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Ezea Vincent Ndika, Umerie Sunday Chidozie and Ubaaji Kingsley Ikechukwu

Full Length Research Paper

Chemical modification of cellulose from palm kernel de-oiled cake to microcrystalline cellulose and its evaluation as a pharmaceutical excipient

Ezea Vincent Ndika*, Umerie Sunday Chidozie and Ubaaji Kingsley Ikechukwu.

Department of Applied Biochemistry, Faculty of Biological Sciences, Nnamdi Azikiwe University Awka, Anambra State, Country, Nigeria.

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Microcrystalline cellulose (MCC) is an important ingredient in pharmaceutical, food, cosmetic and other industries. In this research work, microcrystalline cellulose was synthesized from the *alpha* cellulose content of pretreated palm kernel de-oiled cake. The microcrystalline cellulose from palm kernel cake was obtained through acidified sodium chlorite, sodium hydroxide delignification followed by hydrogen peroxides bleaching and finally acid hydrolysis. The prepared microcrystalline cellulose was characterized by determining some physicochemical properties such as pH, bulk density, tap density, moisture content, ash content, Carrs compressibility index, Hausners ratio, powder porosity, angle of repose and compared with commercial-grade microcrystalline cellulose that is used in pharmaceutical industry as excipient. FT-IR was employed to expose the functional groups and the wavelength inherent by both the produced and commercial microcrystalline cellulose. The swelling property of MCC product was determined based on hydration capacity, swelling capacity and moisture sorption capacity. The results of the physicochemical parameters were given as pH(7.75 ± 0.40%), bulk density (0.49 ± 0.67 g cm⁻³), tapped density (0.54 ± 0.03 g cm⁻³), moisture content (1.00 ± 0.5%), ash content (4.30 ± 0.35 %), Carrs compressibility index (12.96 ± 0.27), Hausners ratio (1.15 ± 0.01), powder porosity (18.8 ± 0.55), angle of repose (27.4 ± 0.26) respectively. The swelling properties were also determined and compared favorably with the commercial grade of microcrystalline cellulose (p>0.05). The findings suggest that palm kernel cake can not only be used as feeds for animals but also a better source of cellulose for the production of microcrystalline cellulose for the industry owing to its availability and cost managements.

Key words: Microcrystalline cellulose, palm kernel de-oiled cake, commercial grade, carr's compressibility index, pharmaceutical excipient.

INTRODUCTION

Microcrystalline cellulose is native from cellulose (Leppänen et al., 2010) and it is a cellulose derivative obtained from natural fiber usable for pharmaceutical, cosmetic, food and plastic composite fields owing to

its compatibility, hydrophilicity, acid-insolubility and biodegradability (Thoorens et al., 2014). In the food industries, microcrystalline cellulose is used as a stabilizer, anti-caking agent, fat substitute and emulsifier

*Corresponding author. E-mail: ezeavincentndika@gmail.com.

(Leppanen et al., 2009; Yakubu et al., 2011). In pharmaceuticals industries, microcrystalline cellulose is used as excipient, and also as a stabilizer (Thoorens et al., 2014).

The most common source of microcrystalline cellulose is fibrous wood pulp (Shokri and Adibkia, 2013). The pulp is hydrolyzed under heat and pressure. In hydrolysis, cellulose polymer breaks in the presence of water and acid. The cellulose polymers in pulp are reduced to small chain polymers or microcrystals. Beta and gamma cellulose, hemicelluloses and lignin are dissolved with acid and water, and separate out during washing and filtration. Alpha cellulose mostly found in plant and natural fiber is the well-known raw material for microcrystalline cellulose preparation. Cellulose chain of any fibrous plant consists of a large number of glucose molecules linked together with β -1-4 glycosidic bond (Thoorens et al., 2014; Haque et al., 2015). Generally, three production steps which include delignification, bleaching and hydrolysis are involved in microcrystalline cellulose preparation from natural fiber. For the first and second steps, the native fiber needs to be treated with alkaline solvent to remove impurities from cellulose fiber before hydrolysis, since cellulose is covered with impurities such as wax, hemicelluloses, lignin and other compositions (Adel et al., 2010); next, the hydrolysis process is used to reduce the crystalline cellulose into small size. The dilute acid hydrolysis, hydrochloric and or sulfuric acid for instance, is widely used in industrial scale owing to its reasonable price. During the acid hydrolysis process, acid solution penetrates into voids of amorphous region prior to crystalline region and break down at glycosidic bond of the cellulose thereby leading to small size of crystalline structure in micro scale (Haque et al., 2015). The acid hydrolysis of cellulose produces mainly glucose from the amorphous part (Jahan et al., 2011).

The preparation of tablets using direct compression method has steadily increased due to the numerous advantages of direct compression technology in tableting, these include economy, elimination of granulation process, and uniformity of particle size and greater stability of tablets on aging (Shangraw, 1984). Currently, microcrystalline cellulose (MCC) is one of the mostly used direct compression excipient (Setu et al., 2014; Haque et al., 2015). MCC is not only highly compressible but increases the compressibility of other excipients when added in small quantities (Kumar et al., 2001). It is an effective dry binder in low concentration. It has sufficient fluidity which aids its disintegrating properties (Bolhuis et al., 1996). It is well accepted that powder properties of the microcrystalline cellulose are vital in tablet formulation (Sakurada et al., 1962) owing to the flow ability of the microcrystalline cellulose. Flow ability of powder play a very important role in drug manufacturing process. In tablet manufacturing process, flow ability involved in mixing and compaction, such as powder flow in hoppers is a crucial factor for direct compression excipients in drug manufacturing to achieve constant and weight

uniformity of tablets. Flow ability is affected by the physical properties of powder such as particle size, shape, bulk density (Soppela et al., 2010).

Commercially available MCC is derived from both gymnosperms (generally conifers) and other softwoods, and from hardwood dicotyledons and cotton. These differ considerably in chemical composition (proportions of cellulose, hemicelluloses, and lignin) and structural organization that the composition of the α -cellulose extracted and the crystallinity of MCC finally produced (Landin et al., 1993). Traditionally, microcrystalline cellulose is been prepared from wood source. In wood pulp cellulose chains are packed in layers held together by a cross-linking polymer (lignin) and strong hydrogen bonds. Wood pulps contain cellulose, hemicellulose and lignin, the cellulose contains alpha cellulose ranging between 92 to 98%, beta and gamma present 1% (Jafar et al., 2011). Besides the wood pulp as a source of cellulose and its derivatives, the purified cotton linters obtained from *Bombax ceiba* L. are also a common source (Evans, 1989).

The use of alternative non-wood sources of fiber in preparation of pulp for industrial applications has received substantial attention. Alternative sources of MCC recently investigated include agricultural wastes and other plants parts not traditionally used for MCC production (Ohwoavworhua et al., 2004; Ohwoavworhua et al., 2005). To the best of the researcher's knowledge, no data has been published in the preparation of microcrystalline cellulose from palm kernel cake or from any other sources available in Nigeria. Palm kernel cake (PKC), is the main by-product of the palm kernel oil extraction process. Palm kernel cake from mechanical extraction contains 5-12% oil and solvent-extracted palm kernel cake contains 0.5-3% oil otherwise known as palm kernel de-oiled cake (Chin, 2001). Palm kernel de-oiled cake is a highly fibrous and medium grade protein feed, hence most suited to ruminant or rabbit feeding (Pickard, 2005), it has a limited applications, thereby, there is a need to optimize the value chain of the palm kernel by-products as they should be channeled into products of greater market values. The only potential method is to convert the agricultural by-product (palm kernel cake) into a useful product. Thus leading to the significance of this study, which was to prepare MCC from palm kernel cake and its evaluation of some physicochemical parameters to check the possibility of the prepared microcrystalline cellulose to be used as an excipient in the pharmaceutical industry. Palm kernel cake is chosen as the microcrystalline cellulose source due to its availability, low cost and eco-friendly.

MATERIALS AND METHODS

Sample collection and preparation

Palm kernel cake was obtained from a local palm kernel oil mill

factory at Ogbete Main Market Enugu, Enugu state of Nigeria. It was first dried in sun light to remove the water content and to ease further grinding, ground in mortar and then sieved using 355 μm laboratory sieve in order to get a fine and homogeneous particles.

Chemicals

Analytical grade chemicals used in this experiment included hydrochloric acid (HCl, Merck, Germany), distilled water, H_2O , sodium hydroxide (NaOH, Merck, Germany).

Instruments

Electrical balance (SPB 31, Kaifeng Group Co. Ltd.), oswald's viscometer (Ernst Haage), hot plate stirrer (Thermolyne Mirak), electric wall oven (temperature EOEM61AS, Electrolux). Gallenkamp bench centrifuge (Gallenkamp, England), sieve shaker machine (Endicott's Ltd UK), Stampfvolometer Model STAV 2003 JEF (Germany), vortex mixer (Vortex-Genie Scientific Industry, USA), pH meter Corning, Model 10 (England), Buck Scientific Infra-red Spectrophotometer (Model: M530).

Methods

Isolation of cellulose from palm kernel cake

Step 1. Sample de-fatting: The sample was defatted followed by the procedure described by Bono et al. (2009), with modification (n-hexane was used here). The palm kernel cake sample weighing 152.67 g was weighed into a 1000 ml flat bottom flask. Then 200 ml of n-hexane was introduced into the sample and the sample shaken vigorously until the whole sample was completely submerged in the n-hexane. The mixture was covered using foil and allowed to stand for 24 h for complete fat/oil extraction. The n-hexane containing the dissolved oil was gradually decanted from the sample and the sample was allowed to dry overnight at ambient temperature. The % oil or fat content was calculated as:

$$= \frac{W_1 - W_2}{W_1} \times \frac{100}{1}$$

Where W_1 is the weight of the sample before de-fating, W_2 is the final weight of the sample after de-fating.

Step 2: Removal of lignin: For acidified sodium chlorite process, 129.27 g of the oil free palm kernel was mixed with 100 ml distilled water, 100 ml glacial acetic acid and 50 g of sodium chlorite. The mixture was stirred for 2 hours at 75°C. The residue was filtered and washed with 200 ml of 95% ethanol and 100 ml of distilled water. This step was repeated. Similarly, the washed palm kernel was dried in oven at 60°C for 24 h. This was done according to Bali et al., (2015) with slight modification. Calculation of lignin % was done as follows.

$$= \frac{W_1 - W_2}{W_1} \times \frac{100}{1}$$

Where W_1 is the initial weight of the sample, W_2 is the final weight of the sample after drying.

Step 3: NaOH Pretreatment (determination of hemicelluloses):

NaOH pretreatment was carried out following the method by Bali et al. (2015). The lignin free palm kernel cake weighing 121.83 g was weighed into a flat bottom round flask, to which 500 ml of 18% NaOH solution was added and stirred vigorously at room temperature for 30 min. During stirring, the flask was covered with aluminum foil, and afterwards filtered using a vacuum pump and then washed with 500 ml of 20% acetic acid in hot water, and washed again with hot distilled water. The hemicelluloses was determined using the equation:

$$= \frac{W_1 - W_2}{W_1} \times \frac{100}{1}$$

Where W_1 is the initial weight of the sample, W_2 is the final weight of the sample after drying.

Step 4: Bleaching with hydrogen peroxide: The bleaching was done by the procedure of Voravadee et al. (2016). The palm kernel cake 50 g was weighed into a three neck round bottom flask and 250 ml of 7.5% w/v of hydrogen peroxide was introduced into the weighed sample. A reflux condenser was fitted into the middle neck of the flask, the whole mixture refluxed at 90°C for 2 h. After 2 h, the sample was allowed to cool and filtered using a vacuum pump. The residue was further washed with hot water and then allowed to dry at room temperature. After the defatting, removal of lignin and hemicelluloses, the remaining sample is cellulose.

Production of MCC

Microcrystalline cellulose was prepared from alpha cellulose of pre-treated pulp by acid hydrolysis (Dong et al., 1998). A 30 g of the alpha-cellulose was added into a 500 ml flat bottom round flask. It was hydrolyzed with 250 ml of 5N HCl for 30 mins at room temperature. The mixture was heated at 100°C for 60 min with continuous stirring using magnetic heating plate with stirring nob. The mixture was allowed to cool and filtered with a Buchner apparatus and a vacuum pump. It was washed with distilled water so as to neutralize the MCC, and finally weighed to determine the weight of sample.

Confirmation of the produced Microcrystalline cellulose

The confirmation of the microcrystalline cellulose was done according to the method used by Monika et al. (2015).

Procedure: A 10 mg of the produced microcrystalline cellulose was weighed into a conical flask, 4 drops of iodinated zinc chloride solution was added into the microcrystalline cellulose, 20 g of zinc chloride and 6.5 g of potassium iodide was dissolved in 10.5 ml of water, followed by the addition of 0.5 g of iodine and was shaken for 20 min before it was added into the microcrystalline solution. Violet-blue color was changed indicating the presence of microcrystalline cellulose.

PREPARATION OF SAMPLE FOR FT-IR ANALYSIS

A 0.5 g of the sample was mixed with 0.5g of Kbr powder after which 1ml of nujol (a solvent for preparation of sample by Buck 530 IR-spectrophotometer) was pipette into the sample with aid of a syringe to form a paste before introducing it into the instrument sample mould and allowed to scan at a wavelength of 600-4000 cm^{-1} to obtain its spectra heights.

Determination of yield value

The yield was determined according to the method by Nuruddin et al. (2012):

$$\text{Yield (\%)} = \frac{A \times 100}{B \quad 1}$$

Whereas; A= weight of obtained microcrystalline cellulose (kg), B= weight of fibrous wood pulp (kg).

Moisture content determination

The moisture content was determined by the WHO (1979).

Principle: This method is used to determine the percentage of water in a sample by drying the sample to a constant weight.

Procedure: Empty shallow bottle was heated in a hot air oven at 105°C for 30 min after that it was cooled in desiccators at room temperature. A 1 g of the MCC was weighed into the shallow bottle and put inside the oven; oven was set at 105°C and kept for 3 h. After 3 h the shallow bottle was removed from the oven and was allowed to cool in desiccators at room temperature. When the shallow bottle was cooled, the weight was noted again and moisture content was calculated by using the following formula. This procedure was repeated three times and the average value was taken.

$$(\text{M.C}) = \frac{\text{After drying weight of shallow bottle} - \text{Empty weight of shallow bottle}}{\text{Sample weight in gram}} \times 100$$

Ash contents determination

The method as described by AOAC (1990) was used to determine the Ash content.

Procedure: Empty platinum crucibles were washed and dried in an oven at 80°C. It was cooled in the desiccators. Later, it was weighed and recorded. A 1 g of the sample was measured into the platinum crucible and put in a furnace and set at 600°C, it was ashed for 4 h. Percentage ash contents were calculated as:

$$= \frac{W_2 - W_3}{W_1} \times \frac{100}{1}$$

Where W_1 is the weight of the sample
 W_2 is the weight of the sample before ashing,
 W_3 is the final weight of the sample after ashing.

pH measurement

It is important to determine the pH value of this sodium salt to know the level of alkalinity or acidity.

Procedure: A 0.5 g of the MCC was dissolved in 25 ml of distilled water and the pH was measured using a Mettler Toledo pH meter.

Bulk and tapped density

For the determination of the bulk and tapped densities, 2.5 g of the cellulose powder was accurately weighed and poured into a 100 ml graduated cylinder. The cylinder was stoppered and the bulk volume (BV) was recorded. For tapped density, the cylinder was tapped from a height of 2.5 cm on a hard surface to a constant volume. The final volume (TV) was noted to be the tapped volume. The bulk and tapped densities were determined using the method by Hausner (1967).

$$\text{Bulk density} = \frac{W}{BV}$$

$$\text{Tap density} = \frac{W}{TV}$$

Whereas; BV = bulk volume, W = weight of the sample, TV = tap volume.

Powdered porosity

The porosity (Pp) of MCC powders was evaluated following the method by Peck et al. (1989).

$$Pp = \frac{BV - PV}{BV} \times 100$$

Where BV is bulk volume,
 PV is porous volume

Angle of repose

The angle of repose, θ , was calculated using Hausner (1967) method.

Procedure: The measurement of the angle of repose was carried out using a long cylindrical tube open at both ends. 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.

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

Carr's index and Hausners ratio

Carr's index and Hausners ratio of the cellulose were calculated from bulk and tapped densities using the methods described by Carr (1965) and Hausners (1967).

$$\text{Carr's index} = \frac{T.D - B.D}{T.D} \times 100$$

$$\text{Hausners ratio} = \frac{T.D}{B.D}$$

Whereas; T.D- Tapped density of powder,
 B.D – Bulk density of powder.

Table 1. Chemical composition of the palm kernel de-oiled cake.

Tests	Values
Lipid/fat content (%)	15.09 ± 0.24
Lignin (%)	4.79 ± 0.08
Hemicelluloses (%)	14.60 ± 0.23
Cellulose (%)	63.83 ± 0.97

Values are given as mean ± S.D of the three determinations.

The determination of the viscosity was done from the method used by Hong et al. (1978). Viscosity is a measure of the resistance of a fluid to deformation under shear stress. It describes a fluid's internal resistance to flow and is a measure of fluid friction.

The method of Kornblum and Stoopak (Kornblum et al., 1973) was adopted with slight modification in the determination of hydration capacity

Procedure: A 2 g each of the samples (produced and commercial MCC) was placed in each of four 15 mL plastic centrifuge tubes and 20 ml of distilled water added and stoppered. The contents were mixed on a Vortex-Gennie vortex mixer for 2 min. The mixture was allowed to stand for 10 min and immediately centrifuged at 1000 rpm for 10 min in a Gallenkamp bench centrifuge. The supernatant was carefully decanted and the sediment was weighed. The hydration capacity was taken as the ratio of the weight of the sediment to the dry sample weight. Swelling capacity was measured at the same time as the hydration capacity determination using the method reported by Ohwoavworhwa et al., (2004) and calculated as follows (Kornbrum et al., 1973):

$$S = \frac{V_2 - V_1}{V_1 \times 100}$$

Where S is the % swelling capacity,
 V_2 is the volume of the hydrated or swollen material,
 V_1 is the tapped volume of the material prior to hydration.

Moisture sorption capacity was determined according to the method described by Ohwoavworhwa et al. (2005)

Procedure: A 2 g of the cellulose material accurately weighed and evenly distributed over the surface of a 70 mm tarred Petri dish. The samples were then placed in a large desiccator containing distilled water in its reservoir (RH = 100%) at room temperature and the weight gained by the exposed samples at the end of a five-day period was noted. The amount of water sorbed was calculated from the weight difference.

STATISTICAL ANALYSIS

Results of the study were expressed as mean ± standard deviation. Differences between means of both the produced MCC and the control (commercial MCC) in this study was analyzed using one way Analysis of Variance (ANOVA) of SPSS 16.0 spread sheet

statistical package. Values were taken to be significant at $p < 0.05$.

RESULTS AND DISCUSSION

Chemical composition of the palm kernel cake

The chemical composition of the palm kernel de-oiled cake showed that the lipid, lignin, hemicelluloses and cellulose contents were 15.09, 4.79, 14.60 and 63.83% respectively. The yield of cellulose extracted from the palm kernel de-oiled cake, measured based on the dry weight basis was obtained after oil extraction, removal of lignin and hemicelluloses. From the value of the cellulose yield, it shows that palm kernel de-oiled cake has a high range of α -cellulose which will improve the quality of the cellulose derivatives (Table 1).

Characteristics of the produced MCC as pharmaceutical excipient

Results obtained from the characteristics of the produced MCC as pharmaceutical excipient are presented in Table 1. The values obtained for the ash, bulk density, tapped density, pH, carr's index, hausner ratio, powdered porosity, and angle of repose were given as $1.84 \pm 0.35\%$, $0.49 \pm 0.07 \text{ g cm}^{-3}$, $0.54 \pm 0.03 \text{ g cm}^{-3}$, $7.75 \pm 0.40\%$, 12.82 ± 0.27 , 1.15 ± 0.01 , 18.53 ± 0.55 and 27.6 ± 0.26 respectively for the produced MCC, which shows no significant difference from the values obtained from the control which were also given as $0.02 \pm 0.60\%$, $0.36 \pm 0.02 \text{ g cm}^{-3}$, $0.42 \pm 0.30 \text{ g cm}^{-3}$, $7.5 \pm 0.70\%$, 13.44 ± 0.02 , 1.16 ± 0.20 , 15.50 ± 1.22 , 30.55 ± 0.34 respectively (Table 2).

Swelling property of the produced and the commercial MCC

The swelling property of MCC product serves as a direct proof of tablets disintegration ability which can be assessed by the determination of hydration capacity, swelling capacity and moisture sorption and as well as moisture content (Table 3). The overlapped Figure 1 show the results of the FT-IR characterization on the both produced and commercial microcrystalline.

DISCUSSION

From the value of the cellulose yield, it showed that palm kernel de-oiled cake has a high range of cellulose content which improved the quality of the cellulose derivative (MCC). The cellulose yield was lower compared with the cellulose gotten from *Sorghum caudatum* carried out by Ohwoavworhwa and Adelakun (2010), but higher when compared with the cellulose produced from *Picea abies*

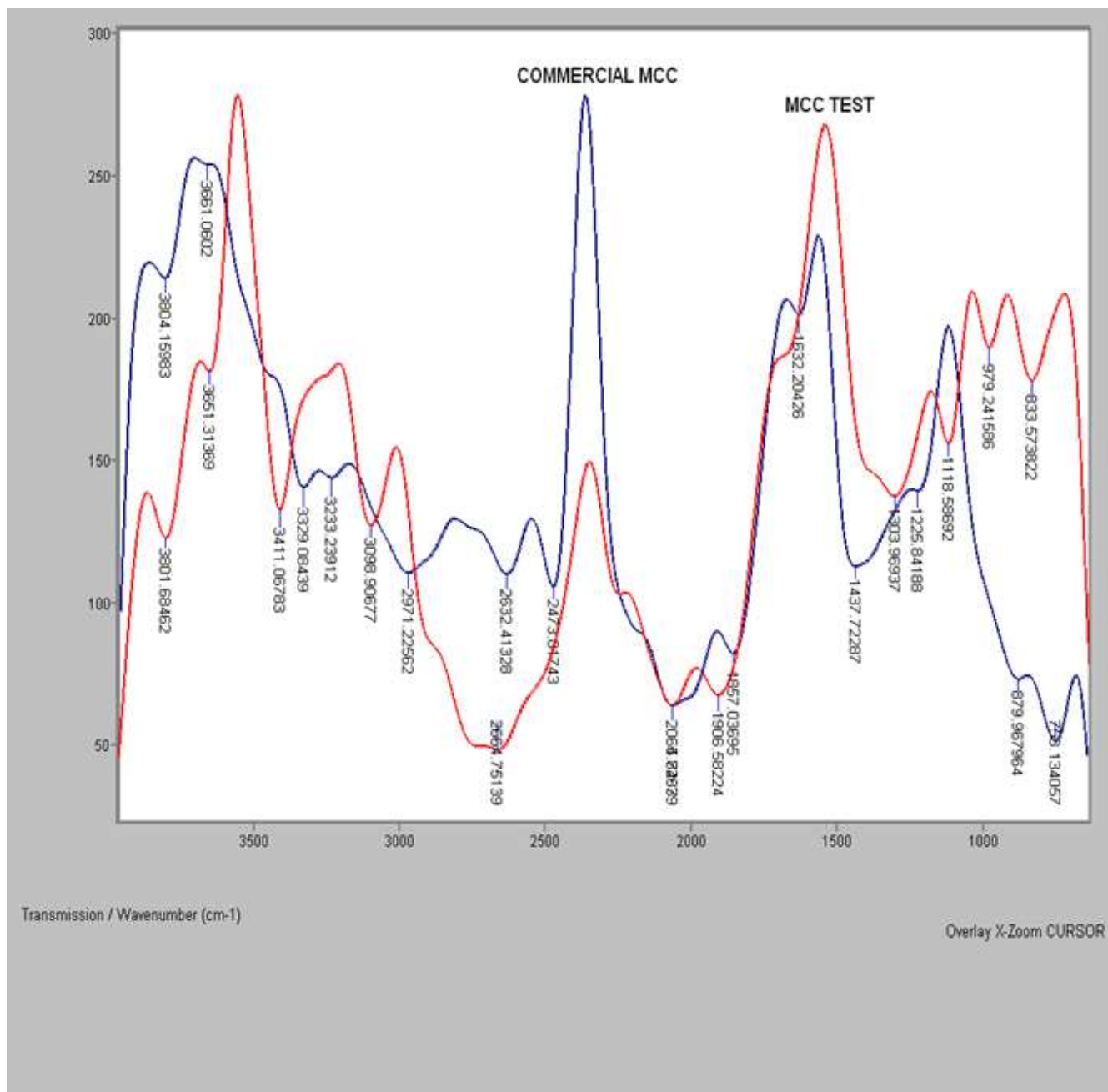


Figure 1. Results of the FT-IR characterization on produced and commercial microcrystalline.

(spruce) by Ambjornson et al. (2013), based on the original material used in the production process. The percentage yield of the cellulose from PKC was higher (98%) than the 90% of cellulose required for good quality cellulose derivatives. The α -cellulose according to Latif et al. (2007) should be greater than 90% for the production of high quality cellulose derivatives. Lower α -cellulose content implies the presence of low molecular oligosaccharides, which may affect the yield and quality of the derivative (Mark et al., 1985). The yield depends

upon the source of the cellulose (Monika et al., 2015).

Microcrystalline cellulose was confirmed when Violet-blue color was changed while testing for the presence of microcrystalline cellulose. Also the FT-IR spectrum of both the produced MCC and commercial MCC showed resemblance in their wavelengths as shown in Figure 1. This suggests that the product is microcrystalline cellulose. According to the result obtained, there was a higher content of hemicelluloses in palm kernel cake than what was observed from Cotton linter pulp which was

Table 2. Physicochemical parameter results on both the produced and commercial microcrystalline cellulose.

Parameter	Values for MCC-A	Values for MCC-B
Yield (%)	98	N/D
Bulk density (g cm ⁻³)	0.49 ± 0.07	0.36 ± 0.02
Tap density (g cm ⁻³)	0.54 ± 0.03	0.42 ± 0.30
Ash content (%)	1.84 ± 0.35	0.02 ± 0.60
p ^H	7.75 ± 0.40	7.5 ± 0.70
Carr's index	12.82 ± 0.27	13.44 ± 0.02
Hausners ratio	1.15 ± 0.01	1.16 ± 0.20
Powder porosity	18.53 ± 0.55	15.50 ± 1.22
Angle of repose	27.6 ± 0.26	30.55 ± 0.34

Values are given as mean ± S.D of the three determinations. Values were taken to be significant at p<0.05.

MCC-A= the produced MCC; MCC-B= the commercial MCC. N/D = Not determined (because it was bought in the market and not practically produced by the author).

carried out by Latif et al. (2007), this suggested that acetic acid and NaOH used for the elimination of the hemicelluloses must be enough during the production for proper removal of the hemicelluloses if not, it will impose a serious problem on any cellulose derivative.

As an oil seed, palm kernel cake contained high oil content, which was why it was further subjected to solvent extraction after the mechanical extraction process otherwise known as palm kernel de-oiled cake. The angle of repose gives a qualitative assessment of internal and cohesive frictional forces. An angle < 30° indicates good flow potential, while an angle of > 40° exhibits poor or absence of flow (Banker and Anderson, 1987). Both the prepared and the commercial microcrystalline cellulose samples are therefore classified as materials with reasonable flow potential because both have value less than 30°.

The bulk and the tapped density are higher than that of the commercial MCC (Table 2). The higher the bulk and tapped densities, the better the potential for a material to flow and to re-arrange under compression Roshni and Yamini (2015). From the work done by Roshni and Yamini (2015), the bulk, tap density, Carrs and Hausner index value compared favorably with the produced microcrystalline cellulose from the palm kernel cake for this research study. This suggested that the produced microcrystalline cellulose sample has good flow properties. However, according to Staniforth (1996), Hausner ratio greater than 1.25 indicates poor flow and Carr's compressibility index below 16% indicates good flow ability while values above 35% indicate cohesiveness. From this research study, the Hausner ratio was 1.15 which was lower than 1.25, which indicated good flow property, and the result of the Carr's compressibility was 12.82, which as well indicated good flow also. The angle of repose, Hausner ratio and Carr's compressibility are considered as indirect measurements of powder flow ability (Staniforth, 1996). 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) directly related to the process of partial hydrolysis of the original cellulose in an acid. The performance of a drug is primarily influence by the disintegration and dissolution behavior of the powder compact. Powder porosity predicts how liquid enters the drug matrix for disintegration and dissolution. The result for powder porosity for this study gave a higher value when compared with the commercial grade MCC this suggests that the produced MCC have a better disintegration and dissolution property.

According to United States Pharmacopoeia (2004), and the British pharmacopoeia (2004), pH of microcrystalline cellulose falls within the range 5-7. The pH of the prepared microcrystalline cellulose samples is 7.7 as shown in Table 2, which is within the acceptable range of standard microcrystalline cellulose. From the table it is observed that the standard commercial grade microcrystalline cellulose sample that is used in pharmaceutical industry as excipient, shows similar pH to the prepared microcrystalline cellulose samples.

The swelling property of MCC product serves as a direct proof of tablets disintegration ability (Caramella, 1991) which can be assessed by the determination of hydration capacity, swelling capacity and moisture sorption and as well as moisture content. The hydration capacity value obtained for MCC, (Table 3), indicates that it is capable of absorbing about two times its own weight of water. The swelling capacity of the MCC, which reflects the increase in volume of cellulose following water sorption, was 60% (Table 3). This is an indication that only a small portion of absorbed water actually penetrated the individual cellulose particles causing them to swell. So the values obtained on the swelling property of the cellulose indicate that if the cellulose is incorporated in tablet formulation as a disintegrant, it would probably

Table 3. Swelling properties.

Parameter	Values for MCC-A	Values for MCC-B
Moisture content (%)	1.00 ± 0.61	4.30 ± 0.50
Hydration capacity	4.00 ± 0.01	3.03 ± 0.04
Swelling capacity (%)	65.00 ± 1.12	70.15 ± 0.13
Moisture sorption capacity (%)	19.00 ± 1.03	17.60 ± 0.30

Values are given as mean ± S.D of the three determinations. Values were taken to be significant at $p < 0.05$.
MCC-A= The produced MCC; MCC-B = The commercial MCC.

produce tablet disintegration by two mechanisms: capillary or wicking due to interparticulate water, and swelling. In addition, the higher hydration and swelling capacities values observed for MCC irrespective of comparable ($p < 0.05$) powder porosity values of MCC and the commercial MCC (Table 3) could possibly be due to the difference in the proportion of amorphous cellulose present in the cellulose powders. Stamm (1964) reported that the amorphous portion is responsible for uptake and swelling of cellulose materials. The swelling property of the cellulose compared favourably well with the commercial MCC (Table 3) and as well as the work done by Ohwoavworhua (2010). It has been reported that the crystallite portion of cellulose does not adsorb water and that the extent of water adsorption by cellulose should thus be proportional to the amount of amorphous cellulose present (Stamm, 1964). Thus, the result is indicative of the higher amount of amorphous cellulose likely present in MCC unit fibrils. Also, study of water sorption is of importance since it reflects the relative physical stability of tablets when stored under humid conditions. Basically, this property showed that the cellulose powders are sensitive to atmospheric moisture and should therefore be stored in air tight container.

Conclusion

Microcrystalline cellulose was successfully produced from an underutilized agricultural by product (palm kernel cake). This work suggests that Palm kernel cake should not only be used as feeds for animals but also valuable raw material for our industries. It is recommended that purity test be further conducted on the MCC product before its incorporation in edible products.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

REFERENCES

Ambjornson HA, Schenzel K, Germgard U (2013). Carboxylmethyl cellulose produced at different mercerization conditions and characterized by NIR FT Raman Spectroscopy in combination with

- multivariate analytical methods. *Biological Resources* 8(2):1918-1932.
- Banker GS, Anderson NR (1987). In: *The Theory and Practice of Industrial Pharmacy*, third ed. Lachman L, Lieberman HA, Kanig JL, editors. Varghese Publishing House; Mumbai pp. 293-299.
- Bolhuis GK, Chowhan ZT (1996). *Materials for direct compaction*. In: Alderborn G, Nystrom C, editors. *Pharmaceutical Powder Compaction Technology*. New York: Merckel Dekker Inc: 419-500.
- Caramella C (1991). Novel methods for disintegrant characterisation, part 1. *Pharmaceutical Technology* 3:48-56.
- Carr RL Jr (1965). Evaluating flow properties of solids. *Journal of Chemical Engineering* 72:163-168.
- Doelker E, Mordier D, Iten H, Humbert-Droz P (1987). Comparative tableting properties of sixteen microcrystalline celluloses. *Drug Development and Industrial Pharmacy* 13(9-11):1847-1875.
- Dong XM, Revol JF, Gray DG (1998). Effect of microcrystalline preparation conditions on the formation of colloid crystals of cellulose. *Cellulose* 5(1):19-32.
- Evans WC (1989). *Trease and Evans' Pharmacognosy*. 13th ed. BailliereTindall pp. 339-377.
- Haque SM, Chowdhury AA, Rana AA, Masum SM, Ferdous T, Rashi MA, Sarker M, Karim MM (2015). Synthesis of microcrystalline cellulose from pretreated cotton obtained from *Bombax ceiba L.* and its characterization. *Bangladesh Journal of Scientific and Industrial Research* 50(3):199-204.
- Hausner HH (1967). Friction conditions in a mass of metal powders. *International Journal of Powder Metall* 3:7-13.
- Jafar EK, Mohammand N, Ahmad S, Hossein C, Nima SM, Hassan N (2011). Studying of effect of the age in *Eucalyptus Camaldulensis* species on wood chemical compounds used in pulping process. *American-Eurasian Journal of Agricultural and Environmental Sciences* 6:17-39.
- Jahan M, Saeed A, He Z, Ni Y (2011). Jute as raw material for preparation of microcrystalline cellulose. *Cellulose* 18(2):451-459.
- Kornblum SS, Stoopak SB (1973). A new tablet disintegrant agent: cross linked polyvinylpyrrolidone. *Journal of Pharmaceutical Sciences* 62:43-9.
- Kumar V, Sanjeev HK, Gilbert SB (2001). Compression, Compaction, and Disintegration Properties of Low Crystallinity Celluloses Produced Using Different Agitation
- Landin M, Martinez-Pacheco R, Gomez-Amoza JL, Souto C, Concheiro A, Rowe RC (1993). Effect of batch variation and source of pulp on the properties of microcrystalline cellulose. *International Journal of Pharmaceutics* 91:133-41.
- Latif A, Anwar T, Noor S (2007). Two step synthesis and characterization of carboxylmethyl cellulose from rayon grade woods pulp and cotton linter. *Journal of Chemistry* 29(2):143-150
- Leppänen K, Pirkkalainen K, Penttilä P, Sievänen J, Kotelnikova N, Serimaa R (2010). Sami-angle x-ray scattering study on the structure of microcrystalline cellulose. *Journal of Physics* pp. 247-249.
- Leppanen K, Seppo A, Tarkkeli M, Matti K, Nina K, Ritava S (2009). Structure of cellulose and microcrystalline cellulose from various wood species and flax studied by X-ray scattering: *Journal of Springer Science + busniness Media* pp. 147-152.
- Mark HF, Bikales NM, Overberger CG, Meufe G (1985). *Encyclopedias of polymer science and engineering*. John wiley and Sons, New York 3:326-340.

- Monika T, Ajay KS, Amit RS (2015). Physicochemical Parameter of Microcrystalline Cellulose and the Most Acceptability in Pharmaceutical Industries. *Journal of Innovations in Pharmaceuticals and Biological Sciences* 2(4):570-578.
- Nuruddin M, Chowdhury A, Haque SA, Rahma A, Farhad SF, Sarwar MJ, Quaiyyum A (2012). Extraction and characterization of cellulose microfibrils from agricultural wastes in an integrated biorefinery initiative. *Journal of Cellulose chemistry and technology* 1:17-20.
- Ohwoavworhwa FO, Adelokun TA (2010). Non-wood Fibre Production of Microcrystalline Cellulose from *Sorghum caudatum*: Characterisation and Tableting Properties. *Indian Journal of Pharmaceutical Sciences* 72(3):295-301.
- Ohwoavworhwa FO, Kunle OO, Ofoefule SI (2004). Extraction and characterization of microcrystalline cellulose derived from *Luffacylindrica* plant. *African Journal of Pharmaceutical Research and Development* 1:1-6.
- Ohwoavworhwa FO, Ogah E, Kunle OO (2005). Preliminary investigation of physicochemical and functional properties of alpha cellulose obtained from waste paper – A potential pharmaceutical Excipient. *Journal of Raw Materials Research* 2:84-93.
- Peck GE, Baley GJ, Mcurdy VE, Banker GS (1989). Tablet formulation and design. In: Lieberman HA, Lechman L, Schwarz JB (eds) *Pharmaceutical dosage forms*. 1 Merce Dekker, New York, 1:75-130.
- Pickard MD (2005). By-products utilization. In: *Bailey's industrial oil products. Edible Oil and Fat Products: Products and applications*. Wiley-Interscience 4(6):46-66
- Rates During their Regeneration from Phosphoric Acid Solutions. *AAPS PharmSciTech* 1:245-250.
- Roshni SV, Yamini DS (2015). Production of Micro Crystalline Cellulose from Corn Husk and Its Evaluation as pharmaceutical Excipient. Department of pharmaceuticals and pharmaceutical technology, L.M. College of Pharmacy, Ahmedabad, Gujarat, India 2(11):2321-2705.
- Sakurada I, Nukushina Y, Ito T (1962). Experimental Determination of Elastic Modulus of Crystalline Regions in Oriented Polymers, *Journal of Polymer Science* 57(165):651-660.
- Setu MNI, Mia MY, Lubna, NJ, Chowdhury AA (2014). Preparation of Microcrystalline Cellulose form Cotton and its Evaluation as Direct Compressible Excipient in the Formulation of Naproxen Tablets, Dhaka. *University Journal of Pharmaceutical Sciences* 13(2):187-192.
- Shangraw RF (1984). Advantages and disadvantages of wet granulation and direct compression processes for making tablets. *Modern Granulation, Tableting and Capsule Technology*, Centre for Professional Advancement. Amsterdam, Netherlands. *Cellulose* 5(1):19-32.
- Shokri J, Adibkia K (2013). Application of cellulose and cellulose derivatives in pharmaceutical industries. *Journal of Intech open science* 1:855-862.
- Soppela I, Airaksinen S, Matti M, Tenho M, Juha H, Heikki R, Jouko Y, Niklas S (2010). Investigation of the powder flow behavior of binary mixtures of microcrystalline cellulose and paracetamol. *Journal of Excipients and Food Chemicals* 1:55-67.
- Stamm AF (1964). *Wood and Cellulose Science*. New York: The Ronald Press Company pp. 132-165.
- Staniforth JN (1996). Powder flow. In: Aulton ME, editor. *Pharmaceutics-The Science of Dosage form Design*. London: Churchill Livingstone pp. 600-615.
- Thoorens G, Krier FB, Evrard CB (2014). Microcrystalline cellulose, a direct compression binder in a quality by design environment-A review. *International Journal of Pharmaceutics* 473:64-72.
- United States Pharmacopoeia (2004). *British Pharmacopoeia* 2004.
- Voravadee S, Pranut P, Duandao A (2016). Preparation and characterization of microcrystalline cellulose from cellulose based-agro waste. *Journal of Engineering and Applied Science* 11(12):2566-2572.
- Yakubu AT, Mumar SSD (2011). Chemical modification of microcrystalline cellulose: improvement of barrier surface properties to enhance surface interaction with some synthetic polymers for biodegradable packing material processing and application in textile, food and pharmaceutical industry: *Journal of Pelagia Research Library Advance In Applied Research* 2:532-540.

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