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Muli bamboo (*Melocanna baccifera)* as a new source of microcrystalline cellulose

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ABSTRACT

Microcrystalline cellulose (MCC) was prepared by hydrochloric acid hydrolysis from Muli bamboo (*Melocanna baccifera*) fibers to tap its potential as a green source of MCC. The cellulose and α-cellulose yield from the original material were 62.5 and 54.8 % respectively. The physicochemical properties of the prepared MCC were investigated using Avicel PH101 as a comparator. Micromeritic properties of the powder MCC samples were analyzed by determining its average particle size, flow properties, particle porosity and density. The total ash and moisture content along with the swelling index were also determined. The prepared MCC was also characterized by fourier transformed infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and x-ray diffraction spectroscopy (XRD). Results from these analyses indicate that the Muli bamboo can be used as a green source of MCC.

INTRODUCTION

Microcrystalline cellulose (MCC) is a purified, partially de-polymerized form of cellulose occurring as a fine, free flowing crystalline powder.MCC has been used for many years in different industries like pharmaceuticals, cosmetics, plastics, food etc. In pharmaceutical industries it is widely used as binder and diluent for tablets and capsules preparations. MCC is one of few materials that have the ability to produce adequately hard, yet rapidly disintegrating tablets mainly due to the swelling of its particles and consequent decrease of the bonding forces holding them together. MCC is listed as generally recognized as safe (GRAS) by the FDA and material obtained from natural sources is safe, stable and physiologically inert (Rowe *et al.*, 2006; Ejikeme, 2008). Cellulose is an abundant, naturally occurring polymer and several approaches have been applied to prepare MCC from different sources (Ohwoavworhua and Adelakun, 2005; Ohwoavworhua *et al.*, 2007; Yang *et al.*, 2008; Das *et al.*, 2010; Jahan *et al.*, 2011; Oliveira *et al.*, 2011), all of them yielding different types of microfibrillar material. Difference in chemical composition and structural organization may be the reasons for obtaining different yields of α-cellulose and crystallinity of the MCC produced (Ohwoavworhua and Adelakun, 2005).

Polymers derived from plants especially those from non-food sources are attracting increased attention in recent years due to their environmental compatibility, unique physical properties and low cost. Bamboo, a naturally occurring composite material growing abundantly in tropical countries offers great potential as a green source for MCC preparation. It has also been reported that the cellulose and α-cellulose content of bamboo is comparable to that of softwoods and hardwoods which are the commercial sources for MCC (Han and Rowell, 1996). Moreover, compared to most wood species, bamboo is cheap and fast growing with comparable physicochemical properties which makes it an ideal alternative to these woods (Yang *et al.*, 2008).

The present study reports the preparation and evaluation of MCC produced from Muli bamboo (*Melocana baccifera*) bamboo collected from the state of Mizoram, India. Muli bamboo is found mainly in India, Bangladesh, Myanmar and cultivated in many Asian countries. It is mainly utilized in paper manufacturing

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industries as a source of cellulose and recently it was also reported to be a potential alternative source of raw material for the manufacturing of plywood materials (Ashaduzzaman *et al.,* 2011). Mizoram, one of the northeastern states of India has abundant natural bamboo resources. About 31% of the state's total forest area is covered by bamboo, of which 90 % belongs to *Melocana baccifera* (Chenkual *et al.,* 2011). The bamboo forest of this state also contributes to about 14 % of the annual Indian bamboo harvest. Therefore, the objective of the present work is to explore the potential of manufacturing MCC from *Melocana baccifera* bamboo and characterizing it for its tableting and other properties.

MATERIALS AND METHODS

Materials

The raw bamboo (*Melocana baccifera*) of about 4 years old was collected from Mizoram (RIPANS/Pharm/Bam/01/2013) and was identified by Professor SK Tripathy, Department of Forestry, Mizoram University as *Melocana baccifera* (Roxb.) Kurz (Poaceae) from the bamboo repository maintained at the University. Microcrystalline cellulose (Avicel PH101) and Bis(ethylenediamine) copper (II) hydroxide solution were procured from Sigma-Aldrich (India). All other chemical and reagents were of analytical grade and used as supplied. De-ionized water was used for all the experiment (Resistivity of the water 18.2 meghaohms/cm produced from Smart2pure, Thermo Scientific, USA).

Methods

Preparation of microcrystalline cellulose

The isolation of cellulose from the bamboo fiber was performed according to the method reported earlier (Abe and Yano, 2009). The raw bamboo was cut into fine pieces using a fine-tooth hacksaw blade and passed through mesh 60. About 50 g of the sample was then dewaxed in a Soxhlet apparatus with 300 ml of 2:1 (v/v) toluene/ethanol mixture for 6 hours. Lignins, hemicelluloses and residual starch and pectin were removed through sequential chemical treatment. First, about 50g the dewaxed sample was delignified with sodium chlorite-acetic acid treatment (0.6 g/g dry biomass of sodium chlorite and 0.6 ml/g dry biomass of acetic acid, the volume made upto 1.5 L with deionized water) at 70 °C for 1 hour which was filtered and washed several times with water. This was followed by treatment at 90 °C with 2 % w/v potassium hydroxide (KOH) solution for 2 hours, filtered and washed with water. It was the treated again with sodium chlorite-acetic acid solution at 70 °C for 1 hour as done previously and finally the sample was heated at 90 °C for 2 hours with 5 % w/v KOH solution. Sample was filtered and rinsed with distilled water until the residue was neutral to litmus paper. Sample was the dried overnight at 50 °C in a tray drier (Tray Drier, OVFU, Indo Scientific & Surgicals, Kolkata). The efficient removal of lignin was monitored during the treatment following the method of Matrone et al (1946) which showed a negative brown color signifying the removal of lignin during the treatment. To extract the α-cellulose, about 30 g of the dried sample was treated with 1 L 17.5 % NaOH at 80 °C for 1 hour. The sample was filtered and repeatedly washed with distilled water until neutral to litmus and dried in tray drier at 50 °C overnight. Microcrystalline cellulose was prepared by following the method of Ejikeme (2008). About 25 g of the α-cellulose obtained was subjected to hydrolysis with 0.5 L 2.5 M HCl at 105 °C for 15 minutes to hydrolyze the amorphous regions of the cellulose. The MCC prepared was collected by filtration, washed with distilled water to neutral pH and dried at 50 °C in a tray drier overnight. The percent yield of MCC was determined and identification test was performed with Iodine solution as described in the Indian Pharmacopoeia (2007).

Degree of polymerization (DP) and intrinsic viscosity

The DP was determined by measuring the viscosity of the microcrystalline cellulose in Bis(ethylenediamine) copper (II) hydroxide solution following the method described in the European Pharmacopoeia (15). Briefly, about 1.300 g of M-MCC was taken in a reagent bottle and dispersed in 25 ml of deionized (resistivity of the water 18.2 meghaohms/cm) water followed by the addition of 25 ml of Bis(ethylenediamine) copper (II) hydroxide solution. The solution was immediately purged with nitrogen gas and stoppered. It was then shaken to completely dissolve the M-MCC after which the viscosity was measured with a Ubbelohde viscosimeter at 40 °C (ASTM D-446, Zenith Glasswares & Instruments Coorporation, Kolkata with constant 132.228336 at 40 °C). The measured flow time was the multiplied by the apparatus constant to a kinematic viscosity and the same procedure was repeated for Bis(ethylenediamine) copper (II) hydroxide solution diluted appropriately as above but without the M-MCC. Dividing the kinematic viscosity of the M-MCC by the kinematic viscosity of Bis(ethylenediamine) copper (II) hydroxide, the relative viscosity was obtained. Then the intrinsic viscosity, $[\eta]_c$ was determined by interpolation using the intrinsic viscosity table from where, DP was calculated by the following equation (European Pharmacopoeia, 2005):

$$DP = \frac{95[\eta]_c}{m[(100-b)/100]} \quad \text{.....1}$$

Where *m* is the mass in grams of the substance to be examined and *b* is the loss on drying as a percentage.

To confirm the accuracy of the DP calculated from the table, a graph data on reduced viscosity of the solution at different concentrations of the MCC (between 0.02 and 0.1 g/dl) was plotted from which an intrinsic viscosity was obtained. The DP was then calculated from the equation $DP^{0.905} = 0.805\ [\eta]\ (ml/g)$, where [η] is the intrinsic viscosity (Le Moigne *et al.,* 2010) which showed the two DP calculated are in complete agreement.

Average particle size

The particle size of the MCC was determined by microscopic method (at 10X, Medstar, Labovision, India). The ocular micrometer was calibrated using stage micrometer and each division of the ocular micrometer was measured in micrometer.

Particles were measured along major axis and about 100 particles were counted and done in triplicate.

Moisture content

Moisture content was expressed as percentage weight loss on drying (% LOD). About 2 g of MCC sample was weighed and oven dried at 105°C for 5 h to a constant weight. The experiment was done in three replications and an average of the three replicates was taken. The percent loss on drying was then calculated (Rankell *et al.*, 1986).

Sulfated ash content

The silica crucible used for the determination was heated to redness for 10 minutes and its weight was taken after it cooled down. About 1 g of the MCC samples were accurately taken in the crucible and the total weight was taken again. The samples were thoroughly charred by ignition. It was moistened with 1 ml of concentrated sulphuric acid (98%) and gently heated until the fumes did not evolve. This was followed by ignition at 800 ℃ in a muffle furnace (ACM 82301, ACMAS Technocracy, New Delhi) for 6 hours. Sulphuric acid treatment and ignition were repeated until two successive weighing do not differ more than 0.5 mg (Indian Pharmacopoeia, 2007). The sulfated ash content was expressed as a percentage ratio of the weight of the ash to the oven dry weight of the powdered MCC.

Swelling index

The swelling index was determined on a graduated measuring cylinder of 25 ml capacity taking water as a medium. About 1 g of the sample was taken into the measuring cylinder and water was added making the volume up to 25 ml. The cylinder was shaken every 10 minutes for 1 hour to wet the sample thoroughly. After 24 hrs, readings were taken and the swelling index was calculated using the initial and swollen height of the MCC.

Where H_0 is the initial height of the MCC in the graduated measuring cylinder and H_f is the height of the swollen MCC in the measuring cylinder recorded after 24 hours.

Angle of repose (θ)

Angle of repose (θ) was measured using a fixed height funnel fitted at the height of 10 cm from the base (The funnel is 60 °, 10 cm in diameter, 0.7 cm internal stem diameter with 9.6 cm stem length). About 20g of the dried powder was allowed to flow through the funnel into the base and a pile was formed at the base. The angle of repose was then calculated from the height, h and radius, r of the pile.

Compressibility index

The compressibility index of the powder was determined according to Carr's index and Hausner ratio after determining bulk and tap densities. About 20 g of the dried powder was taken into 50 ml graduated measuring cylinder and the initial volume (V_0) was recorded. The cylinder was then tapped 100 times using bulk density apparatus (ACM-157, Acmus Technocracy, New Delhi) to achieve a final volume (V_f). The bulk density was calculated from the initial volume and tap density from the final volume after hundred tappings. Carr's index and Hausner ratio were then determined (Nep and Conway, 2011).

True density

The true density of the powder was determined by liquid displacement method taking xylene as the immersion liquid at 20 °C. The true density for both the prepared microcrystalline cellulose and Avicel PH101 were determined according to the following equation (Ohwoavworhua and Adelakun, 2005):

$$\rho_t = \frac{w}{[(a+w)-b]} \; x \; SG \quad \text{.....2}$$

Where 'w' is the weight of the powder, 'SG' is the specific gravity of the solvent at 20 °C 'a' is the total weight of the bottle and the solvent, 'b' is the total weight of the bottle, solvent and the powder.

Porosity

The porosity (ε) of the test powders was derived from the values of the true and bulk densities fitted into the following equation (Kumar and Kothari, 1999):

$$\varepsilon = \left[1 - \left(\frac{\rho_{tap}}{\rho_{true}}\right)\right] x\ 100 \quad \text{.....3}$$

FTIR spectroscopy

FTIR spectrum was recorded on Alpha FT-IR spectrophotometer (Bruker, USA). Powder samples were taken and the absorbance at 32 scans to achieve an acceptable signal-to-noise ratio was recorded between 400 and 4000 cm^{-1} for both the prepared MCC sample and the standard Avicel PH101. The spectra thus obtained were analyzed for functional groups and compared to characterize the MCC samples.

Thermogravimetric analysis

TG analysis was performed on TGA (Pyris TGA, Perkin Elmer) between 40˚C - 855˚C at a heating rate of 10˚C/min while nitrogen purging was maintained at 20 ml/min. For each analysis, about 6 mg of the sample was taken into the aluminium sample pan and sealed. Empty aluminium pan was used as a reference and the thermogram was then recorded for the prepared M-MCC as well as the Avicel PH101.

XRD studies

XRD patterns were recorded on Philips analytical X'pert X-ray diffractometer (Philips Co., Netherland). The data were collected at 2θ between 5° and 45°. The degree of crystallinity was calculated and expressed as percentage crystallinity index using the equation (Das *et al.*, 2010):

$$Crystallinity\ Index\ (\%) = \frac{I_{002} - I_{am}}{I_{002}} \; x\ 100 \quad \text{.....4}$$

Where I_{002} is the counter reading at peak intensity at 2θ angle close to 22° representing the crystalline portion and I_{am} is the counter

reading at peak intensity at 2θ close to 18° representing the amorphous portion of the cellulose.

Scanning electron microscopy

The shape and surface characteristics of the MCC was analyzed by scanning electron microscopy (JEOL JSM-6300, Japan). Samples were mounted on the aluminium stub and photomicrographs of the powders were taken after sputter coating with a thin layer of gold. The quality of the MCC with respect to surface properties was studied.

Statistical analysis

Statistical analysis was performed using computer software SigmaStat 2.03 (SPSS, USA). One-way analysis of variance followed by Tukey Test was performed to compare the properties of the two MCC samples.

RESULTS AND DISCUSSION

Physicochemical properties

A white or almost white, free flowing MCC powder was obtained after acid hydrolysis of the α-cellulose obtained from the bamboo, *Meloccana baccifera*. The powder was tasteless and odorless which give a blue-purple coloration on identification test performed confirming the identity of MCC. The results of various physicochemical tests on the prepared MCC and the standard Avicel PH101 are summarized in Table 1. The yield of cellulose and α-cellulose was about 62.50 % and 54.8 % respectively from the original raw material as given in Table 2. The yield of M-MCC from the obtained α-cellulose was about 92.5 % which is about 46.68 % from the original material. The α-cellulose content of 54.8 % from the original Muli bamboo fiber is slightly higher than 46.4, 41.8 and 39.8 % reported for wood, Moso bamboo and wheat straw fibers respectively (Chen *et al.*, 2011). The final MCC yield of 46.68 % for M-MCC was also higher than the MCC yield of 25.3 % and 21 % reported for orange mesocarp (Ejikeme, 2008) and raw cotton (Ohwoavworhua and Adelakun, 2005) respectively while it was slightly less than 48 % reported for Jute (Jahan *et al.*, 2011). The Degree of polymerization (DP) of the M-MCC as determined by viscometric method was found to be 248.5. The FAO defines the DP of MCC to be typically less than 400 (Vanhatalo and Dahl, 2014) while the European Pharmacopeia (2005) said that it should not be more than 350. A plot of reduced viscosity vs concentration was also depicted in Fig. 1 which shows a linear graph. Extrapolation of the line intersects the y-axis at 1.796 dl/g or 179.6 ml/g which in the intrinsic viscosity [η]. Calculation of DP by substituting this value to the equation $DP^{0.905} = 0.805\ [\eta]\ (ml/g)$ shows the DP to be 245.5 which is in agreement with the value 248.5 obtained from the previous table.

Moisture content of the samples was determined at 105 °C for 3 hours and expressed as percent loss on drying (LOD). An excess of water in plant materials encourage microbial growth and deterioration by hydrolysis. When the moisture content of MCC exceeds 5 %, water molecules act as plasticizer affecting the mechanical properties of the MCC which results in lower tensile strength of MCC tablets (Wu *et al.*, 2001). Limit of moisture content for MCC has been set at 6 % by the Indian Pharmacopoeia (2007). The percent LOD determined on the Muli bamboo MCC (M-MCC) and Avicel PH101 were within this limit. Determination of the swelling index shows that both the standard Avicel and M-MCC swells on contact with aqueous media with Avicel showing slightly higher swelling index than the prepared M-MCC. Determination of swelling property is important particularly for MCC as its disintegration property is mainly attributed to the swelling of MCC particles and the decrease of bonding forces holding them together.

Table 1: Physicochemical properties of Avicel PH101 and M-MCC.

Powder property	Avicel PH101	M-MCC
Moisture content (%LOD)	4.22 ±0.44	5.25±0.64
Swelling index	24.00±2.11	23.08±1.84
Sulfated ash (%)	0.12±0.01	0.14±0.01
Average particle size (µm ± SD)	69.11±24.29	128.26±40.42
Bulk density (g/ml)	0.39±0.02	0.35±0.05
Tap density (g/ml)	0.52±0.12	0.55±0.06
True density (g/ml)	1.47±0.04	1.89±0.02
Porosity (%)	66.90±1.33	70.89±2.12
Carr's Index	23.73±2.04	36.05±1.66
Hausner ratio	1.311±0.45	1.56±0.51
Angle of repose	31.61±1.06	53.92±3.21
Crystallinity index	72	68

Table 2: Cellulose fractions yield of M-MCC in comparison with other sources

Source	Cellulose	α-Cellulose	MCC
Muli (*Melocanna* baccifera) bambo	62.50	54.8	46.68
Cotton (*Cochlospermum planchonii*)[1]	-	32	21
Orange mesocarp[2]	-	62.5	25.3
Moso bamboo[3]	-	41.8	-
Wood[3]	-	46.4	-
Wheat straw[3]	-	39.8	-
Jute[4]	59.8	97.8% of cellulose	48-52.8

[1]*Ohwoavworhua and Adelakun, 2005;* [2]*Ejikeme, 2008;* [3]*Chen et al., 2011;* [4]*Jahan et al., 2011*

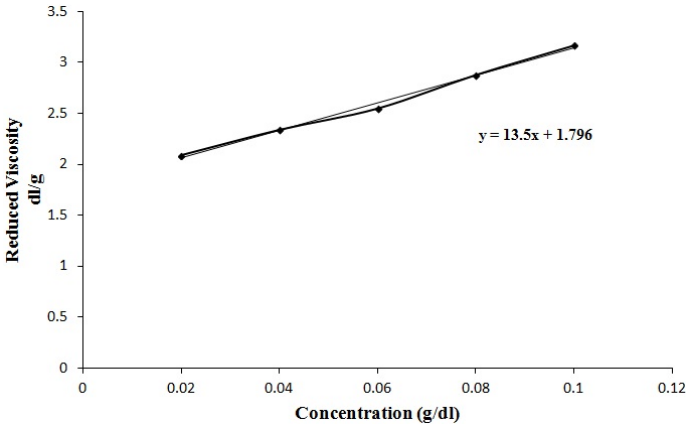

Fig. 1: Reduced viscosity vs concentration plot.

The sulfated ash test measures the amount of residual substance which is not volatilized from a sample when the sample is ignited in the presence of sulfuric acid. The test is usually used for

determining the content of inorganic impurities in an organic substance. The Indian Pharmacopoeia set the total amount of sulfated ash for MCC to be not more than 0.2 % (Indian Pharmacopoeia, 2007). Therefore, both the samples were found to be within the pharmacopoeial limit of MCC.

Micromeritic properties of MCC such as particles density, angle of repose, average particle size and powder flow characteristics are important indicator for functionality of the MCC products. The flow property of the MCC powder is essential in determining the suitability of its use in pharmaceutical products especially as a direct compression excipient. The bulk density of the M-MCC was found to be less than the standard Avicel PH101. A higher bulk density would result in a lower loading volume during tablet manufacturing. Bulk density determined for both the standard and M-MCC was found to be comparable with the values reported for MCC obtained from other sources (Ejikeme 2008, Ohwoavworhua and Adelakun, 2005). The average particle size as determined by microscopic method was found to be 69.11±24.29 μm and 108.26±30.42 μm for Avicel and M-MCC respectively. There was statistically significant differences in the average particle size ($p<0.01$) between the M-MCC and Avicel PH101. Small particle size and low moisture content results in higher bulk density (Korhonen *et al.,* 2002). Therefore, the smaller average particle size and lower moisture content of the Avicel may be the reason for its higher bulk density. The true density of M-MCC was found to be slightly higher than the standard Avicel. It was reported earlier that there is a direct correlation between the true density of the particles and the degree of crystallinity (Ejikeme, 2008). Results from true density determination suggest that the degree of crystallinity for both the samples would be very close. However, detail X-ray diffraction study has to be performed to confirm the degree of crystallinity of the samples.

Compressibility index, Hausner ratio and angle of repose provides information on the powder flowability for which, a small values indicate better flowability (Wu *et al.,* 2001). The compressibility and Hausner indices were determined from the tap and bulk densities. Compressibility index of less than 15 % and angle of repose less than 35 ° are indicative of very freely flowing powders (Ejikeme, 2008). Avicel was found to exhibit smaller compressibility index and angle of repose indicating and confirming its better flow property. During tabletting, enhancement of flowability of the powders or granules would decrease the weight variation of tablets resulting in the production of uniform tablets.

Scanning electron micrographs (SEM)

Morphology of the MCC powders was studied through scanning electron micrograph as it showed particle size in micrometer range and the photomicrographs are depicted in Fig. 2. The particles size distribution of M-MCC fibers is observed to be uneven and narrow channels were formed on the surface and most of the particles in M-MCC were rod shaped with rough surface. The Avicel PH101 was found to show uniform particle sizes and much more spherical in shape than the prepared M-MCC. The obvious morphological difference of M-MCC and Avicel PH101 was probably due to difference in the source of raw materials and difference in the method of MCC preparation.

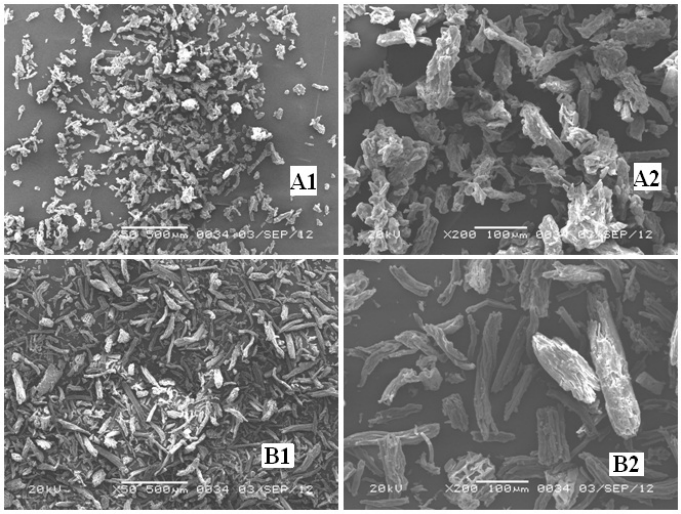

Fig. 2 : Scanning electron micrographs of microcrystalline cellulose (A1 & A2 Avicel PH101; B1 & B2 M-MCC).

FTIR spectroscopy

FTIR spectroscopy is a powerful tool for studying the physicochemical and conformational properties of polysaccharides. FTIR spectra for both the samples were recorded for analysis of functional groups present in the structure. Absorption at 1430, 1158, 1109, 1025, 1000 and 970 cm^{-1} are typical absorption peaks of cellulose (Yang *et al.,* 2008). These peaks are observed in the spectra of both the MCC samples as depicted in Fig. 3. Peaks at 3200 – 3450 cm^{-1} are assigned to stretching of –H bond of –OH group. This –OH stretching peaks are observed at 3275 cm-1 and 3277 cm^{-1} for Avicel and M-MCC sample respectively. This stretching vibration of –OH in the cellulose structure become narrow in the spectrum of M-MCC sample indicating that the acid hydrolysis process weakened the hydrogen bonding of cellulose (Xiong *et al.,* 2012). Peak at 2887 cm^{-1} for Avicel and 2886 cm^{-1} for M-MCC is attributed to the C–H stretching. Small peak appearing at 1729 cm^{-1} for M-MCC was due to the –C–O– stretching of the carboxyl and acetyl groups in hemicelluloses of the M-MCC fibres (Jahan *et al.,* 2011). This particular peak was not observed in the case of Avicel indicating the purity of the Avicel sample. A small peak appeared at 1638 cm^{-1} and 1657 cm^{-1} in Avicel and M-MCC respectively due to the adsorbed water. The bands at 1424 and 1363 cm^{-1} in Avicel spectrum and 1449 and 1364 cm^{-1} in M-MCC spectrum were attributed to the asymmetric –CH2 bending and wagging. Peak associated to the –C–O–C– stretch of the β-1,4- glycosidic linkage in cellulose was observed at 1154 and 1156 cm^{-1} for Avicel and M-MCC respectively. This band has a lower intensity in M-MCC spectrum which could be due to the presence of non-cellulosic constituents in M-MCC (Das *et al.,* 2010). The absorption peak at 892 cm^{-1} is assigned to –C–H out of plane in position ring stretching in cellulose due to β-linkage (Haafiz *et al.,* 2013). The

weak shoulder at around 750 cm^{-1} was assigned to the Iβ (monocyclic) cellulose and the absorption peak at 699 cm^{-1} was assigned to the presence of Iα (tricyclic) cellulose.

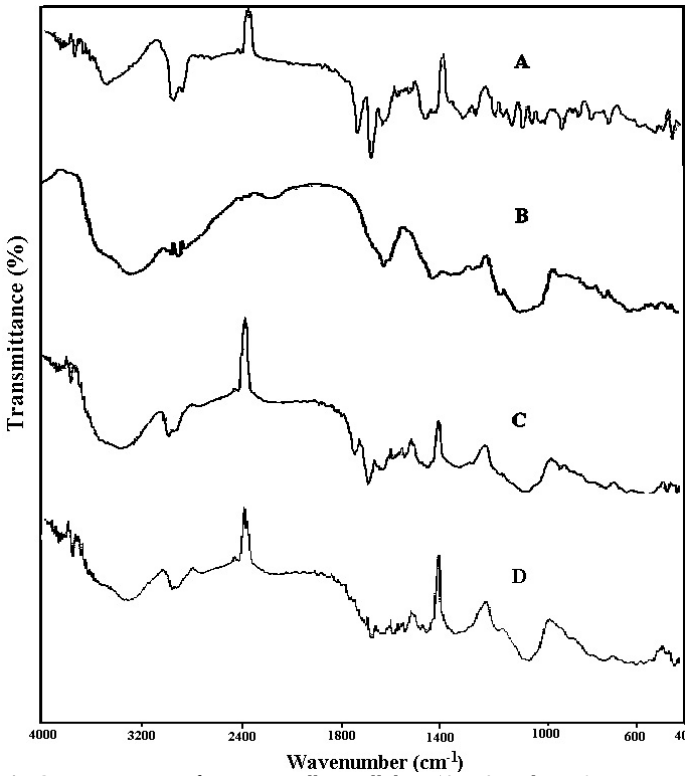

Fig. 3: FTIR spectra of microcrystalline cellulose (A – Avicel PH101; B – M-MCC).

Thermogravimetric analysis (TGA)

TG analysis was performed to investigate the thermal stability and purity of the prepared MCC and compared it to the thermal properties of the Avicel PH101. It is a well-established set of techniques for obtaining qualitative and quantitative information about the effects of various heat treatments on materials and has been proved to be effective in analysis and characterization of lignocellulosic materials. Thermal properties are critical for many applications including the use of MCC for the production of biocomposite where the processing temperature may rise above 200 °C (Jahan *et al.,* 2011). Analysis of TG curve (Fig. 4) for M-MCC fiber showed a first thermal event occurring between 0-104.66 °C where there was about 4.411 % weight loss was observed. This event can be attributed to the evaporation of water and results from moisture content determination also supported this observation. The first thermal event for evaporation of water was also observed for Avicel sample between 0-90.83 °C where 3.479 % weight loss occurred. Thermal weight loss occurring in the range of 200-400 °C was attributed to depolymerization of cellulose (Xiong *et al.,* 2012). This second thermal event for M-MCC was observed between 250.42 – 381.08 °C during which about 60 % weight loss took place. The second thermal event for Avicel occurred at 284.67 – 386.00 °C resulting in 83.450 % weight loss.

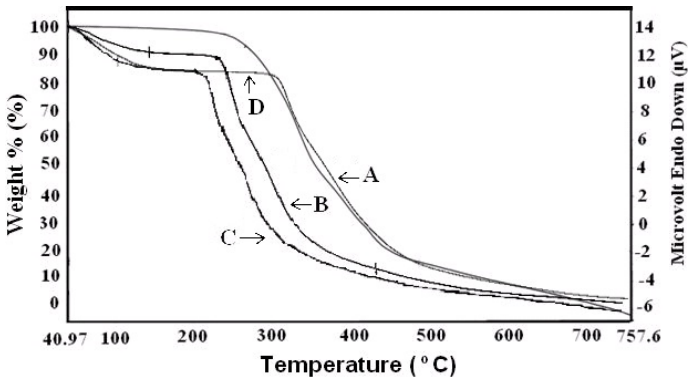

Fig. 4: TG thermogram for A- Avicel PH101, B – M-MCC.

At 400 °C almost all the cellulose was pyrolyzed and the solid residuals were about 20 % and 8 % respectively for M-MCC and Avicel. These percent of weight remaining after heating to about 400 °C in both the samples could be however, almost completely volatilize after sulfuric acid treatment and ignition in presence of sulfuric acid at higher temperature in the range of 800 °C as evident from the low percentage of sulfated ash remaining. According to earlier reports, natural bamboo fiber has maximum thermal degradation at about 365.1 °C and regenerated bamboo fiber showed maximum at about 339.9 °C (Liu *et al.,* 2012). This higher thermal stability of the natural bamboo fiber than reinforced or mercenarized bamboo fiber is probably due to the removal of lignin during material preparation such as in preparation of MCC. The maximum thermal degradation for the sample was observed at 315.74 °C and 335.34 °C for M-MCC and Avicel respectively indicating the higher thermal stability of the Avicel PH101.

X-ray diffraction

MCC is typically characterized by a high degree of crystallinity, ranging between 55 – 80 % as determined by X-ray diffraction spectroscopy (Haafiz *et al.,* 2013). Powder X-ray diffraction patterns for M-MCC and the standard Avicel PH101 are shown in Fig. 5.

Cellulose I, represented by native cellulose showed typical peaks at 15, 16 and 23°2θ due to 1 $\bar{1}$0, 110 and 002 reflections respectively, while Cellulose II types represented by mercenerized and regenerated cellulose exhibited typical peaks at about 12, 20 and 22°2θ due to 1 $\bar{1}$0, 110 and 002 reflections respectively (Kumar and Kothari, 1999).

As evidence from the figure, the two materials exhibit slightly different X-ray diffraction patterns which may be due to the difference in the source of the MCC. The resultant diffraction peaks in M-MCC showed a typical Cellulose II peaks and the degree of crystallinity calculated are given in Table 1. The diffraction pattern observed for M-MCC prepared from the bamboo cellulose is the result of having mercerized it with the strong NaOH extraction. The degree of crystallinity, calculated for both the MCC value showed a slightly higher value for Avicel PH101 than the prepared M-MCC sample. The diffraction pattern for Avicel PH101 also showed Cellulose I peaks which is usually a

characteristic of wood cellulose. The degree of crystallinity calculated for Avicel PH101 was also found to be in agreement with the values found in earlier reports (Oliveira *et al.*, 2011; Nep and Conway, 2011).

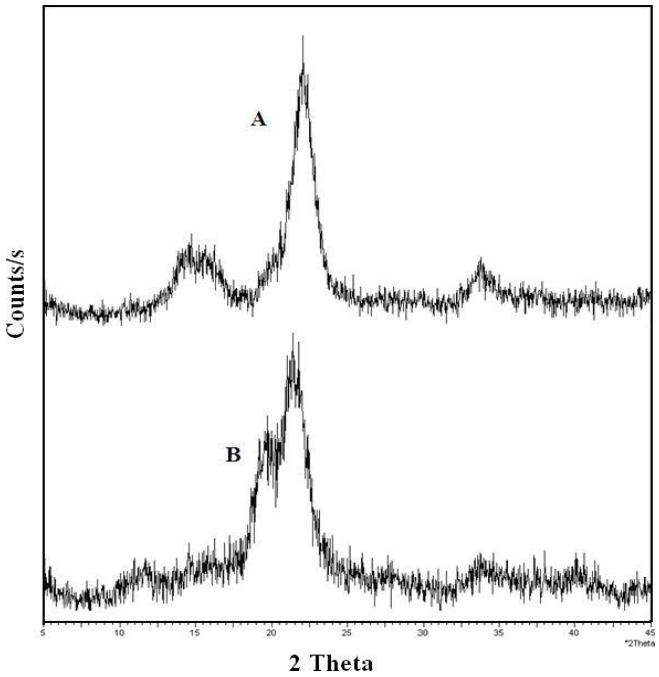

Fig. 5: XRD of A- Avicel PH101, B – M-MCC.

CONCLUSION

MCC was produced successfully from the Muli (*Melocanna baccifera*) bamboo fibers. The M-MCC product conformed to the official specifications in the Indian Pharmacopoeia (2007).

Compared to the standard Avicel PH101 there was statistically significant differences in micromeritic properties of the powder which could be attributed to the differences in the source of material and method of preparation. However, considering that the yield of α-cellulose was comparable to, if not higher than, other sources and the availability of the bamboo, the Muli bamboo (*Melocanna baccifera*) could be a potential green source of MCC.

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