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Isolation of microcrystalline alpha-cellulose from jute: A suitable and economical viable resource

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Abstract

Cellulose is a natural linear chain homopolymer that is an abundant and common component in all plants. Partially pure depolymerized cellulose, known as microcrystalline cellulose (MCC), is synthesized by mineral acids hydrolysis from α-cellulose precursors obtained from fibrous plants such as jute. Virgin soft and hardwoods are used as the main source of cellulose for raw materials of MCC production. These can be replaced by jute fiber to a great extent as it is considered one of the most promising alternatives. A proximate analysis had been carried out to determine the percentage of cellulose, hemicellulose, fats, and lignin in cellulose by standard methods. The cellulose purity of BJRI Tossa Pat-8 (Robi-1) fiber is identified from FT-IR. The IR results of MCC analysis were indicated 3,337.40cm-1 for the moisture and 1656.45cm-1 for carboxyl groups. In thermogravimetry analysis, at the first phase, 20-95°C is associated with moisture release. The oxidation of Tossa Pat-8 (Robi-1) MCC occurred in the range of 200-400°C. The remaining 0.65% of inorganic materials ash, was obtained at 425oC. This study indicates the cost-effective isolation of MCC from Tossa Pat-8 (Robi-1) and that can be promisingly applicable in several fields such as coatings and membranes explosives, cellulose, textiles, adhesives, films, textiles, food and tobacco, films, pharmaceutical and cosmetics industry, which needs further research.

Keywords: Jute Fibre Robi-1; Acid Hydrolysis; MCC; FT-IR; Thermogravimetric Analyzer (TGA).

1. Introduction

Jute is a natural fiber that is cheap, biodegradable, and abundantly available in Bangladesh. The genome sequencing of jute in recent times has revealed the high hopes for breakthrough success, which has led to the innovation of new products from jute. The sticks from jute are usually used as fuel but can also for the production of charcoal and activated carbon, which are value-added products [1, 2]. As such, the new information of progress in the development of jute products production is certainly an admirable success. Subsequently, the best quality jute produces in Bangladesh and has a great impact on the local economy because it is widely used in the apparel industrial fabrics for furnishing purposes. Recent research demonstrated the use of jute stick for the production of D-xylose [3] which eventually can be used for the production of various rare aldo-pentoses. It has a relatively high strength-to-weight ratio and reasonable low density that are good to moderate tensile strength and initial modulus. Those characteristics make jute an attractive class fiber for the production of yarn and fabric in the textile sector. However, this type of shorter length fiber is not suitable for the spinning process so, results in waste [4]. For that reason, several techniques have been implemented to use the short-length waste fibers as they hold some useful inherent properties.On the other hand, jute is a lignocellulosic

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composite bast fiber; the chemical compositions of jute fibers are alpha-cellulose (60-66%), hemicellulose (17-24%), and lignin (12-16%), respectively [5]. On this view, cellulose can be isolated from these short fibers and can be used for value-added products. Cellulose is non-toxic, renewable, biodegradable, biocompatible, and readily modifiable substance, and it is very attractive as a sustainable material for industry. Microcrystalline cellulose is one of the industrial raw materials that were introduced in the early sixties. It is a purified, partially depolymerized polysaccharide with the formula $(C_6H_{10}O_5)_n$. This linear polysaccharide chain contains several hundred D-glucose units with β(1→4) links. Microcrystalline cellulose is prepared by applying mineral acids to the alpha-cellulose, where solvents and chemical reagents attack the amorphous regions of the alpha-cellulose earliest. The degree of polymerization of microcrystalline cellulose is reduced after that white, odorless, tasteless, crystalline powder particle is obtained [6]. It has hygroscopic nature but is insoluble in water, but in contact with water, it swells. Researchers isolated microcrystalline cellulose by using potassium hydroxide, sodium chlorite, glacial acetic acid, and hydrochloric acid which is very costly [7].

Therefore, the same concept was applied but in a slightly different way, and as a result, the production cost decreases several times (Fig. 1). Micro and nano cellulose derivatives have very promising application in several fields such as cellulose acetate coatings and membranes, cellulose nitrate membranes and explosives, cellulose xanthate textiles, carboxymethyl cellulose coatings, paints, adhesives and pharmaceuticals, methylcellulose films, textiles, food and tobacco industry, ethylcellulose pharmaceutical industry and hydroxyethylcellulose paints, coatings, films, and cosmetics [8]. Bangladesh Jute Research Institute (BJRI) feels to find out value-added jute products that can solve the cellulose-imported problem for the industry, therefore, keeping this burning issue in mind, this research was conducted in order to produce cost-effective and profitably production of MCC, which will earn foreign currency and contribute to the national economy of Bangladesh. MCC production on a huge amount from jute can increase jute usage and thus a vast quantity of jute will be consumed by a single product which will eventually help revitalize the jute sector of Bangladesh. Therefore, an investigation had been done in the present research to produce cost effectively high quality microcrystalline cellulose on a laboratory scale.

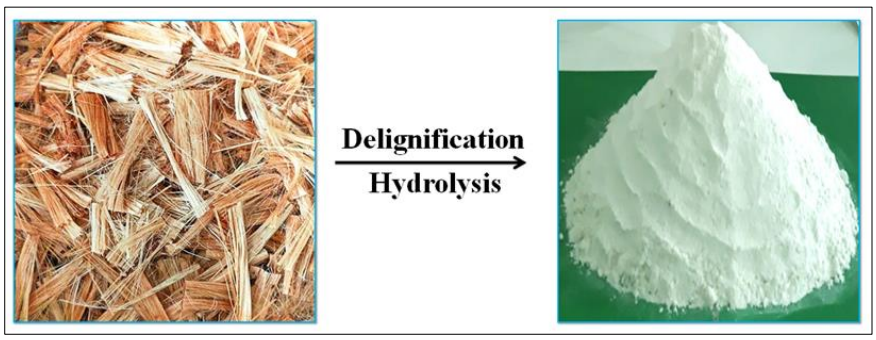

Figure 1 Flow diagram for the preparation of MCC from jute fiber Robi-1

2. Material and methods

BJRI Tossa Pat-8 (Robi-1) fibers were used as precursor material in this study which was obtained from the Head Office of Bangladesh Jute Research Institute (BJRI), Dhaka, Bangladesh. All the chemicals are reagent grade (Merck), such as potassium hydroxide (KOH), glacial acetic acid (CH_3COOH), sodium chlorite ($NaClO_2$), sulfuric acid (H_2SO_4).The research was conducted in the laboratory of Chemistry and Textile Physic Division, BJRI, Dhaka Bangladesh.

2.1. Physical measurements, analysis and product yield of MCC

Fourier transform infrared (FT-IR) spectra of samples were recorded on an FT-IR spectrophotometer in the region of $400cm^{-1}$-$4000cm^{-1}$. The thermal stability of the MCC samples was tested by a thermogravimetric analysis (TGA) analyzer. Proximate analysis had been determined by standard method [9]. The weight of the product after finalization denoted the yield value of MCC which was calculated using the following formula:

$$\text{Yield (\%)} = \frac{\text{weight of MCC}}{\text{weight of jute fiber without moisture}} \times 100$$

Standard methods were used for the determination of fats, alpha-cellulose, hemicelluloses, and lignin of Robi-1 jute fiber.

2.2. Determination of fats content

5.0gm of grinded jute fiber was extracted with petroleum ether in a soxhlet apparatus for 8h. The extract was evaporated to dryness. The liquid fats sample was dried in an oven at 110°C overnight after the cooling weight was taken.

$$\text{Fat (\%)} = \frac{\text{weight of fat}}{\text{weight of jute fiber without moisture}} \times 100$$

2.3. Determination of lignin

5.0gm of grinded jute fiber was taken in the round bottom flask (250ml). 100ml 72% (v/v) sulfuric acid was then added and the flask was put in an ice bath. The reaction is allowed for 1h with occasional stirring by a glass rod. The reaction mixture was refluxed for about 6h, after cooling 200ml distilled water was added to the reaction mixture. The reaction mixture was filtered by a sintered crucible, and was washed thoroughly with hot water until free from acid. The lignin was dried overnight at 105°C for constant weight and lignin percentage was measured using the formula:

$$\text{Lignin (\%)} = \frac{\text{weight of lignin}}{\text{weight of jute fiber without moisture}} \times 100$$

2.4. Determination of alpha-cellulose

Holocellulose was extracted from5.0gm of grinded jute fiber. Extracted holocellulose was taken in a 250ml conical flask and it was treated with 24%, (w/w) 100ml potassium hydroxide with vigorous stirring for 6h. The mixture solution was filtrated and the residue was washed with water several times. Finally, the residual material was washed successively with aqueous acetic acid, water, alcohol, and petroleum ether. The sample was dried overnight at 105°C for constant weight. Percentage of alpha-cellulose was thus obtained by the following formula -

$$\text{Alpha} - \text{cellulose (\%)} = \frac{\text{weight of alpha} - \text{cellulose}}{\text{weight of jute fiber without moisture}} \times 100$$

2.5. Determination of hemicelluloses

For the extraction of hemicelluloses, 9.3% sodium hydroxide solution was added to the lignin-free jute fiber with stirring at cold conditions. After three times extracting with these same methods, the total extracts were neutralized with acetic acid. After the addition of alcohol, the precipitate was collected by centrifuge. The isolated hemicelluloses were then washed with alcohol and finally with acetone and dried overnight at 105°C for constant weight. The percentages of hemicelluloses were then calculated by the following formula

$$\text{Hemicellulose (\%)} = \frac{\text{weight of hemicellulose}}{\text{weight of jute fiber without moisture}} \times 100$$

2.6. MCC isolation from jute fiber

Before going to the isolation of MCC jute fiber was washed three or four times by clean water. Clean jute fiber was swelled in water for 24h.

2.7. Delignification and MCC isolation

Sodium hydroxide (14% w/v) treatment was performed at 120°C for 2h. The dark brown extract was decanted and the highly alkaline precipitate was neutralized by washing with cold water. The precipitate was then dried in a oven for 8h at 105°C. The dried material was taken for subsequent bleaching and further delignification treatment. The mixture of 17.0% sodium chlorite and acetic acid (for keeping pH at 4-5) were used for the removal of lignin present in the jute fiber at 80°C for 2h. Finally, the material was dried in a hot air oven overnight at 90°C. The lignin-free residual matter was further treated with 10% sulfuric acid. The treatment was carried out at 90°C for 1h, keeping the material-to-liquor ratio at 1:30.

3. Results and discussion

Jute is cellulose-based helical microfiber by the combination of crystalline and amorphous area, each unit fiber is connected by 20-30 degrees helical angles to amorphous lignin and hemicelluloses make a complete layer [6]. Such type

multiple layers contain cellulose, hemicelluloses, lignin, pectin, fat, and other organic and inorganic materials creating one primary and three secondary cell walls that form a composite, which is complete fiber. Therefore, understanding the chemical compositions of jute fibers (Robi-1) are needed for chemical analysis. The chemical analysis results of Robi-1 jute are shown in Fig. 2 and the analysis results are alpha-cellulose (65.8%), hemicellulose (18.12%), lignin (14.67%), pectin (0.23%), fat (0.53%), and ash (0.65%), respectively.

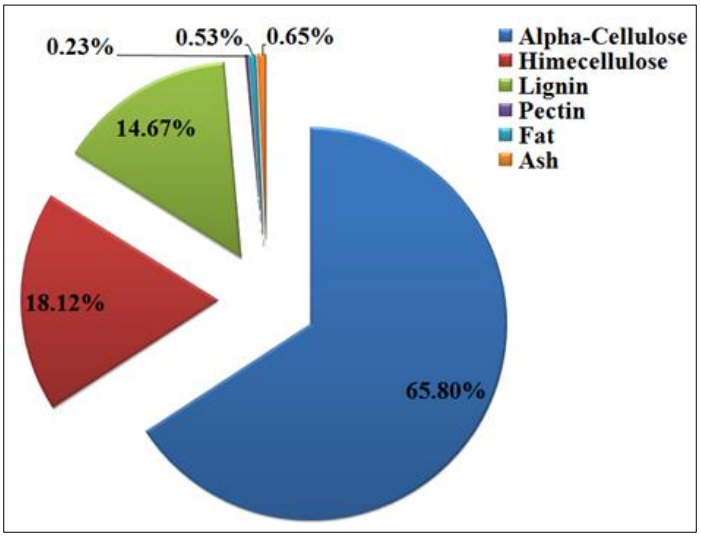

Figure 2 Chemical constituents percentage of Robi-1

Acid hydrolysis is the main process used to isolate the microcrystalline cellulose, which are the smaller building blocks released from the original cellulose fibers. In fiber, cellulose consists of amorphous and crystalline regions [10]. The crystalline regions have a high density compared to the amorphous regions, so when acid hydrolysis is applied to the cellulose fibers, the amorphous regions just lightly disintegrate and produce the individual crystallites. The properties of MCC depend on various factors, such as applied temperature, reaction time and sources of cellulose, and types of acid that are used for hydrolysis [11]. Tappi standard methods have applied potassium hydroxide for the delignification of both hardwood and softwoods. Sodium chlorite and glacial acetic acid were used mainly for the discoloration of fibers [12]. For the isolation of microcrystalline cellulose, hydrochloric acid was used [13]. Many researchers have been following the same process until today, it is very costly. After pretreating with water, we have used sodium hydroxide as an alternative to potassium hydroxide. After draining the dark brown extract was neutralized by cold water. The dried fibrous material was taken for ensuing bleaching only once by treatment of sodium chlorite and glacial acetic acid. The lignin-free residual matter was further treated with (2N). Sulfuric acid for the making of MCC.

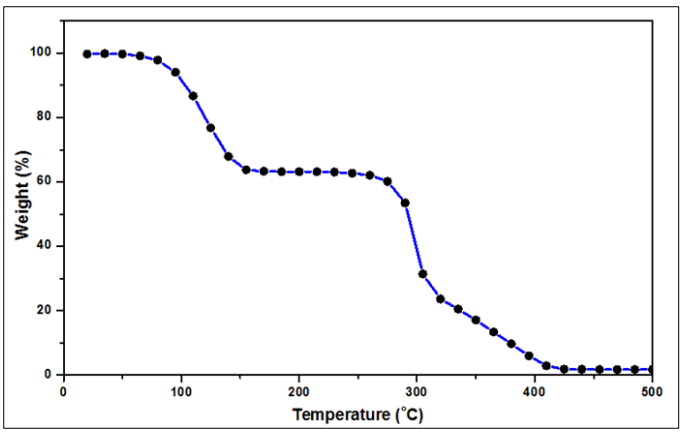

Figure 3 Thermogravimetric graph of jute microcrystalline cellulose

Thermogravimetric analysisof sample was revealed the thermal decomposition of Robi-1 microcrystalline cellulose occurred in three main phases (Fig. 3). The first phase (20-95°C) is associated with moisture release (6.0% weight loss). The most intensive decomposition of an organic matter occurred in the range of 155-275°C (additional 54.0% weight

loss) due to the breaks of C-C and C-O bonds. The maximum weight loss rate was reached at 275-425°C (39.35% weight loss). At 425°C, almost all cellulose was pyrolyzed, and the solid residuals were relatively small (0.65%) [14].

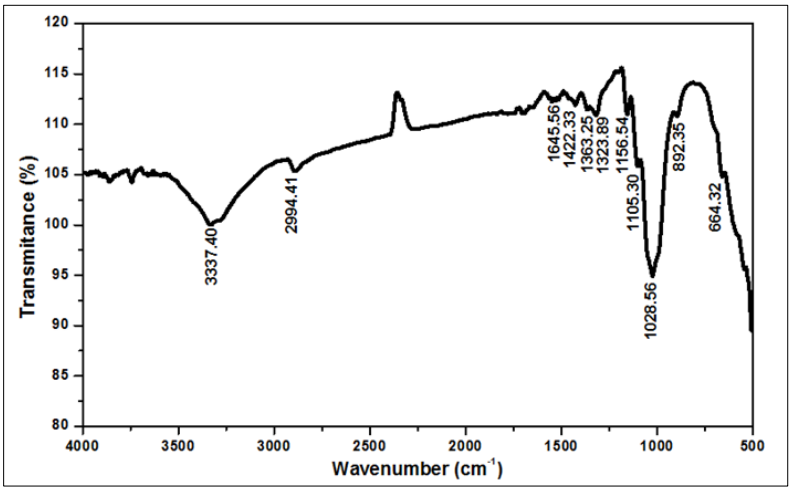

Figure 4 FTIR spectra of Robi-1 microcrystalline cellulose

Table 1 Summary of FT-IR data of Robi-1 and other commercial MCC

Commercial Name of MCC	O-H covalent bond, hydrogen bonding (cm^{-1})	C-H (cm^{-1})	CH_2 (symmetric) at C-6; crystalline region (cm^{-1})	CH_2 (wagging) at C-6 (cm^{-1})	γCOC at β-glycosidic linkage (cm^{-1})	CO at C-6 (cm^{-1})	Glycoside (cm^{-1})
BJRI	3337.40 1645.56	2994.41	1422.33	1320.04	1156.54	1028.56	892.35
Avicel -1	3329.50	2892.70	1428.99	1319.07	1156.12	1024.02	-
Avicel -2	3285.14	2886.92	1424.03	1314.25	1159.97	1026.91	-
Benecel KI5CR	3419.17 1644.98	2894.63	1455.03	-	1194.69	1050.05	944.95 -
Methocel E6 Premium LV	3444.24 1640.16	2901.38	1445.06	-	1190.83	1050.05	943.99
Methocel 5CPS	3449.06 1644.98	2899.45	1454.06	-	-	1051.1	943.96
MCC (Type 101)	3327.57	2895.59	1428.03	-	1157.08	1024.02	552.51
MCC (Type 102)	3326.61 1641.13	-	1427.07	-	1157.08	1032.05	553.47
MCC (Type 106)	3334.32	2893.66	1428.99	-	1157.08	124.02	505.26

The infrared spectra of Robi-1 MCC ranged from 4000 cm^{-1} to 400 cm^{-1} are shown in Fig. 4. The absorptions of approximately 3337.40 cm^{-1}, 2994.41 cm^{-1}, 1422.33 cm^{-1}, 1363.25 cm^{-1} and 892.35 cm^{-1} exhibited in all spectra were associated with the characteristics of native cellulose [15]. All infrared spectra of the samples displayed a broad and intense peak at around 3337.40 cm^{-1} per the characteristic absorption of the OH stretching vibration of cellulose [16, 17]. In addition, peaks observed in the region of 1645.56 cm^{-1} were related to the bending mode of absorbed water in the cellulose [18]. The absorption band at 1422.33 cm^{-1} was associated with intermolecular hydrogen attraction in the C6 group [19], and the peak observed at 1363.25 cm^{-1} was associated with the bending vibration of cellulose C-H. The peak appearing in the 1323.89 cm^{-1} range in the spectra of all samples was attributed to the skeletal vibration of the C-C and C-O groups. The peaks observed at 1156.54 cm^{-1} and 1105.30 cm^{-1} were associated with the stretching vibration of C-O groups in cellulose. The strong absorption peak of the sample in the range of 1028.56 cm^{-1} indicated a skeletal

vibration of the C-O-C pyranose ring skeleton in the cellulose [20]. The diagnostic absorption peaks at 892.35 cm^{-1} for the sample was ascribed to β-type glycosidic linkages in cellulose.

Table 1 demonstrated the summary of FT-IR data of Robi-1 and some commercial MCC those were collected from various sources and named avicel-1, avicel-2, benecel KI5CR, methocel E6 premium LV, methocel 5CPS, MCC (Type 101), MCC (Type 102), and MCC (Type 106), respectively. After analyzing all functional groups, it is understood the Robi-1 MCC is pure and similar to that of other commercial MCC.

4. Conclusion

Microcrystalline cellulose is prepared by simple process of hydrolysis from various fibre sources and way for the rising its uses and demand such as pharmaceuticals, textiles paints, food, and cosmetics. The physical, chemicals properties of the Robi-1 microcrystalline cellulose were comparable with properties of commercially available microcrystalline cellulose. The current research was undertaken for the preparation of microcrystalline cellulose from jute in a cost-effective way. In order to produce very cheap MCC from jute fibre for the local and international market, further research need to done.

Compliance with ethical standards

Acknowledgments

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Disclosure of conflict of interest

The authors declare that they have no conflict of interest.

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