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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**

Department of Pharmaceutics, L.M. College of Pharmacy, Ahmedabad, Gujarat, India

**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.*

**Corresponding author:****Roshni Vora,**

Department of Pharmaceutics,

L. M. College of Pharmacy,

Navrangpura, Ahmedabad - 380 009,

Gujarat, India.

Phone: +91 9426451629

E-mail: [mehtaroshni1989@gmail.com](mailto:mehtaroshni1989@gmail.com)

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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 [ $\eta_{sp}$ ] of the polymer solution was calculated from the equation 3.

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

The intrinsic viscosity [ $\eta_0$ ] 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  $5 \times 10^{-4}$ .

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 Stoppered 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:

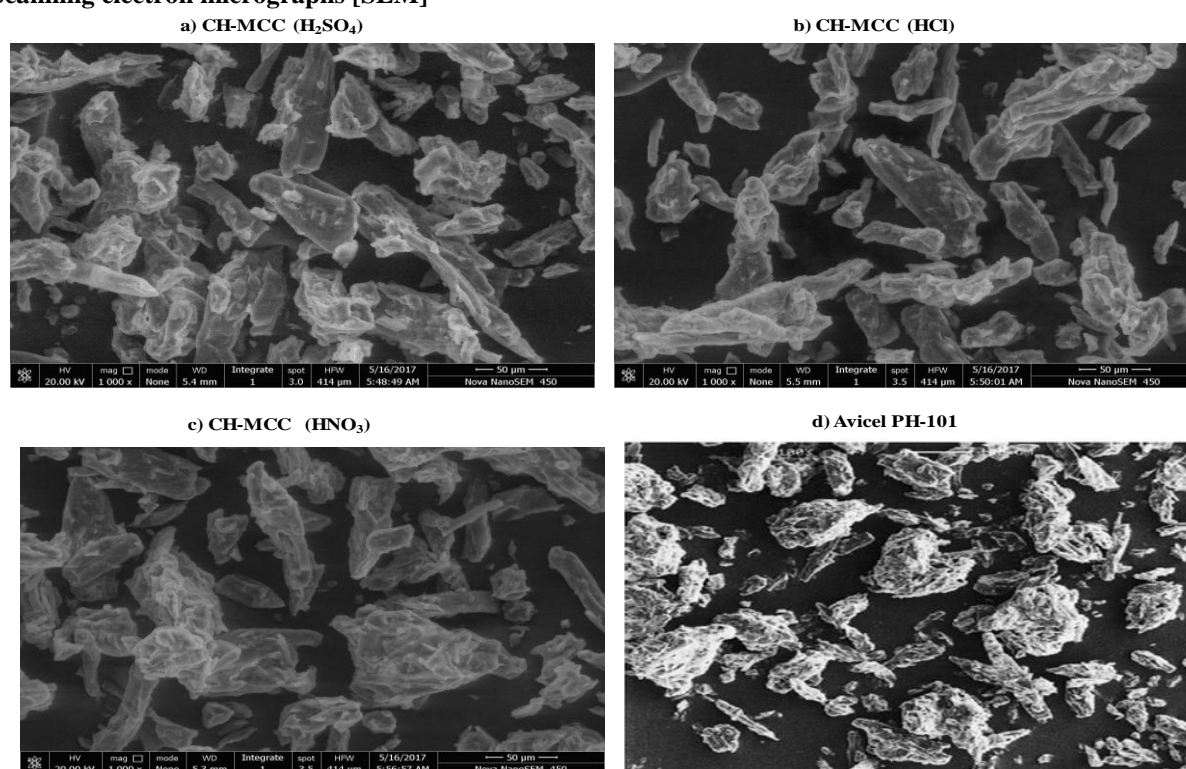
### Physicochemical Properties

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 <sub>2</sub> SO <sub>4</sub> ]	CH-MCC [HCl]	CH-MCC [HNO <sub>3</sub> ]	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 [H<sub>2</sub>SO<sub>4</sub>], CH-MCC [HCl], CH-MCC [HNO<sub>3</sub>]

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 aggragation [49, 50].



### FT-IR Spectroscopic Studies:

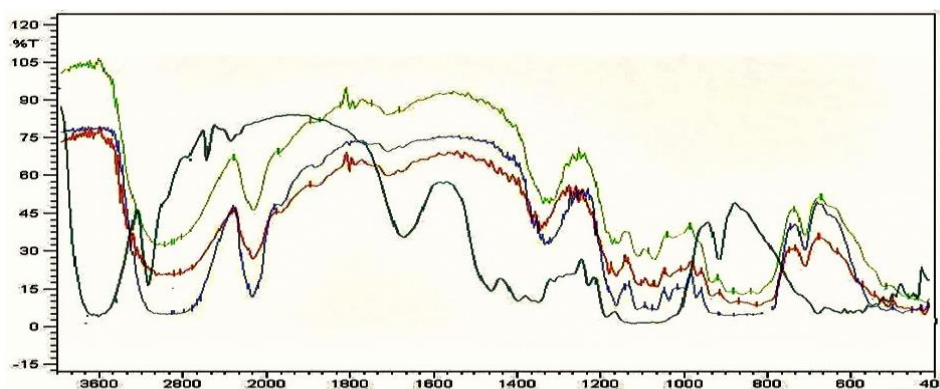


Fig 2. FTIR spectra. Black indicates Avicel PH101; green, CH-MCC [HNO<sub>3</sub>]; blue, CH-MCC [H<sub>2</sub>SO<sub>4</sub>] and red, CH-MCC [HCl].

FT-IR spectra of CH-MCC [H<sub>2</sub>SO<sub>4</sub>, HCl, HNO<sub>3</sub>] and Avicel PH101 are shown in Fig.2 and the spectral assignments are summarized in Table 2. CH-MCC [H<sub>2</sub>SO<sub>4</sub>, HCl, HNO<sub>3</sub>] and Avicel PH101 showed a high wave numbers [2800–3500 cm<sup>-1</sup>] and low wave numbers [500–1700 cm<sup>-1</sup>], respectively [51,52]. which is an indication that all samples have similar chemical compositions. The broad absorption band located from 3400 to 3500 cm<sup>-1</sup> is due to stretching of –OH groups and an absorption at 2900 cm<sup>-1</sup> is related to CH<sub>2</sub> groups [53,51, 54].

According to other studies and present investigation, the absorption at 1645 cm<sup>-1</sup> 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<sup>-1</sup> is associated to the intermolecular hydrogen at the C<sub>6</sub> [aromatic ring] group [55]. The absorption band at 1163 cm<sup>-1</sup> corresponds to C O C stretching, and the peak at 896 cm<sup>-1</sup> 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<sup>-1</sup>, 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

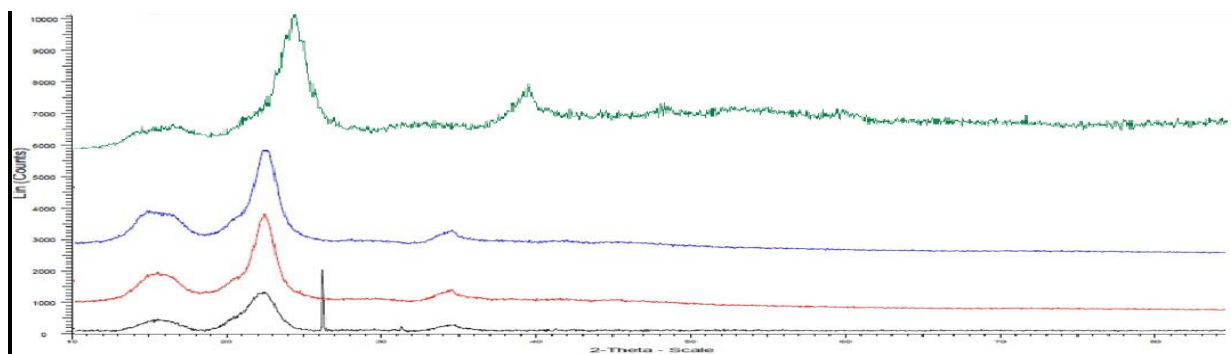


Fig 3: X-Ray Diffractograms Black indicates CH-MCC [H<sub>2</sub>SO<sub>4</sub>]; green, Avicel PH101; blue, CH-MCC [HNO<sub>3</sub>], and red, CH-MCC [HCl].

the region 1700–1740 cm<sup>-1</sup>, 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 <sup>-1</sup>
Ring vibration and C-OH bending	1,060 cm <sup>-1</sup>
Pyranose ring	1090 cm <sup>-1</sup>
C-O-C aryl-alkyl	1265 cm <sup>-1</sup>
O—H in plane bending vibration	1315 cm <sup>-1</sup>
CH <sub>2</sub> due to crystalline nature	1415 cm <sup>-1</sup>
Intermolecular hydrogen bonds at the C group	1429, 1426 cm <sup>-1</sup>
–O- tensile vibration neighboring hydrogen atoms	1,650 cm <sup>-1</sup>
CH <sub>2</sub>	2854 cm <sup>-1</sup>
C-H asymmetric and symmetric tensile vibration	2,900 cm <sup>-1</sup>
Intermolecular O—H stretching vibration band	3327 cm <sup>-1</sup>
Intramolecular O—H stretching vibration band	3330 cm <sup>-1</sup>
Broad peak of OH stretching, hydrogen bonds	3,300 to 3,500 cm <sup>-1</sup>

The X-ray diffraction [XRD] patterns of CH-MCC [ $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ ] and Avicel PH101 are presented in Fig 3. The crystallinity of each sample is listed in Table 5. The diffractograms of the CH-MCC [ $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ ] samples of 101 and 002 has diffraction peaks of the  $2\theta$  angles at  $10.24^\circ$ ,  $13^\circ$ ,  $13.68^\circ$  and  $21.16^\circ, 22.51^\circ$ ,  $21.96^\circ$  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 [ $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ , and  $\text{HNO}_3$ ] and Avicel PH101 have similar thermal properties.

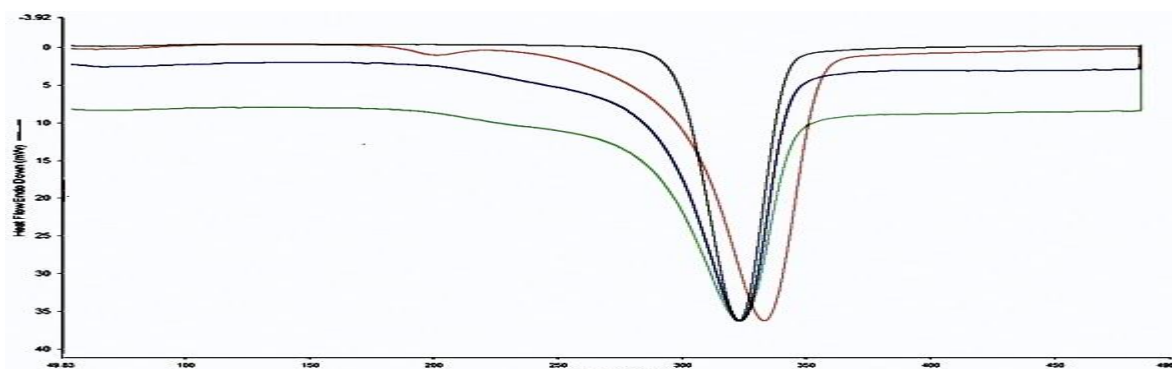


Fig 4: DSC thermograms. Black indicates Avicel PH101; green, CH-MCC [ $\text{HNO}_3$ ]; blue, CH-MCC [ $\text{H}_2\text{SO}_4$ ] and red, CH-MCC [ $\text{HCl}$ ].

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

Parameter [ $^\circ\text{C}$ ]	CH-MCC [ $\text{H}_2\text{SO}_4$ ]	CH-MCC [ $\text{HCl}$ ]	CH-MCC [ $\text{HNO}_3$ ]	Avicel PH101
Onset	250	252	250	300
Midpoint	325	330	325	325
Inflection Point	350	360	350	350

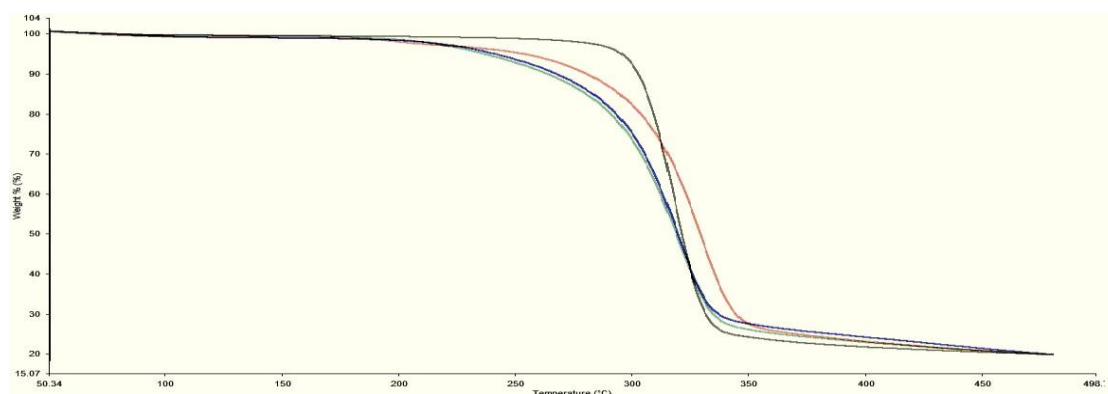


Fig 5: TGA thermograms. Black indicates Avicel PH101; green, CH-MCC [ $\text{HNO}_3$ ]; blue, CH-MCC [ $\text{H}_2\text{SO}_4$ ] and red, CH-MCC [ $\text{HCl}$ ].

Table 4: Thermal properties obtained from TGA experiments for MCC

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

The temperature, for which 5% of the mass of the sample is decomposed, T<sub>on 5%</sub>, and the temperature at 50% weight loss, T<sub>50%</sub>, is high and similar for all CH-MCC [H<sub>2</sub>SO<sub>4</sub>, HCl, HNO<sub>3</sub>] 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 [HNO<sub>3</sub>] as compared to CH-MCC [H<sub>2</sub>SO<sub>4</sub>] and CH-MCC [HCl]. Among the CH-MCC samples, CH-MCC [HNO<sub>3</sub>] 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 [H<sub>2</sub>SO<sub>4</sub>, HCl, HNO<sub>3</sub>] 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 <sub>2</sub> SO <sub>4</sub> ]	CH-MCC [HCl]	CH-MCC [HNO <sub>3</sub> ]	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 [ $\text{H}_2\text{SO}_4$ , HCl,  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ , HCl, and  $\text{HNO}_3$ ].

There is no significant differences among the bulk and tap densities of CH-MCC [ $\text{H}_2\text{SO}_4$ , HCl,  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ , HCl, and  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ , HCl,  $\text{HNO}_3$ ] 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  $35^\circ$  indicate good flow potential of the solid powders, whereas those samples with angles greater than  $35^\circ$  exhibit fair flow [64]. Here, CH-MCC [ $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ ] shows fair flow potential while that of CH-MCC [HCl] showed good result as Avicel PH101.

The moisture content measured for all CH-MCC [ $\text{H}_2\text{SO}_4$ , HCl, and  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ ,

HCl,  $\text{HNO}_3$ ] 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 [ $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ ]. 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.

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