallite preparation conditions on the formation of colloid crystals of cellulose. Cellulose 5(1):19–32

Eichhorn SJ, Young RJ (2001) The Young's modulus of a microcrystalline cellulose. Cellulose 8:197–207

Fahma F, Iwamoto S, Hori N, Twata T, Takemura A (2010) Isolation, preparation, and characterization of nanofibers from oil palm empty-fruit-bunch (OPEFB). Cellulose 17:977–985

Fengel D, Wegener G (1989) Wood chemistry, ultrastructure, reactions, Paperback edition edn. Walter de Gruyter, Berlin

Gassan J, Bledzki A (2001) Thermal degradation of flax and jute fibers. J Appl Polym Sci 82:1417–1422

He Z, Ni Y, Zhang E (2004a) Further understanding on the cationic demand of dissolved substances during peroxide bleaching of a Spruce TMP. J Wood Chem Technol 24(2):153–168

He Z, Ni Y, Zhang E (2004b) Alkaline darkening and its relationship to peroxide bleaching of mechanical pulp. J Wood Chem Technol 24(2):1–12

He Z, Ni Y, Zhang E (2005) Further understanding on the sodium borohydride assisted peroxide bleaching of mechanical pulps (the P-R process). Appita J 58(1):72–76

Hermans PH, Weidinger A (1950) Quantitative investigation of X-ray diffraction by "amorphous" polymers and some other noncrystalline substances. J Polymer Sci 5: 269–281

Jahan MS (2009) Studies on the effect of prehydrolysis and amine in cooking liquor on producing dissolving pulp from jute (*Corchorus capsularis*). Wood Sci Technol 43:213–224

Jahan MS, Mun SP (2009) Isolation and characterization of lignin from tropical and temperate hardwood. Bangladesh J Sci Ind Res 44(3):271–280

Jahan MS, Chowdhury DAN, Islam MK, Islam MS (2007a) Organic pulping of jute and its mechanism. Cellulose Chem Technol 41(2–3):137–147

Jahan MS, Chowdhury DAN, Islam MK (2007b) Atmospheric formic acid pulping and TCF bleaching of dhaincha (*Sesbania aculeata*), kash (*Saccharum spontaneum*) and banana stem (*Musa Cavendish*). Ind Crop Prod 26(3): 324–331

Jahan MS, Chowdhury DAN, Islam MK (2007c) Pulping of dhaincha (Sesbania aculeata). Cellulose Chem Technol 41:413–421

Jahan MS, Maruf AA, Quaiyyum MA (2007d) Comparative studies of pulping of jute fiber, jute cutting and jute caddis. Bangladesh J Sci Ind Res 42(4):425–434

Jahan MS, Saeed A, Ni Y, He Z (2009) Pre-extraction and its impact on the alkaline pulping of bagasse. J Biobased Mater Bioenergy 3(4):380–385

Jimenez L, Torre M, Maestre J, Ferrer Perez F (1998) Delignification of wheat straw by use of low molecular weight organic acid. Holzforschung 52(2):191–196

Kham L, Bigot YE, Delmas M, Avignon G (2005) Delignification of wheat straw using a mixture of carboxylic acids and peroxoacids. Ind Crops Prod 21:9–15

Kubo S, Uraki Y, Sano Y (1998) Preparation of carbon fibers from softwood lignin by atmospheric acetic acid pulping. Carbon 36(7–8):1119–1124

Lam HQ, Bigot YL, Delmas M, Avignon G (2001) Formic acid pulping of rice straw. Ind Crop Prod 14:65–71

Lee SY, Mohan DJ, Kang IA, Doh GH, Lee S, Han SK (2009) Nanocellulose reinforced PVA composite films: effects of acid treatment and filler loading. Fibers Polym 10(1): 77–82

Leonard YM, Martin PA (2002) Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization. J Appl Polymer Sci 84:2222–2234

Leschinnsky M, Sixta H, Patt R (2009) Detailed mass balances of the autohydrolysis of *Eucalyptus globulus* at 170 °C. BioRes 4(2):687–703

Li H, Saeed A, Jahan MS, Ni Y, van Heiningen ARP (2010) Hemicellulose removal from hardwood chips in the prehydrolysis step of the kraft-based dissolving pulp production. J Wood Chem Technol 30(1):48–60

Liu Z, Fatehi P, Jahan MS, Ni Y (2010) Separation of lignocellulosic materials by combined processes of prehydrolysis and ethanol extraction. doi:10.1016/j.biortech.2010.08.049

Mathew AP, Oksman K, Sain M (2005) Mechanical properties of biodegradable composites from poly lactic acid (PLA) and microcrystalline vellulose (MCC). J Appl Polym Sci 97:2014–2025

Moran JI, Alvarez VA, Cyras VP, Vazquez A (2008) Extraction of cellulose and preparation of nanocellulose from sisal fibers. Cellulose (Dordrecht, Netherlands) 15(1): 149–159

Muurinen E (2000) Organosolv pulping—a review and distillation study related to peroxy acid pulping. Ph.D Thesis, Faculty of Technology, University of Oulu, Finland

Oh SY, Dong YI, Younsook S, Gon S (2005) FTIR analysis of cellulose treated with sodium hydroxide and carbon dioxide. Carbohydr Res 340(3):417–428

Pan X, Sano P (2005) Fractionation of wheat straw by atmospheric acetic acid process. Bioresour Technol 96: 1256–1263

Ragauskas AJ, Nagy M, Kim DH, Eckert CA, Hallett JP, Liotta CL (2006) From wood to fuels: integrating biofuels and pulp production. Ind Biotechnol 2(1):55–65

Ray PK (1969) On the degree of crystallinity in jute and mesta fibers in different states of purifications and moisture conditions. J Appl Polym Sci 13(12):2593–2600

Poppius K, Mustonen R, Huovila T, Sundquist J (1991) Milox pulping with acetic acid/peroxy acid. Paperi ja Puu 73(2):154–158

Sain M, Panthapulakkal S (2006) Bioprocess preparation of wheat straw fibers and their characterization. Ind Crop Prod 23(1):1–8

Sakurada I, Nukushina Y, Ito T (1962) Experimental determination of the elastic modulus of crystalline regions of oriented polymers. J Polym Sci 57:651–660

Samir ASA, Alloin F, Dufresne A (2005) Review of recent research into cellulosic whiskers, their properties and their application in nanocomposite field. Biomacromolecules 6:612–626

Samir ASA, Alloin F, Paillet M, Dufresne A (2004) Tangling effect in fibrillated cellulose reinforced nanocomposites. Macromolecules 37:4313–4316

Satheesh MNK, Mohanty AK, Erickson L, Misra M (2009) Lignin and its applications with polymers. J Biobased Mater Bioenergy 3(1):1–24

Sedlak M, Ho YWN (2004) Production of ethanol from cellulosic biomass hydrolysates using genetically engineered *saccharomyces* yeast capable of cofermenting glucose and xylose. Appl Biochem Biotechnol 114(1–3):403–416

Seisto A, Poppius KL (1997) Peroxyformic acid pulping of nonwood plants by the milox method—part 1: pulping and bleaching. Tappi J 80(9):215–221

Sugiama J, Persson J, Chanzy H (1991) Combined infrared and electron diffraction study of the polymerization of native cellulose. Macromolecules 24:2461–2466

Sun XF, Xu F, Sun RC, Fowler P, Baird MS (2005) Characteristics of degraded cellulose obtained from steam-exploded wheat straw. Carbohydr Res 340(1):97–106

Sun Y, Lin L, Deng H, Li J, He B, Sun R, Ouyang P (2008) Structural changes of bamboo cellulose in formic acid. BioResources 3(2):297–315

van Heiningen ARP (2006) Converting a kraft pulp mill into an integrated forest biorefinery. Pulp Paper Canada 107(6): 38–43

Wang H, Li H, Lu Y (2009) Preparation and characterization of micro- and nano-fibrils from jute. Fibers Polym 10(4): 442–445

Wang L, Han G, Zhang Y (2007) Comparative study of composition, structure and properties of Apocynum venetum fibers under different pretreatments. Carbohydr Polym 69(2):391–397

Yang H, Yan R, Chen H, Dong HL, Zheng C (2007) Characteristics of hemicellulose, cellulose and lignin pyrolysis. Fuel 86:1781–1788

Yoon SH, MacEwan K, van Heiningen A (2008) Hot-water pre-extraction from loblolly pine (Pinus taeda) in an integrated forest products biorefinery. Tappi J 7:27–31

Yu X, Atalla RH (1998) A staining technique for evaluating the pore structure variations of micricrystalline cellulose powders. Powder Technol 98:135–138