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Extraction and characterization of microcrystalline cellulose obtained from the back of the fruit of *Lageriana siceraria* (water gourd)

Achor M*, Oyeniyi YJ and Yahaya A

Faculty of pharmaceutical sciences, Usmanu Danfodiyo University, Sokoto, Nigeria.

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ABSTRACT

The work was aimed to investigate the suitability of acid modified alpha cellulose obtained from the back of the fruit of *Lageriana siceraria* (water gourd) in production of pharmaceuticals, particularly solid dosage forms. Cellulose is the commonest Filler/Binder or disintegrant used in tablet formulation. Cellulose was extracted from the fruit of *Lageriana siceraria* and subjected to modification by controlled acid micronisation to produce microcrystalline cellulose (LS-MCC) using 2.5N hydrochloric acid and Avicel pH 101 was used as the basis for comparison. The powder and flow properties of the microcrystalline cellulose was investigated and the result of both the LS-MCC and Avicel pH 101compared. Microcrystalline cellulose (LS-MCC) displayed superior swelling and hydration capacities as compared to Avicel PH101 but lesser flow properties in view of its higher angle of repose, Carr's and Hausner's index.

INTRODUCTION

Significant amount of wastes is being produced each day, which contain high quantities of organic matter. The agricultural wastes produced in a particular period of the year pose potential pollution problems. Therefore, an efficient utilization of such agricultural wastes is of great importance not only for minimizing the environmental impact, but also for obtaining a higher profit (Nuruddin *et al*., 2011). Water gourd is widely produce in west Africa and most importantly in Nigeria in large bulk, hence it also grow wild on its own, with little or no consumption, its use is limited to certain people as food and drink containers, now use only as ornamentals, and tourism in African countries, it generate bulk waste, hence not use off.

Lageneria siceraria (bottle guard) also known as calabash in Nigeria is a fleshy, densely hairy, indehiscent, green, maturing yellowish or pale brown, pulp drying out on ripening, leaving a thick, hard, hollow pod. Seeds are many, embedded in a spongy pulp, compressed, with two flat facial ridges, in some variants rather irregular and rugose (Rahman, 2003).

Existence of cellulose as the common material of plant cell walls was first recognized By Anselm Payen in 1838. It occurs in almost pure form in cotton fiber and in combination with other materials, such as lignin and hemicelluloses, in wood (of which water gourd fits into), plant leaves and stalks, etc. Although generally considered a plant material, cellulose is also produced by some bacteria. Microcrystalline cellulose is purified, potentially depolymerized cellulose prepared by treating Alpha cellulose obtained as pulp from fibrous plant material with mineral acid. Microcrystalline cellulose is basically cellulose and can only be derived from a specialized grade of Alpha cellulose from fibrous plant, treated with mineral acid. Commercially available MCC is derived from both Gymnosperm and other soft wood and from hard wood dicotyledon. Microcrystalline cellulose has many uses in both food, cosmetics and pharmaceutical industries as an anti-caking agent, emulsifier, stabilizer, dispersing agent, thickeners and gelling agent and one of the most used filler-binder in direct tablet compression is due to its excellent binding properties, where its use as a dry binder. The objective of this study is to investigate the suitability of microcrystalline cellulose obtained from *Lageriana siceraria* as a Tableting excipient.

* *Corresponding Author*
Faculty of pharmaceutical sciences, Usmanu Danfodiyo University, PMB 2346 Sokoto, Nigeria. E-mail: munchors001@yahoo.com

MATERIALS AND METHODOLOGY

Materials

Dried bark of the fruit of the plant *Lageriana siceraria* were collected at Sokoto and identified in Department of Pharmacognosy, Usmanu Danfodiyo University, Sokoto. Avicel PH 101, Sodium hydroxide (BDH, England), 3.5% w/v sodium hypochlorite as (Jik®, Reckitt and Colman Ltd, Nigeria), hydrochloric acid (Fisons, UK), xylene, phloroglucinol and iodine crystals (Hopkin and Williams, London) were used as obtained. All other chemicals used were of analytical or reagent grade and water was distilled.

Methodology

Isolation of α- cellulose

A dried bark of the fruit of the plant *Lageriana siceraria* was obtained and grinds in to powder for the extraction. A method of Ohwoavworhua *et al* (2005) was adopted with a slight modification. 50 g quantity of this material was placed in a stainless steel container to which was added 1.0 L of 2 % w/v sodium hydroxide and digestion effected for 5 h at 80^0 C in a water bath, Following thorough washing and filtration, it was bleached with 1.0 L of a 1:1 aqueous dilution of sodium hypochlorite for 15 min at 80^0 C. The material was then washed sufficiently with water and treated with 1.0 L of 17.5 % w/v sodium hydroxide at 80^0 C for 1 h. The resulting α-cellulose was washed thoroughly with distilled water.

Bleaching

The extraction process was then completed by whitening with a 1:3 aqueous dilution of sodium hypochlorite for 60 min at 80^0 C and 1:1 for 24hr and subsequent washed with water until neutral, the procedure was repeated for the remaining samples. The cellulose material was filtered, and the water manually squeezed out using calico cloth to obtain small lumps, which were dried in a Hot air oven at a temperature of 60^o C for 1 h.

Production of microcrystalline cellulose (LS-MCC)

Method of preparation of microcrystalline cellulose adopted by Ohwoavworhua *et al* (2005) with a slight modification was used. A 30 g quantity of the α-cellulose obtained was placed in a glass container and hydrolyzed with 500 ml of 2.5N hydrochloric acid, at a boiling temperature of 105^o C for 15 min. The hot acid mixture was poured into 1.5 L of cold tap water which was followed by vigorous stirring with a spatula and allowed to stand overnight. The microcrystalline cellulose obtained by this process was filtered, washed with water until neutral, filtered, pressed and dried in a hot air oven at a temperature of 60^o C for 60 min. in a hot air oven (NUVE FN055 oven, Germany). Following further milling and sieving, the fraction passing through 650µm sieve aperture was used for the characterization.

Physicochemical properties of LS-MCC

The Organoleptic characteristic (Taste, odour and colour), identification, organic impurities (with phloroglucinol), starch and dextrin, solubility and total ash were carried out in accordance with British Pharmacopoeia (2004) specifications. An optical microscope was used for preliminary assessment of the nature of particles.

PH determination:

1 g of the powder material was shaken with 50 ml of distilled water for 5 min and the pH of the supernatant liquid was determined using a pH meter (3510 model, Jenway, England).

Total ash determination:

Ash content was estimated by the measurement of the residue left after combustion in a furnace. 2 g of the powder was weighed and the weight of the empty furnace and furnace containing the powder was determined using an Electronic balance. The weight of the furnace containing the residue was also determined and the total ash content computed using expression below

Ash content (%) = $w_1 – w_2/ w_1 x\ 100$……(1).

Where W_1 is the weight of the sample prior to the combustion and W_2 is the weight after combustion.

Powder Properties

Sieve analysis

A sieve-shaker (WQS Vibrating screen, B Bran Sci. and instrument company England) was used for this assessment. Test sieves were arranged in a descending order after recording individual weight of the empty sieves and the pan. A 10 g quantity of MCC powder was placed on the top sieve and the set-up was shaken at amplitude of 1.50 mm/g for 5 min. The weight of material retained on each sieve was determined by subtracting the weight of the empty sieves from the weight of sieves containing the powder. Percentage retained was then determined.

Particle size analysis

A given amount of the sample was mounted in glycerin and view under Electron microscope (XS2-21Kyowa optical limited Japan) and magnified with Digital electron eye piece (YJEYE01-130) and the average diameter of the magnified particles was determined taken the sample size of 200 particles

Flow Properties

Angle of Repose

The static angle of repose, a, was measured according to the fixed funnel and free standing cone method. 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:

$$\text{Tan } a = h/r \quad \ldots\ldots\ldots\ldots\ldots\ldots 2$$

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

Bulk and Tap Densities

A 5.0 g quantity each of the powder samples was placed in a 50 ml clean, dry measuring cylinder and the volume, V_o occupied by each of the samples without tapping was determined. After 200 taps occupied volumes, V_{200}, were determined. The bulk and tap densities were calculated as the ratio of weight to volume (V_0 and V_{200} respectively). The Carr's index and Hausner's ratio were determined from the values of the bulk and tapped densities results obtained above.

True Density

The true densities (D_t), of cellulose powders were determined by the liquid displacement method using xylene and pycometer. 0.5 g quantity of cellulose powder was placed in a dry pre weighed pycometer and the rest filled with 50 ml xylene (SG 0.86) as the immersion fluid, the weight of the pycometer filled with only liquid has previously been established and density of the powder was computed according to the following equation:

$$D_t = w\,[(a + w)\text{-}b] \times SG \quad \ldots\ldots\ldots\ldots 3$$

Where w is the weight of powder, SG is specific gravity of solvent, a is weight of bottle + solvent and b is weight of bottle + solvent + powder.

Powder Porosity

This was derived from the values of true and bulk densities when fitted into the equation:

$$e = 1\text{-}P_B/D_t \times 10 \quad \ldots\ldots\ldots\ldots\ldots 4$$

Where P_B is the bulk density, D_t is the true density and e is the porosity.

Hydration Capacity

The method of Kornblum and Stoopak (1973) was used. A 1.0 g each of the samples was placed in each of four 15 ml plastic centrifuge tubes and 10 ml distilled water was added from a 10 ml measuring cylinder and then Stoppard. The contents were mixed for 2 min. The mixture was allowed to stand for 10 min and immediately centrifuged at 1000 rpm for 10 min on a bench centrifuge (TDL4 Tabletop low speed centrifuge Gallenkamp, England). The supernatant was carefully decanted and the sediment weighed. The hydration capacity was taken as the ratio of the weight of the sediment to the dry sample weight.

Swelling capacity:

The swelling capacity of the powder was estimated as described by Okhamafe *et al.* (1991) with slight modification. In this method the tapped volume occupied by 3.0 gm of the powder V_x, was noted and the powder was dispersed in 85 ml. of water and the volume made up to 100 ml with water. After 24 hrs of standing, the volume of the sediment Vv was measured. The swelling capacity was then computed as the ratio of V_v/V_x.

Moisture Sorption capacity

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

$$W_2\text{-}W_1/W_1 \text{ X } 100 \quad \ldots\ldots\ldots\ldots 5$$

Where W_1 is the weight of the samples before exposure, W_2 is the weight of the samples after exposure.

Loss on drying

The powder sample 3 g was transferred into a Petri dish and then dried in an oven at 105 ^oC until a constant weight was obtained. The % moisture content was then determined as the ratio of weight of moisture loss to weight of sample expressed as a percentage.

RESULT AND DISCUSSION

The yield of α-cellulose was approximately 29% w/w of the original material. The yield of the microcrystalline, LS-MCC, obtained from α-cellulose was 44 % w/w. Thus the yield of CP-MCC was 16 % w/w of the starting powdered material.

Table 1. shows the result of some physicochemical properties carried out in accordance with British Pharmacopoeia (2004) specification. The Organoleptic properties of the LS-MCC produced were good as the material was odorless, tasteless, almost white granular powder. The value obtained for the total ash was 2.5 %. The value of which is indicative of the inorganic content of sample and the level of care taken in the preparation of the substance. And the pH was almost neutral (6.8). The result of sieve size analysis is shown in Fig. 2. Over 85 % of the particle population is less than 250 µm for both the LS-MCC and Avicel PH101, making them to fall under the class of conventional powders, with the average particle size diameter of 42.184 µm for LS-MCC as determined using a micrometer eye piece.

The flow properties of powders are essential in determining the suitability of a material as a direct compression excipient. The angle of repose, Hausner's index and Carr's percent compressibility are considered as indirect measurements of powder flowability, The Hausner's index is indicative of inter-particle friction, while the Carr's index shows the aptitude of a material to diminish in volume (Rubinstein, 1996). As the values of these indices increase, the flow of the powder decreases. The values obtained for all the materials (LS-MCC and Avicel PH 101) shown in Table 2 were high giving an indication of poor flow of the material. This could be attributed to the small particle size of the material. Consequently, a glidant will be needed when these materials are to be used in solid dosage production processes.

Table. 1: Some physicochemical properties of microcrystalline cellulose (LS-MCC).

TEST	LS- MCC
Organoleptic	Odorless, almost white, tasteless granular powder
Identification	Turns violet blue with iodinated Zinc chloride
Organic impurities	No red colour with acidified phloroglucinol
Starch and dextrin's	No blue/ reddish brown ppt. with Iodine solution
pH	6.8
Water soluble substances	insignificant
Total ash content	2.5 %
Microscopy	Irregular shaped particles seen (Fig. 1.)

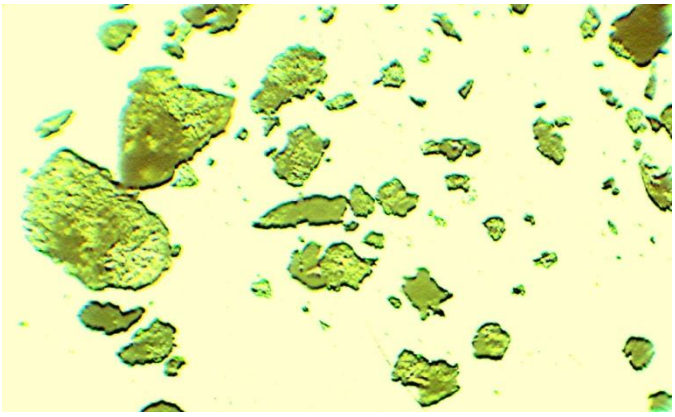

Fig. 1: photomicrograph of LS-MCC (Mag. x 100).

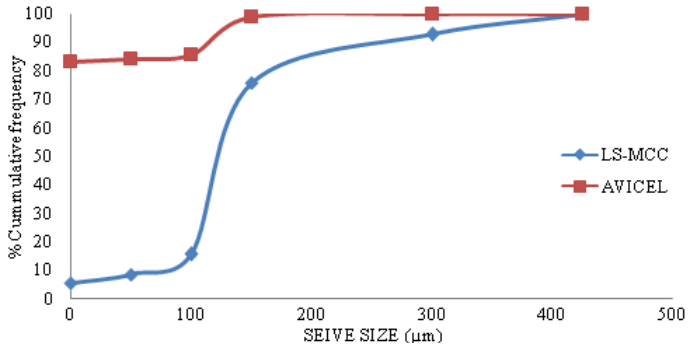

Fig. 2: percentage cumulative frequency (undersize) versus sieve size.

True density is the density of the solid material excluding the volume of any open and closed pores. Depending on the molecular arrangement of the material, the true density can equal the theoretical density of the material and therefore be indicative of how close the material is to a crystalline state or the proportions of a binary mixture. The true density for LS-MCC was higher than that of the Avicel as seen in Table 2. Swelling which is generally accepted as an indication of tablet disintegration ability can be assessed by the determination of hydration capacity, swelling capacity and moisture sorption profile. The hydration capacity value indicates that the LS-MCC is capable of absorbing and retaining more water than Avicel. The swelling capacity which reflects the increase in volume of samples following water absorption followed the same trend as the hydration capacity index and was found to be 6.55 %. The higher hydration and swelling capacities values observed for LS-MCC compared to Avicel PH101 could possibly be due to the higher powder porosity of LS-MCC as seen in Table 2. Porosity consists of volume of the pores relative to the envelope volume used, the porosity of pharmaceutical materials and medical devices can impact production, material movement, and pharmacokinetic behavior. The moisture sorption capacity is important since it reflects the relative physical stability of tablets made from it when stored under humid conditions, the result in Table 2 shows that the LS-MCC has higher moisture sorption capacity than Avicel.

Table. 2: Micromeritic properties of LS-MCC and Avicel.

Parameters	LS-MCC	Avicel PH 101
True density (g/ml)	2.00 (0.23)	1.50 (0.22)
Bulk density (g/ml)	0.29 (0.06)	0.32 (0.01)
Tapped density (g/ml)	0.39 (0.01)	0.35 (0.01)
Porosity (%)	8.50 (0.34)	7.80 (0.18)
Angle of repose	32.9 (1.47)	28.4 (1.61)
Hausner's index	1.31	1.28
Compressibility index (%)	23.5	19.5
Hydration capacity (%)	6.55 (0.06)	5.96 (0.04)
Swelling capacity	1.18 (0.21)	1.09 (0.18)
Moisture sorption capacity (%)	0.28 (0.05)	0.19 (0.18)
Loss on drying (%)	9.0 (0.08)	7.6 (0.12)

**value is mean and standard deviation is in parenthesis, number of replicate = 3.*

CONCLUSION

Microcrystalline cellulose obtained from the bark of the fruits of *Lageneria siceraria* conformed to the official specifications in the British Pharmacopoeia (2004). The swelling and hydration parameters indicate that LS-MCC would be a better disintegrant than Avicel PH 101. Despite the decreased flow properties as compared to Avicel, LS-MCC would be expected to produce tablets with desired properties when used as a direct compression filler-binder.

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