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JUTE STICK - A SUITABLE BIOMATERIAL AND ECONOMICAL VIABLE RESOURCE FOR THE PREPARATION OF MICROCRYSTALLINE CELLULOSE

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ABSTRACT

Cellulose is basically a simple linear chain that is the basic component of all plants and it is a natural homopolymer. Microcrystalline cellulose is actually partially pure depolymerized cellulose and it is obtained from the hydrolysis by mineral acids of lignin-free soft plant fiber. Although the main source of raw material for microcrystalline cellulose production is virgin soft or hardwood, jute sticks are quite useful as an alternative source of raw material. The percentages of jute stick moisture, cellulose, hemicelluloses, lignin and ash have been determined by standard methods and these results are cellulose 38-42(%), lignin 22-26(%), pentosan 22(%) and fiber Length 0.8-1.0mm, respectively. The cellulose purity of BJRI Tossa jute stick is identified by FT-IR spectroscopy. The IR results of the MCC analysis indicated 3450.85cm^{-1} for the OH stretching vibration of all jute sticks' cellulose, 2911.41cm^{-1} is attributed to the C-H stretching vibration of polysaccharides, and 1655.45cm^{-1} for the bending mode of the absorbed water molecules. In thermogravimetry analysis, in the first phase, 50–110^{o}C is associated with moisture decomposition. The organic matter decomposition of jute stick MCC occurred in the range of 100-260^{o}C. At 450^{o}C, the remaining 1.22-1.37% of inorganic material ash was obtained. In this study, we have tried to find out the easiest way the release alpha-cellulose and MCC from jute sticks, which can be nicely applicable in various fields such as coatings, food, pharmaceuticals, adhesives, cosmetics, membranes, films, explosives, tobacco, and the textiles industry.

KEYWORDS: Jute stick; Acid Hydrolysis; MCC; FT-IR; Thermogravimetric Analyzer (TGA).

INTRODUCTION

Jute grows in many parts of the world, of which Bangladesh, India, and China produce significant amounts. The chemical components of jute may diversify due to its different geographic locations, soil conditions, climates, and ages.[1,2] However, a huge amount of jute grows in Bangladesh among all the countries. Although jute is a perennial plant, the cultivation starts in April and the retting start in July. Jute is an agricultural cash crop and it plays an important role in the development of the socio-economic conditions of Bangladesh. Most of the export earnings are dependent on jute and jute-related products after garments. Due to the sustainable economic necessity and increasing environmental awareness nowadays, jute fiber shows the potential for developing prospects due to its low cost, biodegradation, renewability, and all-over abundance. In Bangladesh, around 1.8million tons of jute are produced every year. On the other hand, jute stick is agricultural waste, which is left after the extraction of fiber by retting and it amounts to about 2.5-3.0 million tons every year. Jute fiber is a very demanding material for the commercial manufacturing of household goods and industries. Alternatively, there is no commercial importance to the jute stick, although its production is double that of the retted fiber. Local people just use it for their household purposes, i.e., making fences and burning it for cooking. A lot of extracted jute sticks remain unused. Numerous studies have been conducted on the use of jute sticks in making charcoal and activated carbon.[3,4] Normally, jute stick charcoal is not suitable for making good activated carbon. Because of the morphology and crystallite of the carbon in jute stick charcoal is not like natural coal. That is why the conductivity of jute-stick charcoal is much lower than that of natural coal. The morphological and chemical characteristics of jute stick are favorable for the isolation of cellulose because jute stick contains a considerable amount of cellulose (41%). It contains not only cellulose but also lignin (23%), hemicelluloses (24%), and other minor constituents, which are a potential resource for lignocellulosic biomass. Besides that, D-xylose from jute stick was recently reported[5]

which could be used for medicinal purpose. In bio-based products (such as food additives) and formaldehyde resins, jute stick lignin and hemicellulose can be used, respectively. Moreover, jute stick is also a potential raw material for making pulp in various grades of paper (such as writing paper, wrapping paper, packaging paper, printing paper).[6-11] This interesting renewable bio-resource jute stick can be used for the manufacturing of cellulose to reduce deforestation, resulting in a sustainable economy as well as a green environment. From this perspective, cellulose can be separated from jute sticks, and this may provide value-added products to the national economy. Cellulose is a biodegradable, biocompatible, non-toxic, and readily modifiable material, and it is very interesting as a durable substance for the industry. Microcrystalline cellulose is the smaller part of alpha-cellulose and one of the most important raw materials for the industry with the formula $(C_6H_{10}O_5)_n$. Several hundred D-glucose units make a linear polysaccharide chain that contains β(1→4) links.[12] The degree of polymerization of Glucopyranose units of cellulose, in wood and cotton usually is about 10,000, and 15,000 times, respectively. Many researchers have been applying mineral acid solutions to isolate the microcrystalline cellulose, where early hydrolysis happens within amorphous regions of the alpha-cellulose. After reducing the degree of polymerization of cellulose, a white, odorless, tasteless, crystalline powder is obtained.[11,12] Microcrystalline cellulose is insoluble in the water though it is hygroscopic in nature it swells in contact with water. Many studies have been done on the isolation of microcrystalline cellulose by using potassium hydroxide, sodium chlorite, glacial acetic acid, and hydrochloric acid. Herein, we have used jute stick to make microcrystalline cellulose because jute sticks are more suitable sources than jute fiber and hardwood due to it is a renewable crop, abundant, ready availability, and cheapness, and it is anticipated that it will be a more ecological and economical source for making microcrystalline cellulose (**Fig. 1**).

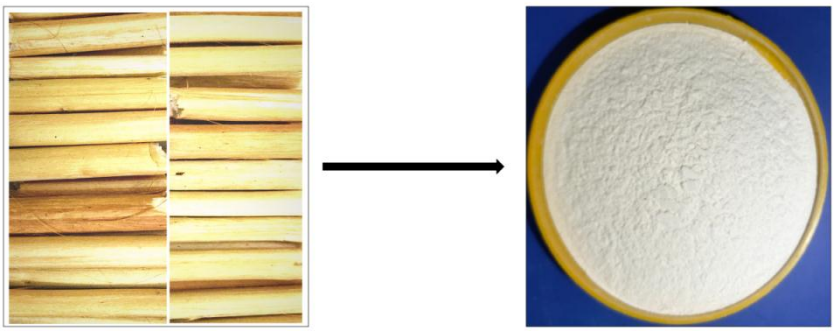

Figure 1: Jute stick microcrystalline cellulose.

MATERIAL AND METHODS

Tossa jute (*C. olitorious*) sticks 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 Industrial Chemistry Department, Chemistry Division, BJRI, Dhaka, Bangladesh.

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.[13] 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, ash of Tossa jute stick.

(A) Determination of ash content 5.0g 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.

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

The chopped jute stick was heated at 900°C in a muffle furnace for 5h. During this time, all the samples converted into ash. Ash content was calculated using the following formula.

$$\text{Ash (\%)} = \frac{\text{Weight of ash}}{\text{Weight of ovendried jute stick}} \times 100$$

(B) Determination of lignin 1.0g of grinded jute stick was taken in the round bottom flask (250ml). 20ml 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 8h, after cooling 40ml distilled water was added to the reaction mixture. The reaction mixture was filtered by a sintered crucible and was washed rigorously 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.

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

(C) Determination of alpha-cellulose Holocellulose was extracted from2.0gm of grinded jute stick. Extracted holocellulose was taken in a 250ml conical flask and it was treated with 24%, (w/w) 40ml potassium hydroxide with vigorous stirring for 8h. 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 -

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

(D) Determination of hemicelluloses For the extraction of hemicelluloses, 10% sodium hydroxide solution was added to the lignin-free jute stick 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.

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

Preparation of MCC from jute stick The jute stick was chopped and washed with clean water, then the clean jute stick was dried at 110°C for 24h in an oven. The dried jute stick was grinned and then submerged in water for 24h.

Delignification of jute stick and microcrystalline cellulose isolation The swelled jute stick was then treated by the sodium hydroxide (18% w/v) at 160°C for 2h in a digester. 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 overnight at 105°C. The dried material was taken for subsequent bleaching and further delignification treatment. The mixture of 15.0% sodium chlorite and acetic acid (pH at 4-5) were used for the removal of lignin present in the jute stick at 80°C for 1h. After wash by clean and cold water, the material was dried in an oven overnight at 80°C. The lignin-free white material is alpha-cellulose. The jute stick alpha-cellulose 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.

RESULTS AND DISCUSSION

Jute stick is one types of soft wood mainly contains cellulosic fiber with the combination of lignin, hemicelluloses and pentosan.[1] To understanding, the chemical compositions of jute stick are needed for chemical analysis. The chemical analysis results of jute stick and the analysis results are alpha-cellulose (40.2%), hemicellulose (24%), lignin (29%), ash (0.8%), respectively (**Fig. 2)**.

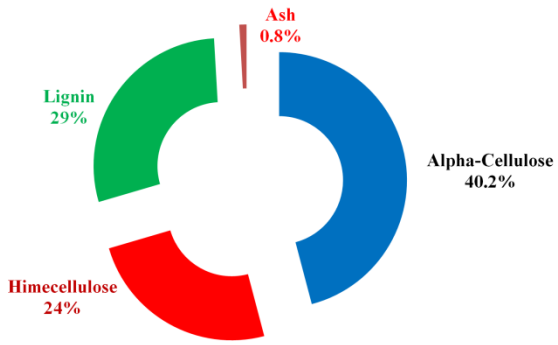

Figure 2: Chemical constituents percentage of jute stick.

Jute fibre, jute stick and other fiber plants have attracted the attention of the cellulose preparation, principally because of the excellent quality of fibre. These annual fibre plants have 30-40% surface bast and 55-70% inside core. The external bust of the fibrous plant is denoted by a strong and long-fiber strand, and the internal core is made up of small fibers and pith. Both fiber of the whole plant gives good quality cellulose. **Table I** indicates the comparative analysis of cellulose, lignin, pentosan, ash, and fiber length of the jute fiber, jute stick, mesta fiber and stick and bamboo[14], however, the cellulose percentage of jute stick is lower than other fiber's cellulose. Besides that, the fiber length of jute stick is shorter than that of other. This is the advantage of making microcrystalline cellulose from a jute stick.

Table 1: Chemical composition of Jute, Mesta fiber and stick and Bamboo.

SL No.	Chemical Composition	Jute Fiber	Jute Stick	Mesta Fiber	Mesta Stick	Bamboo
1.	Cellulose (%)	58-62	38-42	60	38	57
2.	Lignin (%)	12-15	22-26	12.5	22.3	25
3.	Pentosan (%)	18-22	22	18	19.4	14
4.	Ash (%)	0.2-2	0.8	0.8	0.7	2
5.	Fiber Length (mm)	2.6	0.8-1.0	2.31	0.95	2.5

Acid hydrolysis of jute cellulose Sometimes cellulose polymer can be fragment when overheating at high temperature with dilute sulfuric acid to make shorter groups of molecules such as glucose and more degraded molecule hydroxymethylfurfural. Normally, high temperatures and highly concentrated acids are beneficial to liberate glucose from these tightly associated cellulose chains. The yields of liberated glucose are related to increasing temperature and acid concentration.[15,16] However, the temperature may be an important parameter for the acid hydrolysis of cellulose because, under these circumstances, cellulose turns to tars and other by-products which are much more difficult to handle (**Fig. 3**). It was revealed the production of MCC from the jute stick alpha-cellulose. So, for the production of MCC from biomass, always must be careful to apply the temperature and acid concentration.[17]

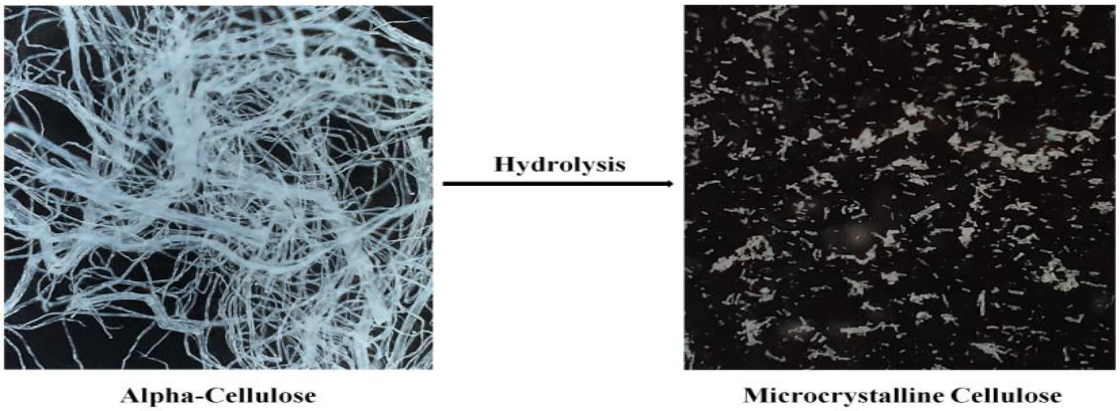

Figure 3: Hydrolysis of Alpha-cellulose.

Though, the modern consequences of the flow of hot water through the biomass that is compressed during the reaction to reduce empty space show close to theoretical yields of MCC from acid hydrolysis of cellulose in pretreated biomass. Regrettably, no commercially viable equipment has yet been demonstrated to duplicate this performance.[18,19]

The absorptions of approximately 3450.85cm^{-1}, 2911.41cm^{-1}, 1655.45cm^{-1}, 1466.12cm-1, 1371.25cm-1, 1308.85cm^{-1}, 1190.55cm^{-1}, 1050.24cm^{-1}, 943.42cm^{-1}, and 840.42cm^{-1}, exhibited in all spectra (a, b, c, d) were associated with the characteristics of native cellulose.[20] A broad and intense peak at around 3450.85cm^{-1} is the OH stretching vibration of all jute sticks' cellulose.[21,22] The band at 2911.41cm^{-1} is attributed to the C-H stretching vibration of all hydrocarbon constituents in polysaccharides.[23,24] Peaks were observed in the region of 1655.45cm^{-1} for water molecules that were absorbed by cellulose, which is related to the bending mode of the water.[25] The 1466.12cm^{-1} absorption band was associated with intermolecular hydrogen attraction in the C6 group and the 1371.25cm^{-1} observed peak was associated with the bending vibration of cellulose C-H.[26] Besides that, the appearance peak in the range of 1308.85cm^{-1} in all the sample spectra was attributed to the skeletal vibration of the C-C and C-O groups. The peaks observed at 1190.55cm^{-1} and 1105.30cm^{-1} were associated with the stretching vibration of C-O groups in cellulose. In addition, the strong absorption peak of the sample in the range of 1050.24cm^{-1} indicated a skeletal vibration of the C-O-C pyranose ring skeleton in the cellulose.[27] The β-type glycosidic linkages in jute stick cellulose were attributed to the indicative absorption peaks at 840.82cm^{-1} for all samples (**Fig. 4**).

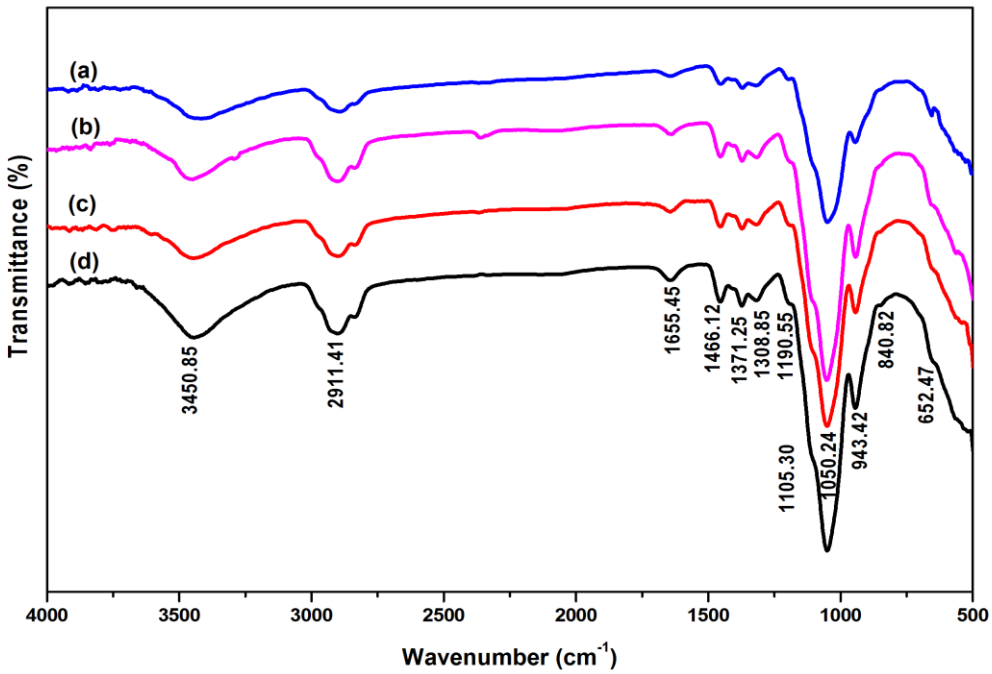

Figure 4: FTIR spectra of jute stick microcrystalline cellulose.

Crucial information about the thermal properties and degradation behavior of the microcrystalline cellulose under consideration in this study (**Fig. 5(I)**). The weight loss of these celluloses has three stages. In the first stage, the decomposition range is 50–110^{o}C, which is attributed to the loss of moisture and possibly of some extractives. Early weight loss at 100^{o}C represented 8.57%, 8.05%, 11.03%, and a slightly higher value of 13.22% for samples a, b, c, and d, respectively, which may be the hydrophilic character of the jute stick celluloses. Organic matter decomposition occurred in the second stage at temperatures ranging from 110 to 260^{o}C due to the breaking of C-C and C-O bonds. This stage, weight loss was 31.43%, 31.80%, 30.19%, and 29.09% for samples a, b, c, and d, respectively, and the maximum weight loss rate was reached at 260-450^{o}C where almost all cellulose pyrolyzed into ash.[28] Whereas, the ashes were obtained at 1.22-1.37% at 450^{o}C in all of the cases (**Fig. 5(II)**). On the above results, it can be assumed that all MCC samples made from jute sticks have excellent thermal stability.

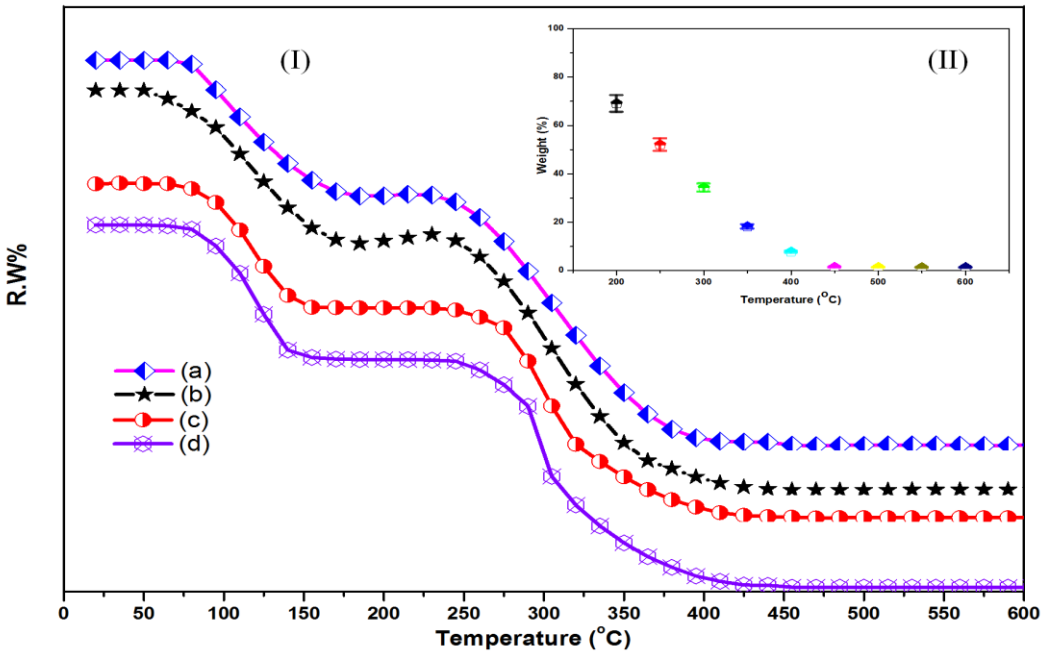

Figure 5: Thermogravimetric graph of jute stick microcrystalline cellulose.

CONCLUSION

The physical and chemical properties of MCC from tossa jute sticks were analysed where the organic matter decomposition occurred in the range of 100-260^{o}C and ash was obtained at 450^{o}C. The produced Alpha-cellulose and MCC in present can be applicable in various fields such as coatings, food, pharmaceuticals, adhesives, cosmetics, membranes, films, explosives, tobacco, and the textiles industry. It may confer from the results that produced product has good impact in global market but further research is needed to fulfil the cost effective of the final product.

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Declaration: The authors declare that they have no conflict of interest.

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