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A Method for Microwave Assisted Synthesis of Microcrystalline Cellulose from Jute Stick Alpha Cellulose

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ABSTRACT

In India, a huge quantity of jute (*Corchorus* spp.) stick is being produced as a primary by-product of jute fibre (economic part) cultivation every year, and the common practice in vogue is to burn such resides as firewood for domestic energy purposes. Present study aimed to utilize this jute stick alpha cellulose as raw material for synthesis of microcrystalline cellulose (MCC) under microwave radiation. The samples were characterized by FTIR and degree of polymerization. Effect of various mineral acids under microwave radiation were also tested. This study reported a new method for preparing MCC from jute stick alpha cellulose by microwave radiation (650W for 45 min at 80 °C). The order of finer MCC was HCl>HNO_3> H_2SO_4 whereas, the yield order was HCl>H_2SO_4> HNO_3. The developed MCC could be explored for pharmaceutical and other applications.

Highlights

- A microwave assisted method for preparation of MCC from jute stick alpha cellulose was developed.
- Mineral acids had strong influence on the properties of MCC developed.
- Finer MCC was obtained in the order of HCl>HNO_3> H_2SO_4

Keywords: Jute stick cellulose, Microwave, Microcrystalline cellulose

Microcrystalline cellulose (MCC) is a partially depolymerised cellulose prepared by treating α-cellulose, obtained as a pulp from fibrous plant with mineral acids. Microcrystalline cellulose is used as a texturizer, an anti-caking agent, a fat substitute, an emulsifier, an extender, and a bulking agent in food production. The most common form is used in vitamin supplements or tablets for pharmaceutical application. The global demand of MCC was around 50,000 T in 2003 (http://www.asahi-kasei.com/asahi/en/news/2002/e030213.html). Out of which 40 and 60% were shared by the food and pharmaceutical industries, respectively. Presently around 50, 25 and 25% of global MCC demand is from North America, Europe, and, Asia-Africa-others, respectively. The global demand for MCC is growing spontaneously. During 11th August- 11th Sept, 2014, India exported MCC worth USD 3,081,648 and the major importing countries were Brazil, Vietnam, Yemen, UAE etc (http://www.infodriveindia.com/india-import-data/microcrystalline-cellulose-import-data.aspx). Average value per shipment of microcrystalline cellulose exports in India is USD 47,460. Whereas, during 11th Aug 2014 to 11th Sept 2014, India imported microcrystalline cellulose worth USD 9,483,007. Average value per shipment of microcrystalline cellulose imports in India is USD 790,251. The huge difference between import and export, clearly indicated the national and international demand of MCC. The conventional source of MCC is derived from both hardwoods and softwood. However, due to national and international rules related to deforestation issue, researches have started looking for new alternative raw source of MCC preparation

(Ahmed *et al.* 2014). In recent times, several other alternative sources had also been studied like rice husk (Rosa *et al.* 2012), bamboo (Pachuau *et al.* 2014), water hyacinth (Murigi *et al.* 2014), corn cobs (Azubuike, C.P. and Okhamafe, 2012), oil palm (Haafiz *et al*. 2013) etc.

In India, a huge quantity (around 4 Mt per annum) of jute (*Corchorus* spp.) stick is being produced as a primary by-product of jute fibre (economic part) cultivation (Nayek *et al.* 2013). However, the common practice in vogue is to burn such resides as firewood for domestic energy purposes. Though, some small-scale applications include utilization in betel vine cultivation, preparation of particle board and handicrafts. Now the question comes that whether jute stick, the waste materials from jute cultivation and industries, could be explored and utilized as a resource material for better application. Low lingo-cellulosic residues are used for fodder and composting purposes, however, the high lingo-cellulosic residues are treated as 'trouble material'. Generation of huge crop residue/biomass is not only problematic for jute cultivation, but for other crops across India also. Residue generated by different crops was grouped in four categories based on the type of crop, namely cereals (rice, wheat, maize, jowar, bajra, ragi and small millets), oilseeds (groundnut and rapeseed mustard), fibers (jute, mesta and cotton) and sugarcane. The residue to grain ratio varied 1.5–1.7 for cereal crops, 2.15–3.0 for fiber crops, 2.0–3.0 for oilseed crops and 0.4 for sugarcane. Total amount dry crop residue generated by nine major crops was 620.4 Mt. Crop residues (C:N< 20:1) are used as animal feed, thatching for rural homes, residential cooking fuel and industrial fuel.

However, a large portion of the crop residues is not utilized and left in the fields. The disposal of such a large amount of crop residues is a major challenge. To clear the field rapidly and inexpensively and allow tillage practices to proceed unimpeded by residual crop material, the crop residues are burned in situ. According to Street *et al.* (2003) approximately 730 Tg of biomass burned annually, from both anthropogenic and natural sources in Asia with 18% contribution from India. Farmers opt for burning because it is a quick and easy way to manage the large quantities of crop residues and prepare the field for the next crop well in time. Utilization of jute stick for new utilities can increase the economic return from jute cultivation system.

As a result of numerous studies by various researchers across the world, various methods for conversion of alpha cellulose to MCC were reported. It included mostly physical (ultrasonication), chemical (acidolysis) and biological (enzymatic) means of conversion to MCC. The chemical pathway via acidolysis has been reviewed and reported most frequently. The present study aimed to prepare MCC from jute stick derived alpha-cellulose by acidolysis under microwave irradiation. Microwave irradiation is known for various chemical reaction (Kushnir*et al.* 2015). Effect of various acids (H_2SO_4, HNO_3 and HCl) on yield and degree of polymerization of synthesized MCC was also investigated.

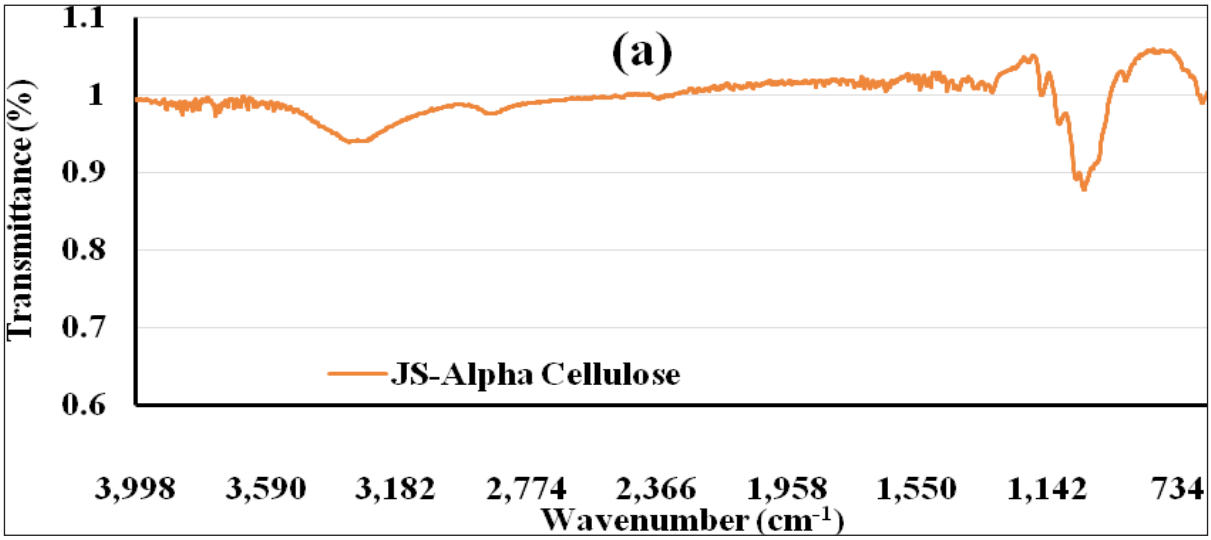
MATERIALS AND METHODS

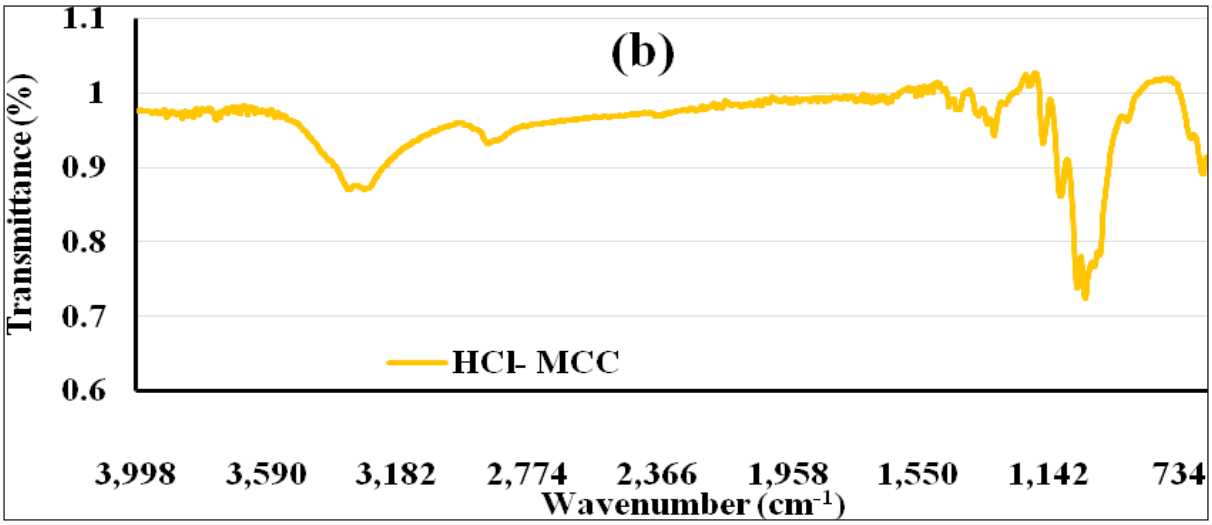
Alpha cellulose from jute stick was collected from CBP laboratory, ICAR-NIRJAFT, Kolkata, India. The purity was >99%. Analytical grade chemicals namely mineral acids (H_2SO_4, HNO_3 and HCl) were procured from Merck India. De-ionized water was used in experimental studies.

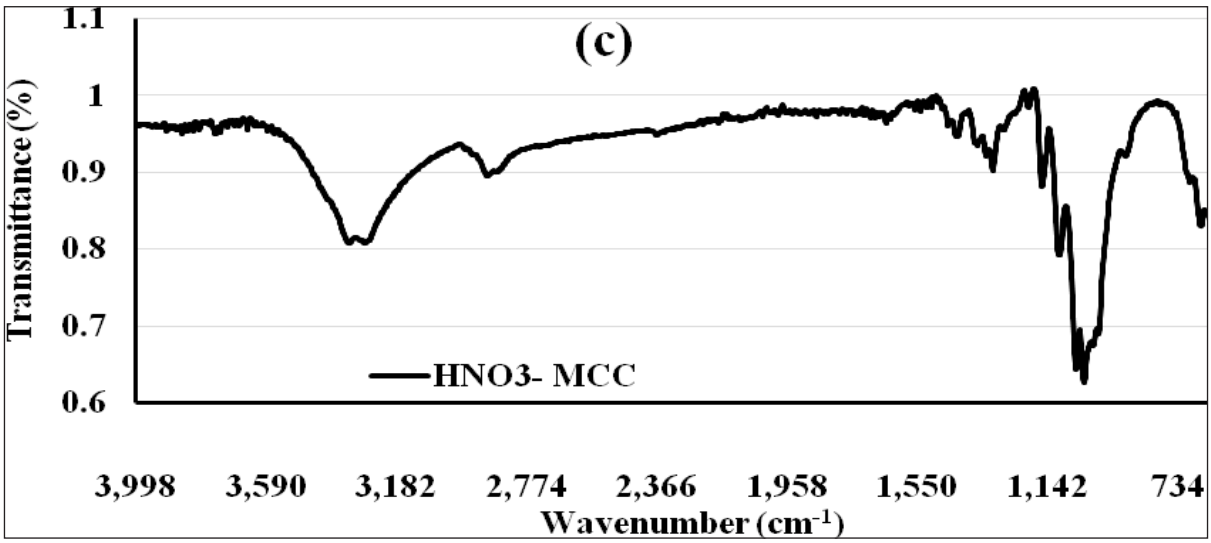
Jute stick alpha cellulose was mixed different mineral acid solutions at solid to solution ratio of 1:20 (w/v). The samples were heated at 80 ^oC under microwave irradiation of 650W for 45 min. The Microwave treatments were carried out with a microwave digester (Catalyst system, Pune, India). Then samples were washed to neutral pH and analysed for degree of polymerization (DP). The number-average DPn was calculatedas the ratio of glucosyl monomer concentration determined by the phenol-sulfuric acid method divided bythe reducing-end concentration determined by the modified 2,2'-bicinchoninate (BCA) method (Zhang and Lynd, 20015). All chemicals used in modified BCA method were of analytical grade and supplied by Himedia, India.

RESULTS AND DISCUSSION

The conversion of alpha cellulose to MCC varied with varying the mineral acids, while all other condition under microwave radiation remained same. MCC yields were 84.6, 81 and 79.3% for HCL, H_2SO_4 and HNO_3, respectively. FTIR analysis was used to study the physico-chemical and conformational properties of developed MCCs.


(a)
Transmittance (%)
1.1
1
0.9
0.8
0.7
0.6
JS-Alpha Cellulose
3,998
3,590
3,182
2,774
2,366
1,958
1,550
1,142
734
Wavenumber (cm-1)


(b)
Transmittance (%)
1.1
1
0.9
0.8
0.7
0.6
HCl- MCC
3,998
3,590
3,182
2,774
2,366
1,958
1,550
1,142
734
Wavenumber (cm-1)


(c)
Transmittance (%)
1.1
1
0.9
0.8
0.7
0.6
HNO3- MCC
3,998
3,590
3,182
2,774
2,366
1,958
1,550
1,142
734
Wavenumber (cm-1)

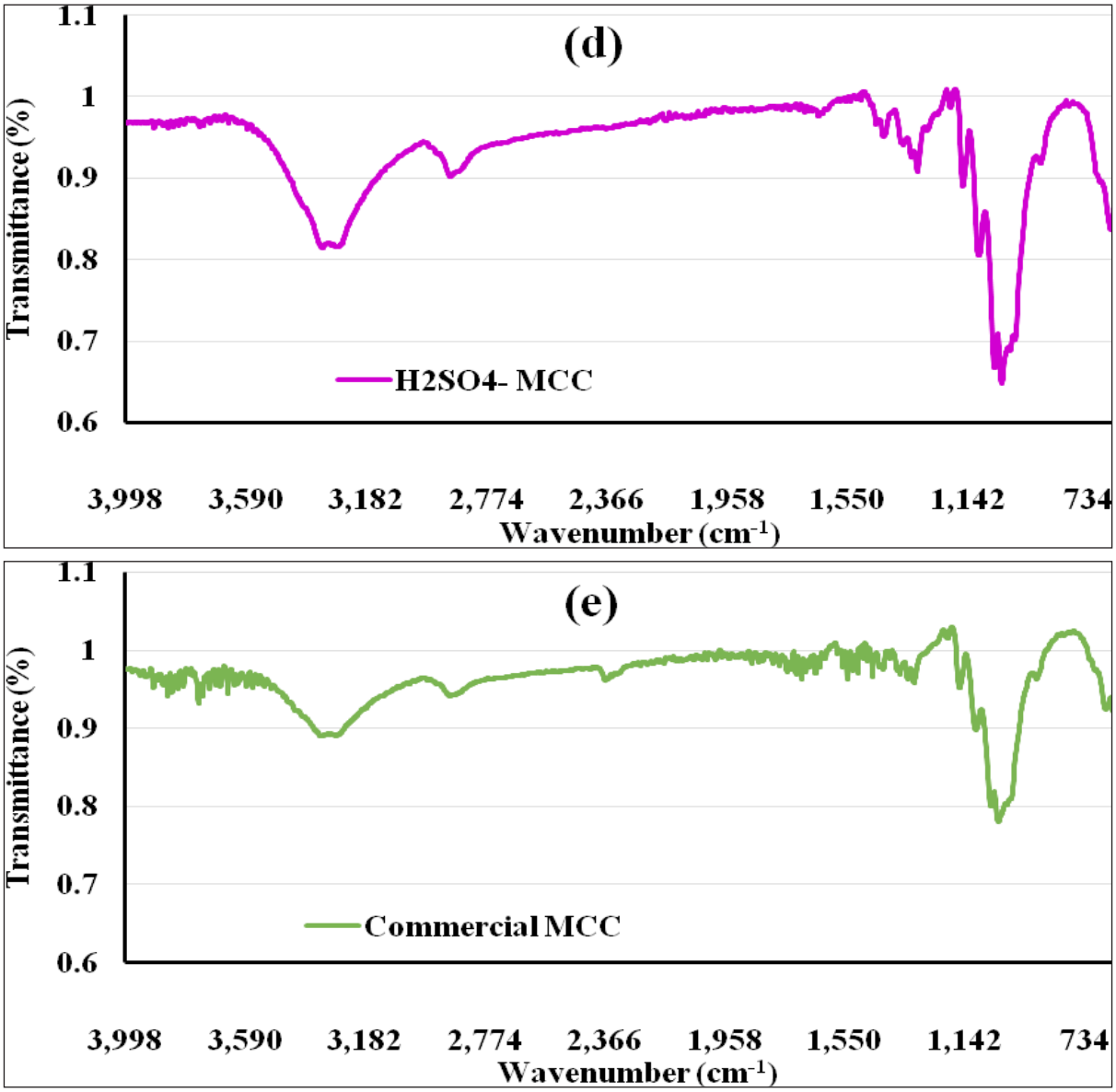


Fig. 1: FITR analysis of (a) jute stick alpha cellulose, (b) MCC from HCl acidolysis, (c) MCC from HNO_3 acidolysis, (d) MCC from H_2SO_4 acidolysis pathway and (e) commercial MCC

FTIR showed the changes in the functional groups present in the structure of cellulose (Fig. 1). Peaks at 3200 – 3450 cm^{-1} are assigned to stretching of –H bond of –OH group. The study showed that theses absorption of these peaks in MCCs were higher as compared to that of the parent alpha cellulose. Further, peaks at 1430, 1158, 1109, 1025, 1000 and 970 cm^{-1} are typical characteristic peaks of cellulose. Peak around at 2885-2890 cm^{-1} is attributed to the C–H stretching. Peak due –C–O– stretching of the carboxyl and acetyl groups inhemicelluloses of lignocellulosic biomasses was not absent which indicated the purity of the samples. The peaks around 1420-1428 and 1362-1370 cm^{-1} were characteristic for the asymmetric –CH2 bending and wagging. Peak around 1150-1158 cm^{-1} was associated with the –C–O–C– stretch of the β-1,4- glycosidic linkage of cellulose. This band has a lower intensity in R-MCC spectrum which could be due to the presence of trace amounts of non-cellulosic constituents in R-MCC. The absorption peak at 892 cm^{-1} is assigned to –C–O asymmetric stretching. A weak shoulder around 700 cm^{-1} was associated with the Iα (tricyclic) cellulose. The data clearly indicated that during chemical conversion under the microwave radiation the intensity of all these peaks increased as compared to the initial raw jute stick cellulose. The data indicated the purity of

synthesized MCCs as compared to the commercial MCC. These samples were analysed for estimation of DP. The DP also varied with varying mineral acids. The DP were 100, 140, 149and 212 for HCL, HNO_3, H_2SO_4 and commercial MCC, respectively. Therefore, the order of finer MCC was HCl>HNO_3> H_2SO_4 whereas, the yield order was HCl>H_2SO_4> HNO_3. Table 1 showed comparative analysis of MCC prepared from different sources via-vis present products.

Table 1: Comparative performance of MCC from various resources

Raw material	Degree of polymerization	Reference
MCC from book paper	200-207	Okwonna 2013
MCC from groundwood/ newsprint	196-202	
MCC from paperboard	186-195	
Rice bean hull	190	Adel *et al.* 2011
Rice husk	163	
Indian bamboo	181	Ngozi *et al.* 2014
Commercial MCC	150-220	
Jute stick cellulose	100-212	Present work

CONCLUSION

The present study indicated that microwave radiation can be successfully used for converting alpha cellulose to MCC. The DP of MCC varied with nature of mineral acids. However, under studied condition of microwave irradiation of 650W for 45 min at 80 ^oC resulted best MCC by HCL acidolysis from JS- alpha cellulose. The FTIR analysis and DP testing indicated purity of the products as compared to commercial one.

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