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Physicochemical, spectroscopic and thermal properties of microcrystalline cellulose derived from corn cobs

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Abstract

Background: Low-cost and suitable microcrystalline cellulose powders for use in the pharmaceutical industry can be derived from agricultural residues. Most commercial microcrystalline cellulose powders are produced from dissolving pulp obtained from expensive hard woods using concentrated acids. α -Cellulose was extracted from an agricultural residue (corn cob) using a non-dissolving method. The spectroscopic, thermal and physicochemical properties of the derived α -cellulose and microcrystalline cellulose powders were compared with Avicel[®] PH 101 (Fluka, New South Wales, Australia), a commercial brand of microcrystalline cellulose (MCCA), using standard methods.

Results: X-ray diffraction showed that the microcrystalline cellulose samples obtained from maize cobs had diffraction pattern characteristics of both cellulose I and cellulose II, whereas MCCA had that of cellulose I; however, all the microcrystalline cellulose samples had similar crystallinity index (*C*) values. Infrared spectroscopy results showed that the microcrystalline cellulose samples had comparable CI values and molecular structure. Thermogravimetric analysis and differential scanning calorimetry data showed quite similar thermal behaviour for all cellulose samples. Comparison of physicochemical properties of the microcrystalline cellulose powders obtained from maize cob and MCCA mainly suggests that all the celluloses have similar flow and compression properties.

Conclusions: For almost all of the characterizations carried out, it was observed that the microcrystalline cellulose powders obtained from corn cob had similar characteristics to the MCCA, showing that it can be a good low-cost alternative to the expensive commercial brand.

Keywords: Agricultural residue, Corn cobs, Microcrystalline cellulose, Physicochemical properties, Thermal properties

Introduction

The annually renewable agricultural residues represent an abundant, inexpensive and readily available source of renewable lignocellulosic biomass. Each year, farming and agricultural processing generate millions of tonnes of residues, such as corn cobs and husks, groundnut shells, rice straw, banana stems, soy hulls and sugar beet pulp (Ruan et al. 1996). These materials can be obtained at a low cost from a variety of sources, but the content and quality of the three major structural polymeric

components (lignin, cellulose and hemicellulose) depend on the type of material (Taherzadeh and Niklasson 2004). Their utilizations are attracting increased interests around the world, particularly for the production of novel materials for environmentally friendly industrial applications after chemical modification (Pandey et al. 2000; Richardson and Gorton 2003).

Cellulose, one of the major component of lignocellulosic biomass, is a polydisperse polymer of high molecular weight and comprised long chains of D-glucose units joined together by β -1, 4-glucosidic bonds. (Krassig 1985). Significant advances in cellulosic modifications (mechanically, chemically and even enzymatically) and the resultant production of derivatives with unique chemical, physical and physiological properties, have dramatically increased interest in cellulose research over

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the past decade. This renewed focus on cellulose and its derivatives has resulted in the production of cellulose and its derivatives with varied physicochemical and functional properties (Azubuiké et al. 2012; Ohwoavworhwa and Adelakun 2005).

Among other uses, cellulose is widely employed as a raw material to prepare a number of excipients. Microcrystalline cellulose is described as 'purified, partially depolymerized cellulose' prepared by treating alpha cellulose obtained as a pulp from fibrous plant material with mineral acids (Brittain et al. 1993). Commercially available microcrystalline cellulose is derived from highly costly hard wood and also purified cotton. Several methods covered by patents have been described in the literature for the manufacture of cellulose powder. The need for cheaper sources of microcrystalline cellulose has led to the investigation of other lignocellulosic materials based on agricultural residues (Paralikar and Bhatawdekar 1988; Uesu et al. 2000; El-Sakhawy and Hassan 2007; Ejikeme 2008, Ohwoavworhwa et al. 2009; Suesat and Suwanruji 2011). The chemical composition and physical structure of microcrystalline cellulose depend significantly on the characteristics of the raw material employed and the manufacturing conditions (Landin et al. 1993). 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.

Apart from promoting the manufacturability of drug product, excipients are expected to guarantee the stability and bioavailability of the drug substance from the drug product. As a consequence, their characterisation must go beyond the simple tests for identity, purity and strength as prescribed in general by the Pharmacopoeia monographs. Full physical characterisation of solid materials is now made possible with the help of high-resolution analytical techniques (infrared spectroscopy, powder X-ray diffraction, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC)) on the molecular, particulate and bulk levels (Pifferi et al. 1999). This systematic approach is necessary to guarantee the behaviour of the excipient during the formulation and production phases. For example, infrared spectroscopy can determine the molecular structure and possible chemical interactions. TGA and DSC analyses are often adopted to clarify the stability, compatibility and transitions of phase of the excipients. The structure of the single crystal or the powder can be examined with absolute certainty by X-ray diffraction. It provides information on the degree of crystallinity of the powder and also polymorphic forms. The degree of crystallinity in the cellulose is important because it influences the various

properties including the compactibility and absorption of water, which in turn influence the flowability and the stability of the medicinal product (Pifferi et al. 1999).

As part of the on-going efforts to develop low-cost local raw materials from lignocellulosic materials (agricultural residues) with desired physicochemical properties for the industrial applications, we have in this study reported some physical properties of microcrystalline cellulose prepared from corn cob (an agricultural residue) and also evaluate their structural and thermal properties with view of ascertaining its potential as a pharmaceutical excipient. An attempt was also made to find out if the period of the hydrolysis has effect on these properties. These properties were also compared to a commercial brand Avicel[®] PH 101 (Fluka, New South Wales, Australia) that was obtained from highly costly hard wood.

Methods

Materials

Corn cob, an agricultural waste, was kindly supplied by the local farmers in Benin City, Edo State, Nigeria. Avicel[®] PH 101 (a commercial brand of *microcrystalline cellulose*), sodium hydroxide (reagent grade, 398%), hydrochloric acid (ACS reagent, 37%) and calcium hypochlorite (technical grade) were supplied by Sigma-Aldrich (Dorset, UK) and used as received. 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.

Production of microcrystalline cellulose

α -Cellulose (powdered cellulose) was obtained from the corn cob using inorganic substances following a procedure based on that reported elsewhere (Azubuiké et al. 2011). Briefly, the method involved the delignification of the cellulosic material with 3.5% nitric acid and 0.01% sodium sulphite at 90°C for 2 h followed by treatment with 2% sodium hydroxide extraction and 2% sodium sulphite at 50°C for 1 h and 17.5% sodium hydroxide at 80°C for 0.5 h. Bleaching was carried out with 3.2% sodium hypochlorite at 40°C for 1.5 h. The α -cellulose powder obtained from the corn cob was hydrolyzed at room temperature with 2 N hydrochloric acid under reflux for 15 min (Paralikar and Bhatawdekar 1988); the solid-liquor ratio was 1:10. The hydrolyzed cellulose was thoroughly washed with cold distilled water until neutral to litmus paper and then air dried. The microcrystalline cellulose obtained via this process was labelled *MCMSa*. This procedure was repeated for the α -cellulose for reflux times of 30 and 45 min, respectively; the microcrystalline cellulose powders obtained via this process were labelled *MCMSb* and *MCMSc*, respectively. The physicochemical, spectroscopic and thermal properties of the microcrystalline

cellulose powders obtained from the corn cobs were compared with that of Avicel® PH 101 (MCCA).

Characterisation

Physicochemical characterisation, spectroscopic (powder X-ray diffraction and infrared spectroscopy) and thermal (TGA and DSC) characterisations were carried out. OriginPro 8 SR2 v. 0891 (B891) software (OriginLab Corporation, Northampton, MA, USA) was used for statistical evaluation. One-way analysis of variance was used to differentiate between samples and decide where significant differences were established.

Physicochemical characterisation

Bulk and tapped density. For the determination of the bulk and tapped densities, the methods reported in an earlier study were adopted (Azubuike et al. 2012). 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_0) 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_1) was noted to be the tapped volume. The bulk density, D_{bulk} , and tapped density, D_{tap} , were determined using Equations 1 and 2:

$$D_{\text{bulk}} = w/v_0 \quad (1)$$

$$D_{\text{tap}} = w/v_1, \quad (2)$$

where w is the weight of the powder, and v_0 and v_1 are the volumes of the bulk and tapped powders, respectively. The mean of three replicate determinations was taken in each case.

True density. The true density of the cellulose samples was determined using a model MPY-2 helium displacement pycnometer (Quantachrome Corporation, Syosset, NY, USA). The true density, D_{true} , was calculated using Equation 3:

$$D_{\text{true}} = w/v_p, \quad (3)$$

where w and v_p are the weight of the sample and the true volume of the powder, respectively.

Carr's index and Hausner ratio. Carr's index (Carr 1965) and Hausner ratio (Hausner 1967) for cellulose were calculated from bulk and tapped densities using Equations 4 and 5, respectively:

$$\text{Carr's index} = \frac{D_{\text{tap}} - D_{\text{bulk}}}{D_{\text{tap}}} \times 100 \quad (4)$$

$$\text{Hausner ratio} = \frac{D_{\text{tap}}}{D_{\text{bulk}}} \quad (5)$$

Powder porosity. The porosity (P_b) of the tested powders was evaluated from the true and tapped densities, by means of Equation 6:

$$P_b = 1 - \frac{D_{\text{tap}}}{D_{\text{true}}} \quad (6)$$

Angle of repose. The measurement of the angle of repose was carried out using a long cylindrical tube open at both ends as detailed in an earlier study (Azubuike et al. 2012). The tube was perpendicularly placed on a clean cardboard paper and filled flat with the cellulose powder. The tube was then gradually lifted away, vertically, from the cardboard. The height, h , and radius, r , of the conical heap formed were measured and then the angle of repose, θ , was calculated from Equation 7. Determinations were done in triplicate, and the mean was taken.

$$\theta = \tan^{-1}(h/r) \quad (7)$$

Moisture content. The moisture content of the cellulose powders was calculated from the weight loss on heating from room temperature to 225°C on a Perkin Elmer series 7 thermal analyzer (Waltham, MA, USA). Triplicate determinations were carried out.

Powder X-ray diffraction

X-ray diffraction patterns of the cellulose samples were obtained using an X-ray diffractometer (Philips X-ray Analytical, Amsterdam, The Netherlands). Samples for analysis were prepared by pressing the powder into the cavity of a sample holder and smoothing with a glass slide. They were scanned from 5° to 40° 2θ with a Cu anode X-ray operated at 40 kV and 40 mA in combination with a Ni filter to give a monochromatic Cu-K α radiation ($\lambda = 1.5418 \text{ \AA}$). The crystallinity index (CI) was calculated using Equation 8, proposed by Segal et al. (1959):

$$\text{CI} = 100 \times \frac{I_{002} - I_{\text{am}}}{I_{002}} \quad (8)$$

where I_{002} is the maximum intensity of the principal peak (002) lattice diffraction (at 22.7° of 2θ for cellulose I, and 21.7° of 2θ for cellulose II), and I_{am} is the intensity of diffraction attributed to amorphous cellulose (at 18° of 2θ for cellulose I, and at 16° of 2θ for cellulose II). Integration of the crystalline reflection was carried out with OriginPro software.

Infrared spectroscopy

Fourier transform infrared (FT-IR) spectra were recorded using KBr discs on a Perkin-Elmer FT-IR spectrometer. KBr pellets containing 1% of the samples were

prepared. The scanned range was 4,000 to 400 cm^{-1} , with a resolution of 4 cm^{-1} and 32 scans per sample. For the determination of crystallinity in the cellulosic materials, the ratio A_{1372}/A_{2900} was considered, where A_{1372} and A_{2900} are the areas of the peaks at 1,372 and 2,900 cm^{-1} , respectively (Nelson and O'Connor 1964). Integration of the peak areas was carried out with OriginPro software.

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 600°C at a rate of 5°C/min under nitrogen.

Results and discussion

Three maize cob microcrystalline cellulose samples (MCMSa, MCMSb and MCMSc) were obtained, and their characteristics were compared with Avicel® PH 101 (commercial grade) (MCCA).

Physicochemical characteristics

The physicochemical properties of the microcrystalline cellulose samples are reported in Table 1. There is no significant differences among the bulk and tap densities of the four microcrystalline cellulose samples. 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 microcrystalline cellulose samples might have good flow properties (Peck et al. 1989).

Table 1 Physicochemical properties of the cellulose samples, with standard deviations (corresponding to the last significant figure) in parentheses

Property	Microcrystalline cellulose powder samples			
	MCCA	MCMSa	MCMSb	MCMSc
Bulk density (g cm^{-3})	0.35 (5)	0.33 (5)	0.32 (4)	0.30 (4)
Tap density (g cm^{-3})	0.42 (2)	0.42 (4)	0.41 (3)	0.39 (3)
True density (g cm^{-3})	1.60 (3)	1.55 (4)	1.61 (7)	1.62 (5)
Carr's index	16.7	21.9	21.4	23.1
Hausner ratio	1.2	1.3	1.3	1.3
Powder porosity	0.74	0.70	0.75	0.75
Angle of repose (°)	41.2 (7)	41.9 (9)	40.5 (12)	39.5 (15)
Moisture content (%)	5.30 (6)	5.90 (0)	5.40 (7)	5.30 (4)

Apart from the true density of MCMSa sample (1.55 g cm^{-3}), there was no significant difference in the true densities of the other microcrystalline cellulose samples. The true densities of the microcrystalline cellulose samples fairly correspond with those reported in previous studies (Ejikeme 2008). In general, the higher the true density of a powder, the better the compressibility.

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 (Carr 1965; Wells 1988). For Carr's index, values in the ranges 5 to 10, 12 to 16, 18 to 21 and 23 to 28 indicate excellent, good, fair and poor flow properties of the material, respectively (Carr 1965). In the study, the indices for all the microcrystalline cellulose samples are approximately in the range 15 to 20 (fair-good flowability). On the other hand, for the Hausner ratio, a value of less than 1.20 indicates good flowability, whereas a value of 1.50 or higher suggests that the material will have poor flow properties. In our case, the Hausner ratio for the microcrystalline cellulose samples lie around the threshold of 1.20 (\approx good flowability). Therefore, the values obtained for the Hausner ratio are consistent with those of the Carr's index. However, in both cases, the MCCA showed a little superior flow properties compared to microcrystalline cellulose samples obtained from maize cobs.

The angles of repose determined, which are also reported in Table 1, are fairly consistent with the above results. The angle of repose of a powder gives a qualitative assessment of its internal and cohesive frictions. Angles of up to 40° indicate reasonable flow potential of the solid powders, whereas those samples with angles greater than 50° exhibit poor or absent flow (Fowler 2000). In our case, no significant differences were observed among the angles of repose of the microcrystalline celluloses. The angle of repose of the microcrystalline cellulose samples lie around 40° (reasonable flow).

The moisture contents measured for all celluloses in this work (last row in Table 1) fall within the acceptable limits of between 5% and 7%, (United States Pharmacopoeia 2004; British Pharmacopoeia 2004). Furthermore, powder flowability is known to decrease with increasing moisture content (Peck et al. 1989). Overall, the good flow properties for the microcrystalline cellulose are likely to be the result of variations in the particle shape, size and surface area of the powders (Doelker et al. 1987; Landin et al. 1993) directly related to the process of partial hydrolysis of the original cellulose in an acid. The total porosity of a porous powder is made up of voids between the particles as well as pores within

the particles. The results (Table 1, line 6) obtained for all the celluloses are similar and indicating poly-sized particles and easily compressible powder during tableting.

Structural characterisation

The powder X-ray diffraction spectra of all four cellulose samples are shown in Figure 1. The diffractograms of the microcrystalline cellulose samples exhibit diffraction pattern typical of cellulose I, with diffraction peaks of the 2θ angles at 15.0° , 16.6° and 22.4° , which can be assigned to the 101, $10\bar{1}$ and 002 reflections, respectively (Isogai 1994). However, the microcrystalline cellulose samples (MCMSa, MCMSb and MCMSc) obtained from corn cobs also exhibited diffraction pattern typical of cellulose II with diffraction peaks of 2θ angles also at 12.5° and 20.4° . This indicates that the microcrystalline cellulose samples obtained from corn cobs are made up of cellulose I and cellulose II polymorphs. This might be due to short time exposure of the raw materials to high concentration of sodium hydroxide solution (17.5% NaOH) during the cellulose isolation. It has been reported that the lattice transition from cellulose I to cellulose II sets in above 10% of sodium hydroxide, but it is not completed below 15% of sodium hydroxide (Ryshkewitch 1953). In a study of X-ray diffraction of pine wood treated with NaOH, Borysiak and Doczekalska (2005) found that the amount of cellulose II depends on the concentration of NaOH solution as well as on the time of chemical activation. The greatest efficacy of polymorphic transition of cellulose was noted in the highest concentration of NaOH solution. The CI gives a quantitative measure of the crystallinity in powders and can relate to the strength and stiffness of fibres (Wang et al. 2007). Here, the microcrystalline cellulose sample CI values (78% for MCCA, 73% for MCMSa, 76% for MCMSb and 78% for MCMSc) are similar to

those reported in other studies for MCC (El-Sakhawy and Hassan 2007; Suesat and Suwanruji 2011) and are almost in the same range with each other apart from MCSMa sample that is a little bit lower. 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 CI values are therefore consistent with the high true density values of the microcrystalline cellulose samples described later under physicochemical properties with the MCSMa sample's true density value being the lowest.

FT-IR spectroscopy has the ability to predict structural differences not seen by other physicochemical analyses. In spite of minor differences among them, the FT-IR spectra of the MCC samples (Figure 2) show the general characteristic spectrum of cellulose. For example, absorption bands are clearly observed at ca. 900 cm^{-1} (corresponding to β -glycosidic linkages), $1,060\text{ cm}^{-1}$ (ring vibration and C-OH bending), $1,650\text{ cm}^{-1}$ (ν -O- tensile vibration neighbouring hydrogen atoms), $2,900\text{ cm}^{-1}$ (C-H asymmetric and symmetric tensile vibration) and the broad peak at $3,300$ to $3,500\text{ cm}^{-1}$ (OH stretching, hydrogen bonds). A more detailed description of the FT-IR spectrum of cellulose can be found in the literature (Toğrul and Arslan 2003).

Thermal properties

The thermograms in Figures 3 and 4 show that all microcrystalline cellulose samples follow similar degradation patterns. The numerical data from the TGA experiments (Table 2) also reveal that the microcrystalline cellulose samples derived from the corn cob have similar thermal properties with the MCCA sample. The temperature for which 5% of the mass of the sample is decomposed, $T_{\text{on } 5\%}$, and the temperature at 50% weight

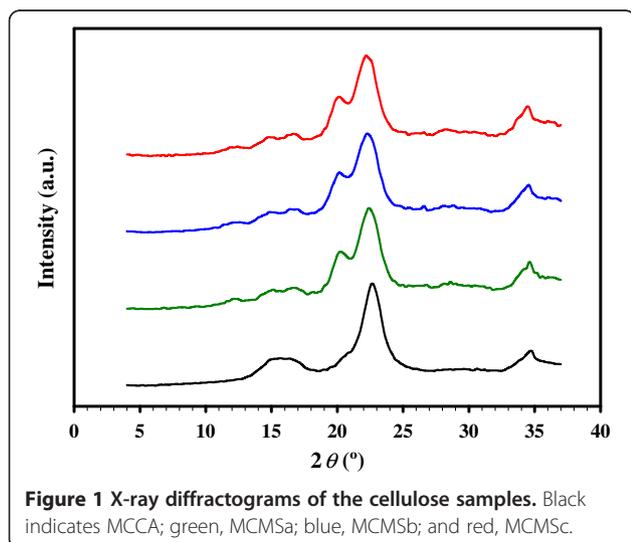


Figure 1 X-ray diffractograms of the cellulose samples. Black indicates MCCA; green, MCMSa; blue, MCMSb; and red, MCMSc.

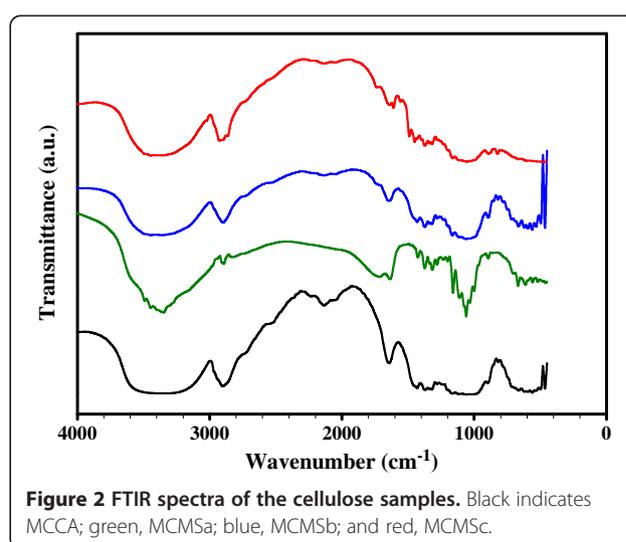


Figure 2 FTIR spectra of the cellulose samples. Black indicates MCCA; green, MCMSa; blue, MCMSb; and red, MCMSc.

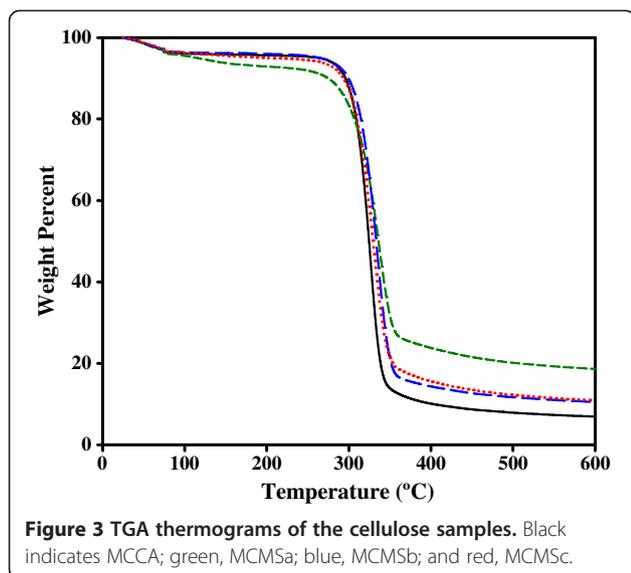


Figure 3 TGA thermograms of the cellulose samples. Black indicates MCCA; green, MCMSa; blue, MCMSb; and red, MCMSc.

loss, $T_{50\%}$, are high and similar for all the microcrystalline cellulose samples. A more notable difference, in particular between the microcrystalline cellulose samples derived from the corn cob and the standard MCCA, is observed at W_{550} (%), residual char weight at 550°C. The char yield (non-volatile carbonaceous material generated on pyrolysis, which is indicated by the residual weight after the decomposition step), specified in Table 2 for the temperature 550°C, is higher for the corn cob-derived microcrystalline cellulose samples compared to the MCCA sample. Among the corn cob-derived microcrystalline cellulose samples, MCMSa sample had higher residual char value at 550°C indicating higher non-volatile carbonaceous material generated on pyrolysis (Sain and Panthapulakkal 2006).

The results of DSC fairly correspond with the observations made from thermogravimetric analysis. The onset

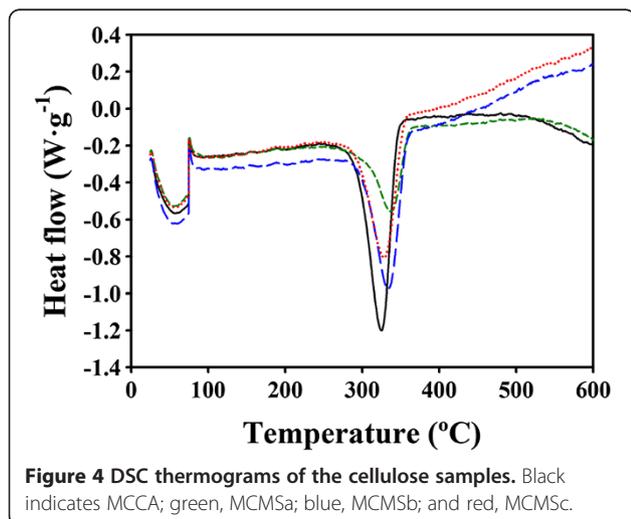


Figure 4 DSC thermograms of the cellulose samples. Black indicates MCCA; green, MCMSa; blue, MCMSb; and red, MCMSc.

Table 2 Thermal properties obtained from TGA experiments for microcrystalline celluloses studied

Parameter	Microcrystalline cellulose powder samples			
	MCCA	MCMSa	MCMSb	MCMSc
$T_{5\%, \text{onset}}$ (°C) ^a	280	270	279	284
$T_{50\%}$ (°C) ^b	325	334	333	330
W_{550} (%) ^c	7	19	11	11

^aOnset temperature for 5% decomposition; ^btemperature at 50% weight loss; ^cresidual char weight at 550°C.

temperatures of the decomposition as well as the mid-point and inflection point temperature data for all the microcrystalline cellulose samples are similar and are presented in Table 3. The higher onset temperatures are associated with higher thermal stability. This behaviour could be attributed to the high degree of crystallinity of the microcrystalline celluloses. A more notable difference among the microcrystalline cellulose samples is observed at inflection slope temperatures. The inflection slope which indicates the rate of degradation is higher for the MCCA sample (−26°C) and lowest for MCMSa sample (−13°C). The higher inflection slope of MCCA sample may be caused by the lower diffusion of the degraded products, which may further enhance degradation (Sain and Panthapulakkal 2006).

Conclusions

The microcrystalline cellulose powders obtained from the corn cob compared favourably with the Avicel® PH 101 (a commercial brand of microcrystalline cellulose), in terms of their physicochemical and thermal properties; however, unlike the commercial brand of microcrystalline cellulose that exhibited diffraction pattern characteristic of cellulose I, all the microcrystalline cellulose samples obtained from corn cob exhibited diffraction pattern characteristic of both cellulose I and cellulose II. Moreover, among the microcrystalline cellulose samples obtained from corn cob, thermal analysis studies showed that the MCMSa sample had the highest residual char weight at 550°C and the lowest value of inflection slope. Since farmers generate a lot of corn cob as a waste material, it would ultimately be a cheaper source than that of the costly Avicel® brand of microcrystalline cellulose.

Table 3 Derived transition temperatures from DSC thermograms of microcrystalline celluloses

Parameter	Microcrystalline cellulose powder samples			
	MCCA	MCMSa	MCMSb	MCMSc
Onset (°C)	308	309	313	308
Midpoint (°C)	323	329	331	327
Inflection point (°C)	326	335	335	329
Inflection slope (%°C ⁻¹)	−26	−13	−21	−19

Competing interests

The authors' declare that they have no competing interests.

Authors' contributions

CPA carried out the extraction and characterisation of the cellulose samples, participated in the design of the study, and performed the statistical analysis. AOO conceived the study, participated in its design and coordination, and helped draft the manuscript. All authors read and approved the final manuscript.

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