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Research Article

**EXTRACTION, CHARACTERIZATION OF MICRO
CRYSTALLINE CELLULOSE OBTAINED FROM CORN
HUSK USING DIFFERENT ACID ALKALI TREATMENT
METHOD****Roshni Vora* and Yamini Shah**

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Abstract:

The aim of this work was to prepare low-cost and suitable microcrystalline cellulose [MCC] powder from corn husk [CH]. Extraction of microcrystalline cellulose was performed by pulping of husks with different acids [Hydrochloric acid, Sulfuric acid, and Nitric acid], alkali [Sodium Hydroxide] treatment and bleaching. The morphology of the treated microcrystalline cellulose was investigated using scanning electron microscopy [SEM], which showed a compact structure and sharp surface. Fourier transform infrared [FT-IR] spectroscopy indicated that characteristic peaks of all prepared microcrystalline cellulose [Hydrochloric acid, Sulfuric acid, and Nitric acid] samples and Marketed product [Avicel PH101] were similar. As per X-ray diffraction [XRD] crystallinity index of the produced microcrystalline cellulose ranged from 73% to 79%. The resultant excipient obtained from above mentioned method demonstrated strong thermal stability. Authenticity of the microcrystalline cellulose was proved by comparing physico chemical and micromeritic properties with Avicel PH101.

Keywords: *Microcrystalline cellulose, corn husk, physicochemical properties, micromeritic properties and thermal properties.*

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INTRODUCTION:

Each year, farming and agricultural processing generate millions of tones of lignocellulosic biomass, such as corn cobs and husks, groundnut shells, rice straw, banana stems, soy hulls and sugar beet pulp and sugar cane bagasse [1- 4].

Cellulose is a classical example of a renewable and biodegradable structural plant polymer which can be processed into whisker-like micro fibrils [5-7]. Cellulose is the world's most ubiquitous and abundant naturally occurring polymer which is produced by plants, as well as by microorganisms. It shows remarkable reinforcing capability, excellent mechanical properties, low density and environmental benefits [8-15].

Micro-crystalline cellulose is described as 'purified, partially depolymerized cellulose' prepared by treating cellulose obtained as a pulp from fibrous plant material with mineral acids [16]. Commercially available microcrystalline cellulose is derived from hard wood and purified cotton. In India there is a need for searching of cheaper sources of microcrystalline cellulose as hard wood is expensive. This has led to the search for other lignocellulosic waste materials based on agricultural residues [17-20]. As a result, several types of microcrystalline cellulose are available in the market with different physicochemical and thermal properties, and therefore, they will have different functional parameters and applications. These differences can affect their functional properties when employed in pharmaceutical formulations [21].

Microcrystalline cellulose [MCC] is a fine, white, odorless, crystalline powder and a biodegradable material, which can be isolated from cellulose. MCC is typically characterized by a high degree of crystallinity, although there are variations between grades; values typically range from 55 to 80% as determined by X-ray diffraction [22, 23].

Isolation of microcrystalline cellulose has been carried out from jute, rice husk cellulose by using the acid alkali hydrolysis [Sulfuric acid] and bleaching approach [26- 32]. They also reported that MCC can be used as suspension stabilizer, a water retainer in cosmetics, food and universal filler for the extrusion/spheronization process in pharmaceuticals industries. [33-35].

In this study we have compared Avicel PH 101 with all CH-MCC [H_2SO_4 , HCl, HNO_3] samples and examined the physical properties along with their structural and thermal properties with view to ascertain its potential as a pharmaceutical excipient.

MATERIAL AND METHODS:

Materials

A corn husk, an agricultural waste was collected by the local farmers in Gujarat, India. Avicel PH 101, sodium hydroxide [NaOH] [reagent grade, 98%], hydrochloric acid [HCl] [ACS reagent, 37%], Sulphuric Acid [H_2SO_4], Nitric acid [HNO_3] and

Calcium hypo-chlorite [technical grade] were supplied by Sigma-Aldrich. Aqueous solution of sodium hypochlorite was prepared by dissolution of calcium hypochlorite and sodium hydroxide in water, with subsequent filtration of the calcium hydroxide precipitate formed.

Method

1Kg corn husk was treated with water for removal of trash at 60°C for 3 hours and pulverized to husk powder. 900g of the powdered corn husk was delignified with 500 ml of a 2 N NaOH at 80°C in a glass container for 2 hours. A 300 g of each batch of powdered corn husk was treated with 500 ml of 1 N H_2SO_4 , HCl and HNO_3 in a glass container in autoclave at 90°C for 45 minutes. The residue was washed, filtered, and bleached with a 1L of 3.5% w/v sodium hypochlorite at 55 °C for 30 min. Residual slurry was washed with distilled water until it was neutral to litmus paper. The resultant cellulose was dried at 60°C for 6 hr.

Characterization

pH determination

2 g of the powder material shaking with 100 ml of distilled water for 5 min and the pH of the supernatant liquid was determined using a pH meter [Corning, model 10 England] [36].

Chemical Evaluation of MCC

The following tests were conducted on the produced MCC, to confirm the identity of extracts [37].

a. Test for the Presence of Lignin

100 mg of obtained MCC was placed on a glass slide and moistened with concentrated hydrochloric acid, two drops of phloroglucinol was added and heated and slide was thereafter examined under light microscope for any coloration.

b. Test for the presence of sugar

Standard I.P test for free reducing sugar was conducted on the extract.

c. Test for the presence of starch

To 0.2g of obtained MCC few drops of iodine solution was added, followed by addition of concentrated sulphuric acid and change in color was noted.

Scanning Electron Microscopy

Scanning electron microscopy [gold coating, Edwards Sputter Coater, UK] was performed using a Joel 6310 [Joel Instrument, Tokyo, Japan] system running at 10 KeV.

Infrared Spectroscopy

Fourier transform infrared [FT-IR] spectra were recorded using KBr discs on a Perkin-Elmer FT-IR spectrometer. The scanned range was 4,000 to 400 cm^{-1}

Powder X-Ray Diffraction

X-Ray Diffraction patterns of the cellulose samples were obtained using an x-ray diffractometer [Philips Xpert Mpd]. Samples for analysis were

prepared by pressing the powder into the cavity of a sample holder and smoothen with a glass slide. The crystallinity index [CI] was calculated using equation 1.

$$CI = [I_{002} - I_{am}] / I_{002} * 100 \dots\dots 1$$

Where I_{002} is the maximum intensity of the principal peak [002] lattice diffraction, and I_{am} is the intensity of diffraction attributed to amorphous cellulose [38].

Thermal Analysis

The thermal properties of the cellulose samples were investigated by TGA and DSC on a simultaneous thermal analyzer [Mettler-Toledo AM, Greifensee, Switzerland]. Samples weighing between 6 and 10 mg were used. Each sample was heated from room temperature to 500°C at a rate of 5°C/min under nitrogen.

Molecular weight and Degree of Polymerization Determinations

The molecular weight of the MCCs was determined using oswald viscometer [39]. Water was used as a blank solution as well as the solvent for the solution. All determinations were done at 25°C. The time taken for water to flow through the viscometer was determined using a stopwatch. The viscosity of each was calculated using the equation 2.

$$\eta_1 / \eta_2 = t_1 / t_2 \dots\dots 2$$

The specific viscosity [η_{sp}] of the polymer solution was calculated from the equation 3.

$$\eta_{sp} = \eta_2 / \eta_1 - 1 \dots\dots 3$$

The intrinsic viscosity [η_0] was calculated from the graphical form of the Huggins equation 4. [40]

$$\eta_{sp} / C = \eta + K_H [\eta]^2 C \dots\dots 4$$

Where K_H is Huggin's constant

The molecular weight was calculated using the integral form of Mark-Houwink equation 5.

$$\eta = KM v^a \dots\dots 5$$

where k and a are constants characteristic of the polymer-solvent temperature system. K ranges from 0.5 and 5×10^{-4} .

The degree of polymerization was then calculated using the equation 6.

$$D_p = M / M_o \dots\dots 6$$

M = molecular weight of the material and M_o = Molecular weight of glucose

Determination of Yield

The microcrystalline cellulose obtained by different acid treatment was weighed and the yield was calculated using equation 7.

$$Yield [\%] = A / B \times 100 \dots\dots 7$$

A [mg] = Weight of obtained microcrystalline Cellulose and B [mg] = Weight of alpha cellulose

Particle Size Analysis

Using a light compound microscope the particle size of hundred particles was determined. The average particle size of MCC was however calculated statistically [41].

Bulk and Tapped Density

For the determination of the bulk and tapped densities, the methods reported in an earlier

study were adopted [42]. Briefly, 25 g of the cellulose powder was accurately weighed and poured into a 100-mL graduated cylinder. The cylinder was Stopped and the bulk volume [V_b] was recorded. For tapped density, the cylinder was tapped from a height of 2.5 cm on a hard surface to a constant volume [i.e. until no more settling of the material occurred]. The final [constant] volume [V_t] was noted to be the tapped volume. The bulk density, D_{bulk} , and tapped density, D_{tap} , were determined using equations 8 and 9.

$$D_{bulk} = W / V_b \dots\dots 8$$

$$D_{tap} = W / V_t \dots\dots 9$$

Carr's Index and Hausner Ratio

Carr's index [42] and Hausner ratio [43] for cellulose were calculated from bulk and tapped densities using equations 10 and 11.

$$Carr's Index = \{ [D_{tap} - D_{bulk}] / D_{tap} \} * 100 \dots\dots 10$$

$$Hausner's Ratio = D_{tap} / D_{bulk} \dots\dots 11$$

Angle of Repose

The static angle of repose was measured according to the fixed funnel and free standing cone method [43]. A funnel was clamped with its tip 2 cm above a graph paper placed on a flat horizontal surface. The powders were carefully poured through the funnel until the apex of the cone thus formed just reached the tip of the funnel. The mean diameters of the base of the powder cones were determined and the tangent of the angle of repose calculated using the equation 12.

$$\Theta = \tan^{-1} [h / r] \dots\dots 12$$

Where h is height of heap of powder and r is the radius of the base of heap of powder.

Moisture Content

The Moisture Content [44] was determined using equation 13.

$$Moisture Content = \{ [Wet Wt. - Dry Wt.] / Wet Wt \} * 100 \dots\dots 13$$

Swelling Capacity

1.0 g of each sample was placed in four 15 ml plastic centrifuge tubes and 10 ml distilled water was added and then stoppered. The tubes were allowed to stand for 10 min and immediately centrifuged at 1000 rpm for 10 min on a Gallenkamp bench centrifuge. This was calculated using equation 14.

$$S = [V_2 - V_1] / V_1 \times 100 \dots\dots 14$$

Where S is the % swelling capacity, V_2 is the volume of the swollen material and V_1 is the tapped volume of the material prior to swelling [45].

RESULTS AND DISCUSSION:

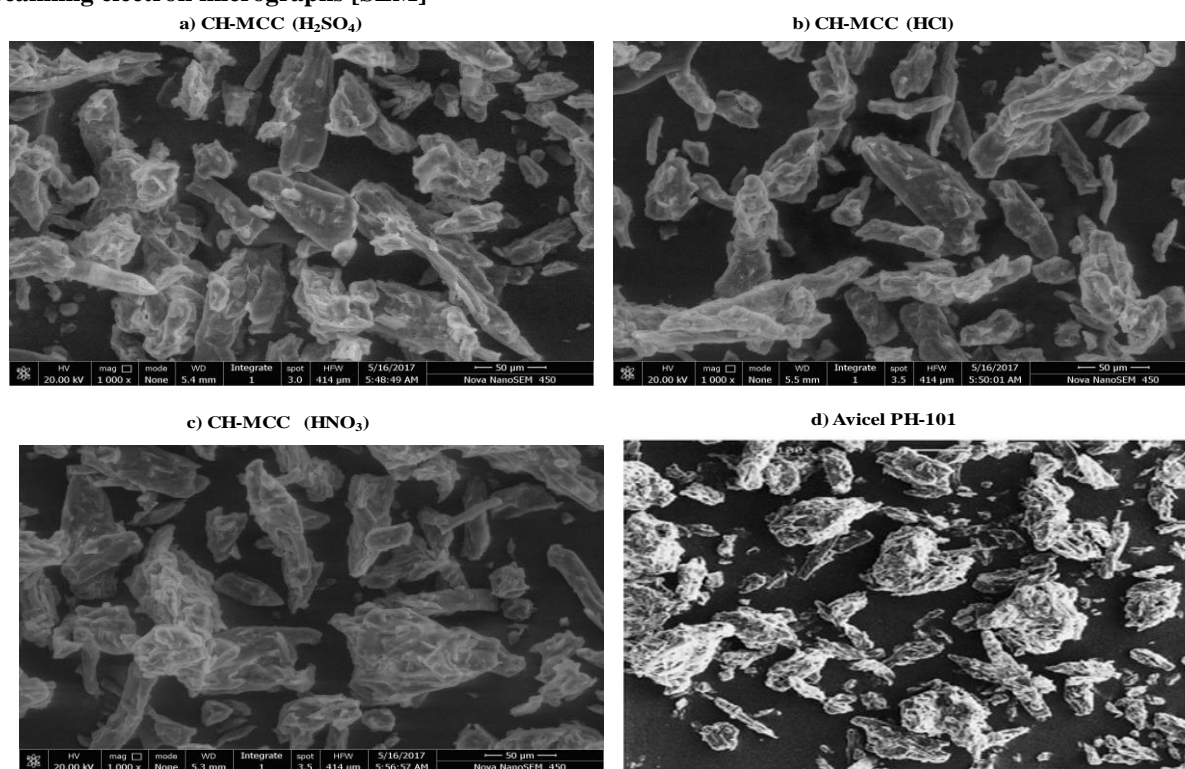
Physicochemical Properties

The physicochemical properties of CH- MCC prepared by different acid treatments [H_2SO_4 , HCl , HNO_3] are shown in Table 1. The Organoleptic properties of the CH-MCC produced were good as the material was odorless, tasteless, white granular powder and the pH was almost neutral 6.8 for all samples. [46]

Table 1: Physicochemical properties of CH- MCC

Organoleptic Properties				
Type of MCC	CH-MCC [H ₂ SO ₄]	CH-MCC [HCl]	CH-MCC [HNO ₃]	Avicel PH-101
Color	White	White	Off White	White
Odor	Odorless	Odorless	Odorless	Odorless
Taste	Tasteless	Tasteless	Tasteless	Tasteless
Appearance	Granular Powder	Granular Powder	Granular Powder	Granular Powder
pH	5.6	6.2	6.4	6.6
Lignin	Negative	Negative	Negative	Negative
Sugar	Negative	Negative	Negative	Negative
Starch	Negative	Negative	Negative	Negative
Solubility				
Disti. Water	Insoluble	Insoluble	Insoluble	Insoluble
Acetone	Insoluble	Insoluble	Insoluble	Insoluble
Ethanol	Insoluble	Insoluble	Insoluble	Insoluble
Dil. HCl	Insoluble	Insoluble	Insoluble	Insoluble

Scanning electron micrographs [SEM]

Fig. 1: SEM of [a] CH-MCC [H₂SO₄], CH-MCC [HCl], CH-MCC [HNO₃]

The morphology of CH- MCC after acid, alkali and bleaching was investigated using SEM and compared to Avicel PH101. Fig.1, table 5 shows individualized and uniform fibers, which correlates with the spectroscopic evidence for the removal of cementing material around the fiber bundles; namely hemicelluloses, and lignin [47, 48]. All the

prepared CH-MCC showed sharper edges, crystalline and transparent image while that of CH-MCC [HCl] showed similar sized uniform fibers.

According to previous studies, cellulose obtained from different sources and hydrolytic conditions differ in overall characteristics of MCC such as particle size and aggregation [49, 50].

FT-IR Spectroscopic Studies:

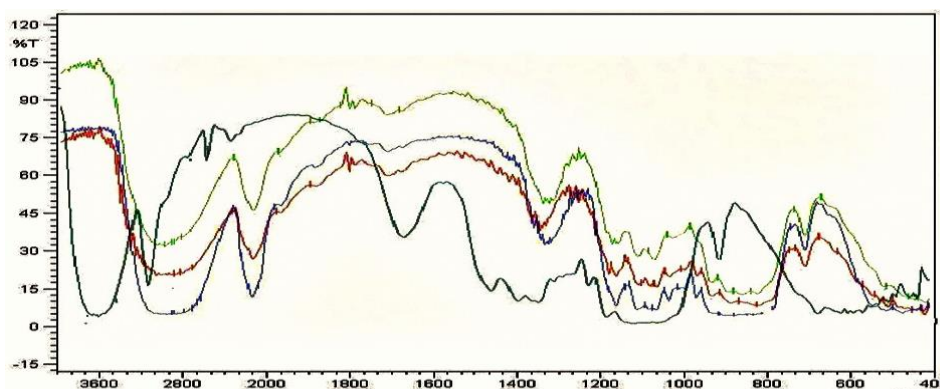


Fig 2. FTIR spectra. Black indicates Avicel PH101; green, CH-MCC [HNO₃]; blue, CH-MCC [H₂SO₄] and red, CH-MCC [HCl].

FT-IR spectra of CH-MCC [H₂SO₄, HCl, HNO₃] and Avicel PH101 are shown in Fig.2 and the spectral assignments are summarized in Table 2. CH-MCC [H₂SO₄, HCl, HNO₃] and Avicel PH101 showed a high wave numbers [2800–3500 cm⁻¹] and low wave numbers [500–1700 cm⁻¹], respectively [51,52]. which is an indication that all samples have similar chemical compositions. The broad absorption band located from 3400 to 3500 cm⁻¹ is due to stretching of –OH groups and an absorption at 2900 cm⁻¹ is related to CH₂ groups [53,51, 54].

According to other studies and present investigation, the absorption at 1645 cm⁻¹ is related to the bending modes of water molecules due to a strong interaction between cellulose and water [51,53]. The absorption band at 1425 cm⁻¹ is associated to the intermolecular hydrogen at the C6 [aromatic ring] group [55]. The absorption band at 1163 cm⁻¹ corresponds to C O C stretching, and the peak at 896 cm⁻¹ is associated to C H rock vibration of cellulose [anomeric vibration, specific for glucosides] observed in CH-MCC samples [47]. The absence of peaks located in the range 1509–1609 cm⁻¹, which would correspond to C-C aromatic skeletal vibrations, indicate the complete removal of lignin [51,52]. The absorption band which corresponds to either the acetyl or uronic ester groups of hemicelluloses normally appears in

X-ray diffraction studies

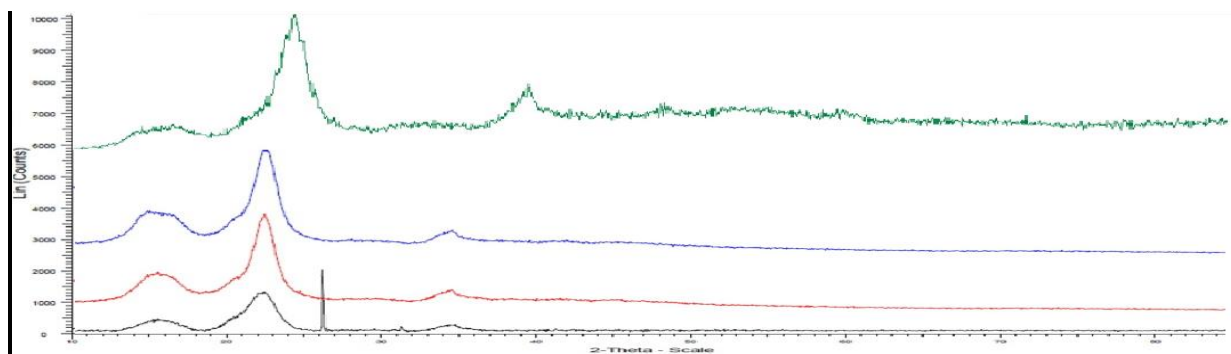


Fig 3: X-Ray Diffractograms Black indicates CH-MCC [H₂SO₄]; green, Avicel PH101; blue, CH-MCC [HNO₃], and red, CH-MCC [HCl].

the region 1700–1740 cm⁻¹, this band is absent, indicating the removal of hemicelluloses [51,47,57]. Similar results have been observed by [53] during production of MCC from jute fibers and [52].

Table 2: FTIR spectral assignments for all CH-MCC samples

Antisymmetric out-of-phase stretching vibration	895 cm ⁻¹
Ring vibration and C-OH bending	1,060 cm ⁻¹
Pyranose ring	1090 cm ⁻¹
C-O-C aryl-alkyl	1265 cm ⁻¹
O—H in plane bending vibration	1315 cm ⁻¹
CH ₂ due to crystalline nature	1415 cm ⁻¹
Intermolecular hydrogen bonds at the C group	1429, 1426 cm ⁻¹
–O- tensile vibration neighboring hydrogen atoms	1,650 cm ⁻¹
CH ₂	2854 cm ⁻¹
C-H asymmetric and symmetric tensile vibration	2,900 cm ⁻¹
Intermolecular O—H stretching vibration band	3327 cm ⁻¹
Intramolecular O—H stretching vibration band	3330 cm ⁻¹
Broad peak of OH stretching, hydrogen bonds	3,300 to 3,500 cm ⁻¹

The X-ray diffraction [XRD] patterns of CH-MCC [H_2SO_4 , HCl , HNO_3] and Avicel PH101 are presented in Fig 3. The crystallinity of each sample is listed in Table 5. The diffractograms of the CH-MCC [H_2SO_4 , HCl , HNO_3] samples of 101 and 002 has diffraction peaks of the 2θ angles at 10.24° , 13° , 13.68° and $21.16^\circ, 22.51^\circ$, 21.96° respectively. The lowest height between 002 peak and 101 peak is I_{am} and represents only the amorphous part. The value of the crystallinity index was calculated using eq-1 for CH MCC [H_2SO_4 , HCl , HNO_3] are 73, 78, 70 respectively which are very close to that found for Avicel PH101 i.e 83 %. [20,23] All XRD diffraction data suggested that the samples were highly crystalline, which can lead to a higher tensile strength to fibers [47, 51]. The crystallinity index for CH-MCC [H_2SO_4 , HCl , HNO_3] is high due to removal of hemicellulose and lignin, which existed in amorphous regions leading to

realignment of cellulose molecules [9, 25]. The crystallinity index gives a quantitative measure of the crystallinity in powders and can relate to the strength and stiffness of fibres. High crystallinity indicates an ordered compact molecular structure, which translates to dense particles, whereas lower crystallinity implies a more disordered structure, resulting in a more amorphous powder. These results mean that the overall multistep procedure employed is adequate to obtain highly crystalline cellulose [56].

Thermal Properties

The thermograms in Figures show that CH-MCC [H_2SO_4 , HCl , HNO_3] and Avicel PH101 follow similar degradation patterns. The numerical data from the TGA [Table 4, Fig 5] and of DSC [Table 3, Fig 4] also reveal that the CH-MCC [H_2SO_4 , HCl , and HNO_3] and Avicel PH101 have similar thermal properties.

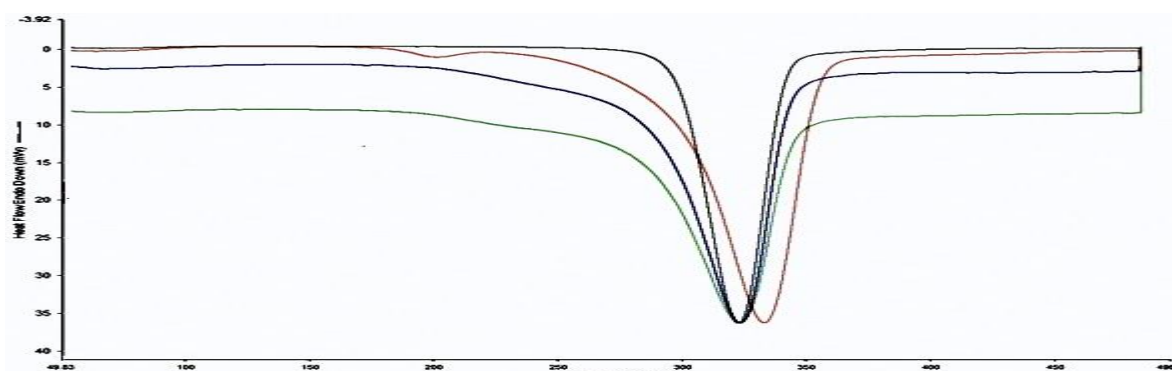


Fig 4: DSC thermograms. Black indicates Avicel PH101; green, CH-MCC [HNO_3]; blue, CH-MCC [H_2SO_4] and red, CH-MCC [HCl].

Table 3: Derived transition temperatures from DSC thermo grams of MCC

Parameter [$^\circ\text{C}$]	CH-MCC [H_2SO_4]	CH-MCC [HCl]	CH-MCC [HNO_3]	Avicel PH101
Onset	250	252	250	300
Midpoint	325	330	325	325
Inflection Point	350	360	350	350

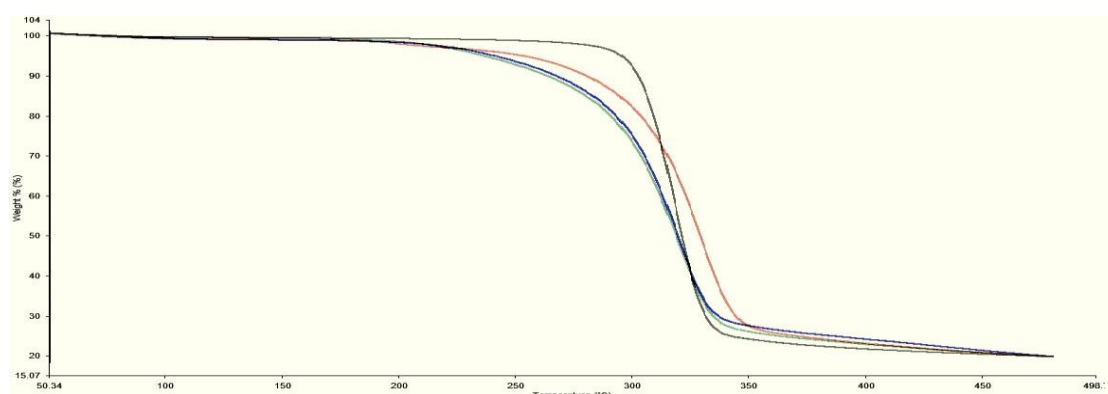


Fig 5: TGA thermograms. Black indicates Avicel PH101; green, CH-MCC [HNO_3]; blue, CH-MCC [H_2SO_4] and red, CH-MCC [HCl].

Table 4: Thermal properties obtained from TGA experiments for MCC

Parameter	CH-MCC [H ₂ SO ₄]	CH-MCC [HCl]	CH-MCC [HNO ₃]	Avicel PH101
T _{5% onset} [°C] ^a	300	238.57	252.29	244.07
T _{50%} [°C] ^b	328	328.93	339.25	330.75
W ₅₀₀ [%] ^c	6.36	17.214	18.436	19.113
^a onset temperature for 5% decomposition; ^b temperature at 50% weight loss; ^c residual char weight at 500°C				

The temperature, for which 5% of the mass of the sample is decomposed, T_{on 5%}, and the temperature at 50% weight loss, T_{50%}, is high and similar for all CH-MCC [H₂SO₄, HCl, HNO₃] and Avicel PH101. The char yield [non-volatile carbonaceous material generated on pyrolysis, which is indicated by the residual weight after the decomposition step] specified in Table 4 for the temperature 500°C, is higher for CH-MCC [HNO₃] as compared to CH-MCC [H₂SO₄] and CH-MCC [HCl]. Among the CH-MCC samples, CH-MCC [HNO₃] had higher residual char value at 500°C indicating higher nonvolatile carbonaceous material generated on pyrolysis [58].

The onset temperatures of the decomposition as well as the midpoint and inflection point

temperature data for all CH-MCC [H₂SO₄, HCl, HNO₃] and Avicel PH101 are similar and are presented in Table 3. The higher onset temperatures are associated with higher thermal stability. This behavior could be attributed to the high degree of crystallinity of the MCC. Cellulosic materials degrade at low to moderate temperatures [59]. It is observed that hydrolysis of cellulose not only dissolves the amorphous regions, but also some crystalline regions. The high char residue of CH-MCC is probably due to the presence of a higher amount of crystalline cellulose I which is intrinsically flame resistant [60].

It was concluded that the CH-MCC samples produced from corn husk has good thermal stability.

Micromeritic Properties

Table 5: Micromeritic properties of the CH-MCC samples

Parameters	CH-MCC [H ₂ SO ₄]	CH-MCC [HCl]	CH-MCC [HNO ₃]	Avicel PH 101
Bulk density [g/ml]	0.36	0.40	0.38	0.38
Tapped density [g/ml]	0.45	0.48	0.47	0.45
Carr's index [%]	20	16.66	19.4	15.55
	Fair	Fair	Fair	Good
Hausner index	1.25	1.2	1.23	1.18
	Fair	Fair	Fair	Good
Angle of repose	36.5	34.56	35.51	33.66
	Fair	Good	Fair	Good
Swelling capacity [%]	18	15	25	17.18
Moisture Content [%]	4.2	4.10	4.33	4.00
Particle Size [µm]	55	51	53	50
Yield [%]	39.3	42.33	40.13	
Molecular Weight	37120	35530	35781	36000
Degree of Polymerization	229.13	219.32	220.8	222.22
Crystallinity Index [%]	73	78	79	83

The yield of CH-MCC [H₂SO₄, HCl, HNO₃] are as shown in table 5, this is relatively high enough to stimulate large scale industrial processing of microcrystalline cellulose from corn husk as an agricultural waste.

The results of particle size analysis of the different CH-MCC samples are summarized in Table 5, the mean particle size of all prepared samples are comparable with Avicel PH101. The acid- alkali treatment affects the particle size in case of CH-MCC [H₂SO₄, HCl, and HNO₃].

There is no significant differences among the bulk and tap densities of CH-MCC [H₂SO₄, HCl, HNO₃] as reported in Table 5. Bulk density gives an estimate of the ability of a material to flow from a hopper into the die cavity of a rotary tablet compression machine, while tap density is a measure of how well a powder can be packed in a confined space on repeated tapping. In general, the higher the bulk and tapped densities, the better the potential for a material to flow and to re-arrange under compression. This suggests that all CH-MCC [H₂SO₄, HCl, and HNO₃] samples have good flow properties as comparable with Avicel PH101 [61].

The Carr's compressibility and Hausner indices were estimated as the ratios of the difference between tapped and bulk densities. The Carr's compressibility index gives an idea of how much a powder can be compressed, while Hausner index measures/estimates cohesion between particles; the values for both varies inversely with particle flow [62,63]. In this study, the compressibility for all the CH-MCC [H₂SO₄, HCl, HNO₃] samples are approximately in the range 15 to 20 [fair-good flowability]. On the other hand, the Hausner ratio for the microcrystalline cellulose samples lie around the threshold of 1.25 [≈fair flow-ability].

The angle of repose of a powder gives a qualitative assessment of its internal and cohesive frictions. Angles of up to 35° indicate good flow potential of the solid powders, whereas those samples with angles greater than 35° exhibit fair flow [64]. Here, CH-MCC [H₂SO₄, HNO₃] shows fair flow potential while that of CH-MCC [HCl] showed good result as Avicel PH101.

The moisture content measured for all CH-MCC [H₂SO₄, HCl, and HNO₃] fall within the acceptable limits of between 5% and 7%, [65]. Furthermore, powder flowability is known to decrease with increasing moisture content [66, 67].

Swelling capacity is generally accepted as an indication of tablet disintegration ability. The swelling capacity value was in the range of 15.62% to 25% [Table 5] indicates that CH-MCC is capable of absorbing water. It seems therefore, that only a small portion of absorbed water actually penetrated the individual cellulose particles causing them to swell [68].

The similarity in Physicochemical properties exhibited by both the prepared CH-MCC [H₂SO₄,

HCl, HNO₃] and the commercial Avicel PH101 proved that the prepared MCC in our laboratory can be a good indigenous substitute for oral solid pharmaceutical dosage forms.

CONCLUSION:

The results indicated that the production of pharmaceutical grade cellulose from Corn husk waste was technically feasible using acid, alkali and bleaching treatment. SEM shows individualized and uniform fibers with crystalline and sharper edges. The results obtained from FT-IR analysis confirmed that chemical structure of the cellulosic fragments is not influenced by the acid hydrolysis. The XRD and TGA analysis of CH-MCC [HCl] shows comparatively good crystallinity index and thermal stability than CH-MCC [H₂SO₄, HNO₃]. CH-MCC [HCl] gave a reasonable yield, along with good flow properties indicating that the flowability of CH-MCC [HCl] is adequate for it to be used as an excipient in tablet formulation. It was shown that the prepared CH-MCC [HCl], compared very well with Avicel PH 101, as well as conformed to the official specifications for MCC as per the IP [2007] and BP [2004]. Hence, CH-MCC [HCl] is a potential pharmaceutical excipient indigenous substitute for Indian manufacturers. Since farmers generate abundant corn husks as a waste material, it would ultimately be a cheaper source than the imported varieties.

REFERENCES:

1. Ruan R, Lun Y, Zhang J, Addis P, Chen P. Structure-function relationships of highly refined cellulose made from agricultural fibrous residues. *Appl Eng Agric*, 1996; 12:465–468.
2. Taherzadeh MJ, Niklasson C. 2004. Ethanol from lignocellulosic materials: pretreatment, acid and enzymatic hydrolyses and fermentation. *ACS Division of Cellulose and Renewable Materials*. Washington DC: American Chemical Society 49–68.
3. Pandey A, Soccol CR, Nigam P, Soccol VT. Biotechnological potential of agro-industrial residues. I. Sugarcane bagasse. *Bioresource Technol*, 2000; 74:69–80.
4. Richardson S, Gorton L. Characterisation of the substituent distribution in starch and cellulose derivatives. *Analytica Chimica Acta*, 2003; 497:27–65.
5. Abraham E, Deepaa B, Pothana LA, Jacobc M, Thomasb S, Cvelbard U. Extraction of nanocellulose fibrils from lignocellulosic fibres: A novel approach. *Carbohydrate Polymers*, 2011; 86:1468–1475.
6. Eichhorn, SJ. Cellulose nanowhiskers: Promising materials for advanced applications. *Soft Matter*, 2011; 7: 303–315.
7. Eichhorn SJ, Dufresne A, Aranguren M, Marcovich NE, Capadona JR, Rowan SJ. Review: Current international research into cellulose nanofibres and nanocomposites. *J of Material Sci*, 2010; 45: 1–33.
8. Klemm D, Heublein B, Fink HP, Bohn A. Cellulose: Fascinating biopolymer and sustainable raw material.

- Angewandte Chemie – International Edition, 2005; 44: 3358–3393.
- 9.Li R, Fei J, Cai Y, Li Y, Feng J, Yao J. Cellulose whiskers extracted from mulberry: A novel biomass production. *Carbohydrate Polymers*, 2009; 76: 94–99.
- 10.Fernandes AN, Thomas LH, Altaner CM, Callow P, Forsyth VT, Apperley DC. Nanostructure of cellulose microfibrils in spruce wood. *Proceedings of the National Academy of Sciences of the United States of America*, 2011; 108:1195–1203.
- 11.Nishiyama Y. Structure and properties of the cellulose microfibril. *J of Wood Science*, 2009; 55: 241–249.
- 12.Ranby BG. Cellulose and muscle – the colloidal properties of cellulose micelles. *Discussions of the Faraday Society*, 1951; 11:158–164 [discussion 208–213].
- 13.Bras J, Hassan ML, Bruzesse C, Hassan EA, El-Wakil NA, Dufresne A. Mechanical, barrier, and biodegradability properties of bagasse cellulose whiskers reinforced natural rubber nanocomposites. *Industrial Crops and Products*, 2010; 32: 627–633.
- 14.De Menezes AJ, Siqueira G, Curvelo AS, Dufresne A. Extrusion and characterization of functionalized cellulose whiskers reinforced polyethylene nanocomposites. *Polymer*, 2009; 50: 4552–4563.
- 15.Goetz L, Mathew A, Oksman K, Gatenholm P, Ragauskas AJ. A novel nanocomposite film prepared from crosslinked cellulosic whiskers. *Carbohydrate Polymers*, 2009; 75: 85–89.
- 16.Brittain HC, Lewen G, Newman AW, Fiorelli K, Bogdanowich S. Changes in material properties accompanying the national formulary [NF] identity test for microcrystalline cellulose. *Pharm Res*, 1993;10[1]:61–67.
- 17.Paralikar KM, Bhatwadekar SP. Microcrystalline cellulose from bagasse pulp. *Biological Wastes*, 1988; 24:75–77.
- 18.Uesu NY, Pineda EAG, Hechenleitner AAW. Microcrystalline cellulose from soybean husk: effects of solvent treatments on its properties as acetylsalicylic acid carrier. *Int J Pharm*, 2000; 206:85–96.
- 19.Ohwoavworhua FO, Adelakun TA, Okhamafe AO. Processing pharmaceutical grade microcrystalline cellulose from groundnut husk: extraction methods and characterization. *International J Green Pharm*, 2009; 3[2]:97–104.
- 20.Suesat J, Suwanruji P. Preparation and properties of microcrystalline cellulose from corn residues. *Adv Mater Res*, 2011; 1781–1784.
- 21.Landín M, Martínez-Pacheco R, Gomez-AmozaSouto JLC, Concheiro A, Rowe RC. Effect of batch variation and source of pulp on the properties of pulp on the properties of microcrystalline cellulose. *Int J Pharm*, 1993; 91:133–141.
- 22.Chuayjuljit S, Su-uthai S, Charuchinda S. Poly[vinyl chloride] film filled with microcrystalline cellulose prepared from cotton fabric waste: Properties and biodegradability study. *Waste Management & Research*, 2010; 28: 109–117.
- 23.El-Sakhawy M, Hassan ML. Physical and mechanical properties of microcrystalline cellulose prepared from agricultural residues. *Carbohydrate Polymer*, 2007; 67: 1–10.
- 24.Ejikeme PM. Investigation of the physicochemical properties of microcrystalline cellulose from agricultural wastes: orange mesocarp. *Cellulose*, 2008; 15:141–147.
- 25.Spagnola C, Rodriguesa FHA, Pereira AGB, Fajardoa AR, Rubiraa AF, Muniz EC. Superabsorbent hydrogel composite made of cellulose nanofibrils and chitosan-graft-poly[acrylic acid]. *Carbohydrate Polymers*, 2012; 87: 2038–2045.
- 26.Johar N, Ahmad I, Dufresne A. Extraction, preparation and characterization of cellulose fibres and nanocrystals from rice husk. *Industrial Crops and Products*, 2012; 37: 93–99.
- 27.Jahan MS, Saeed A, He Z, Ni Y. Jute as raw material for the preparation of microcrystalline cellulose. *Cellulose*, 2011; 18: 451–459.
- 28.Fahma F, Iwamoto S, Hori N, Iwata T, Takemura A. Isolation, preparation, and characterization of nanofibers from oil palm empty-fruit-bunch [OPEFB]. *Cellulose*, 2010; 17: 977–985.
- 29.Wanrosli WD, Leh CP, Zainuddin Z, Tanaka R. Optimization of soda pulping variable for preparation of dissolving pulps from oil palm fiber. *Holzforschung*, 2003; 57: 106–113.
- 30.Wanrosli WD, Rohaizu R, Ghazali A. Synthesis and characterization of cellulose phosphate from oil palm empty fruit bunches microcrystalline cellulose. *Carbohydrate Polymers*, 2011; 84: 262–267.
- 31.Azizi Samir MAS, Alloin F, Dufresne A. Review of recent research into cellulosic whiskers, their properties and their application in nanocomposite field. *Bio macromolecules*, 2005; 6: 612–626.
- 32.Petersson L, Kvien I, Oksman K. Structure and thermal properties of poly[lactic acid]/cellulose whiskers nanocomposite materials. *Composites Science and Technology*, 2007; 67: 2535–2544.
- 33.Mathew AP, Oksman K., Sain M. Mechanical properties of biodegradable composites from poly lactic acid [PLA] and microcrystalline cellulose [MCC]. *J of Applied Poly Sci*, 2005; 97: 2014–2025.
- 34.Sturcova A, Davies GR, Eichhorn SJ. Elastic modulus and stress-transfer properties of tunicate cellulose whiskers. *Biomacromolecules*, 2005; 6: 1055–1061.
- 35.Yano H, Sugiyama J, Nakagaito A N, Nogi M, Matsuura T, Hikita M. Optically transparent composites reinforced with networks of bacterial nanofibers. *Advanced Materials*, 2005; 17: 153–155.
- 36.Ohwoavworhua FO, Kunle OO, Ofoefule SI. Extraction and characterization of microcrystalline cellulose derived from *Luffa cylindrica* plant. *African J Pharm Res Dev*, 2004;1:1-6.
- 37.Oyeniyi YJ, Itiola OA. The Physicochemical Characteristic Of Microcrystalline Cellulose, Derived From Sawdust, Agricultural Waste Products, *Int J Pharm Pharm Sci*, 2011; 4[1]:197-200.
- 38.Segal L, Creely JJ, Martin AE Jr, Conrad CM. An empirical method for estimating the degree of crystallinity of native cellulose using the x-ray diffractometer. *Text Res J*, 1959;29:786–794.
- 39.Huggins ML. Relationship between polymer concentration and intrinsic viscosity of polymeric materials. *J Am Chem Soc*, 1942; 64:2713.

40. Billmeyer FW. 1984. Cellulose ethers. In: Textbook of polymer science, 3rd ed. Wiley Interscience Publishers, New York: 4215-4226.
41. Burgess J, Duffy E, Etzler F, Hickey A. Particle Size Analysis: AAPS Workshop Report, Co sponsored by the Food and Drug Administration and the United States Pharmacopeia, AAPS Journal, 2004; 6 [3].
42. Azubuike CP, Rodríguez H, Okhamafe AO, Rogers RD. Physicochemical properties of maize cob cellulose powders reconstituted from ionic liquid solution. *Cellulose*, 2012; 19:425-433.
43. Carr RL. Evaluating flow properties of solids. *Chem Eng*, 1965; 72:163-168.
44. United States Pharmacopoeia. 2004. National Formulary. United States Pharmacopoeial Convention, Rockville, 22[27]:2845-2847.
45. Okhamafe AO, Azubuike CP. Direct compression studies on low cost celluloses derived from maize cob. *J Pharm Sci Pharmacy Pract*, 1994; 1:26-29.
46. RC Rowe, PJ Sheskey, SC Owen. 2006. Handbook of Pharmaceutical Excipients. Pharmaceutical Press and American Pharmacists Association.
47. Alemdar, A, Sain, M. Isolation and characterization of nanofibres from agricultural residues-wheat straw and soy hulls. *Bioresource Technology*. 2008; 99: 1664-1671.
48. Johar N, Ahmad I, Dufresne A. Extraction, preparation and characterization of cellulose fibres and nanocrystals from rice husk. *Industrial Crops and Products*. 2012; 37: 93-99.
49. De Menezes AJ, Siqueira G, Curvelo AAS, Dufresne A. Extrusion and characterization of functionalized cellulose whiskers reinforced polyethylene nanocomposites. *Polymer*, 2009; 50:4552-4563.
50. Lee SY, Mohan DJ, Kang In A, Doh, GH, Lee S, Han SO. Nanocellulose reinforced PVA composite films: Effects of acid treatment and filler loading. *Fibers and Polymers*, 2009; 10: 77-82.
51. Rosa SML, Rehman N, De Miranda MIG, Nachtigall SMB, Bica CID. Chlorine-free extraction of cellulose from rice husk and whisker isolation. *Carbohydrate Polymers*, 2012; 87:1131-1138.
52. Fahma F, Iwamoto S, Hori N, Iwata T, Takemura A. Isolation, preparation, and characterization of nanofibers from oil palm empty-fruit-bunch [OPEFB]. *Cellulose*, 2010; 17:977-985.
53. Jahan MS, Saeed A, He Z, Ni Y. Jute as raw material for the preparation of microcrystalline cellulose. *Cellulose*, 2011; 18: 451-459.
54. Satyamurthy P, Jain P, Balasubramanya RH, Vigneshwaran N. Preparation and characterization of cellulose nanowhiskers from cotton fibres by controlled microbial hydrolysis. *Carbohydr Polym*, 2011; 83:122-129.
55. Kumar V, Maria De La LRM, Yang D. Preparation, characterization, and tableting properties of a new cellulose-based pharmaceutical aid. *Int J of Pharmaceutics*, 2002; 235: 129-140.
56. Wang L, Han G, Zhang Y. Comparative study of composition, structure and properties of *Apocynum venetum* fibers under different pretreatments. *Carbohydr Polym*, 2007; 69:391-397.
57. Nuruddin M, Chowdhury A, Haque SA, Rahman M, Farhad SF, Sarwar Jahan M. Extraction and characterization of cellulose microfibrils from agricultural wastes in an integrated biorefinery initiative. *Cellulose Chemical and Technology*, 2011; 45:347-354.
58. Sain M, Panthapulakkal S. Bioprocess preparation of wheat straw fibres and their characterisation. *Ind Crops Products*. 2006; 23:1-8.
59. Jonoobi M, Khazaeian A, Md Tahir P, Azry SS, Oksman K. Characteristics of cellulose nanofibers isolated from rubberwood and empty fruit bunches of oil palm using chemo-mechanical process. *Cellulose*. 2011; 18:1085-1095.
60. Mandal A, Chakrabarty D. Isolation of nanocellulose from waste sugarcane bagasse [SCB] and its characterization. *Carbohydrate Polymers*. 2011; 86:1291-1299.
61. Peck GE, Baley GJ, Mcurdy VE, Banker GS. Tablet formulation and design. In: Lieberman HA, Lechman L, Schwarz JB, ed. *Pharmaceutical dosage forms*. New York: Marcel Dekker 75-130.
62. Carr RL. Evaluating flow properties of solids. *Chem Eng*, 1965; 72:163-168.
63. Wells JI. 1988. *Pharmaceutical preformulation: the physicochemical properties of drug substances*, 1st edn. New York, John Wiley and Sons.
64. Fowler HW. 2000. Powder flow and compaction. In: Carter SJ, ed. *Cooper and Gunn's tutorial pharmacy*, 6th ed. Delhi: CBS Publishers.
65. Pharmacopoeia B. 2004. The Stationery Office. The Department of Health, London, 405-407.
66. Doelker E, Gurny R, Schurz J, Janosi A, Matin N. Degrees of crystallinity and polymerization of modified cellulose powders for directing tableting. *Powder Technol*, 1987; 52:207-213.
67. Landín M, Martínez-Pacheco R, Gomez-Amoza Souto JLC, Concheiro A, Rowe RC. Effect of batch variation and source of pulp on the properties of pulp on the properties of microcrystalline cellulose. *Int J Pharm*, 1993; 91:133-141.
68. Caramella C. Novel methods for disintegrant characterisation, part 1. *Pharm Technol*, 1991; 48-56.