Journal of Pharmacy and Bioresources
Vol. 1 no. 1, pp. 41-45 (September 2004)

Journal of
PHARMACY AND
BIORESOURCES

Physicochemical and powder properties of alpha- and microcrystalline-cellulose derived from maize cobs

Jennifer D. Audu-Peter[1*], Joseph E. Ojile[2] and Partap G. Bhatia[2]

[1]*Department of Pharmaceutics and Pharmaceutical Technology, University of Jos, Nigeria.*
[2]*Department of Pharmaceutics and Pharmaceutical Microbiology, Ahmadu Bello University, Zaria.*

Received 19th March 2004; Revised, accepted 16th July 2004

Abstract

Cob alpha-celluloses (CAC) was extracted from maize cobs by defibering, delignification and bleaching; then subjected to acid hydrolysis to obtain Cob- microcrystalline-cellulose (CMCC). Their physicochemical properties were evaluated and compared with those of Avicel®, a commercial variety of microcrystalline cellulose. Identification tests, and tests for possible contaminants were performed on them. Their powder properties were determined to ascertain their suitability and usefulness in tabletting. The results showed that the extraction process was adequate, as pure alpha cellulose was obtained. Also, the CMCC extracted was found to have comparable powder properties with Avicel®; and one can say that it can be used for similar function as Avicel® in pharmaceutical processes.

Keywords: Cob alpha-cellulose (CAC); Cob microcrystalline-cellulose (CMCC); Avicel®; Physicochemical powder properties.

Introduction

Cellulose (and its derivatives like hydroxy ethylcellulose, methylcellulose, sodium carboxymethyl cellulose and microcrystalline cellulose) is one of the frequently used excipient in the pharmaceutical industry; and in Nigeria, they are mostly imported. The high tariff on imported goods and the high rate of currency exchange rate has made it expensive for local manufacturers of pharmaceuticals. Workers like Okhamafe *et al.* (1995) have found cellulose in significant quantity in maize cobs. Cellulose is the main polysaccharide of plant cell wall, made up of linear chains of ß -1,4-linked glucose residues. Powdered cellulose is the purified, mechanically disintegrated cellulose prepared by processing cellulose obtained as a pulp from plant cell materials. (Bolhius and Lerk, 1973). It exists in various grades and exhibit degrees of fineness ranging from a free flowing dense powder to a coarse, fluffy non-flowing material. Gibbon and Pain, 1985, have elucidated on the different varieties of maize, grown for its grains, which are primarily used as food for man and animals. It is also used as a base for industrial products such as oils, syrup, and starch. The grain may be fermented to produce beverages and distilled to produce whisky or industrial alcohol, acids, acetaldehyde, acetone and glycerol. The cob can be used as fodder,

* Corresponding author. *E-mail address*: drambia@yahoo.com

bedding, building material, fuel and more recently, a source of cellulose (Okhamafe *et al*, 1995).

Experimental

Extraction of cob alpha-cellulose (CAC) from maize cobs. Dry maize cobs were obtained from a local maize seed harvesting and shearing mill in Samaru village, Zaria, Nigeria. Debris, sand, leaves and other dirt were removed by brushing. The cobs were crushed into smaller pieces using a locally made wooden mortar and pestle and spread on a stainless steel tray to sun dry for 48hours. These were then dried in a hot air oven (Galenkamp, model BS size 3, England) for 4 hours at 60°C. The coarse powder was then milled into finer powder mass at a local grain-milling machine whose milling chamber was thoroughly washed and dried before use. The powder was sieved with a nest of sieves (Endecott, London, U.K.) set to vibrate on a mechanical sieve shaker for 15 minutes. Fractions that were retained on 250μm sieve and below were collected and used to extract alpha-cellulose using the method of Okhamafe *et al.* (1991). The alpha-cellulose so obtained was milled using a blender, (Atlas Exclusive Alzico, Italy) sieved and the powder retained on 150 μm sieve sizes and those that passed through were collected and used for the work. The yield of CAC was 21%. The procedure was repeated several times to obtain sufficient CAC for the work.

Production of cob microcrystalline-cellulose (CMCC) from CAC. The method of Swinyard and Lowenthal (1990) was used to modify CAC to CMCC and the CMCC treated as reported by Audu-Peter *et al.* (2002) whereby the yield of microcrystalline cellulose from alpha cellulose was about 90%.

Physicochemical investigations on the CAC and CMCC

1. Chemical tests. Identification tests were carried out on the powders to ascertain their identity and presence of possible contaminants like lignin, free reducing sugars and monosaccharides. (Evans 1997, Brain and Turner; 1975).

2. Particle size distribution analysis using light microscope. The eyepiece graticle was calibrated with reference to a stage micrometer. Samples of each powder were prepared and placed on a slide and mounted in dilute glycerol to adequately disperse particles and prevent agglomeration. The samples were viewed using the X40 objective lens and X10 eye lens. The diameter of about 100 particles per sample were randomly selected and measured. The mean particle size, the standard deviation and the size distribution were determined for each powder sample and the frequency Vs particle size range was plotted to give the particle size distribution curve.

3. Determination of moisture content. The B.P (1980) method was used and the ratio of the weight lost with the final weight expressed in percentage was the moisture content.

4. Determination of moisture sorption capacity. 1.0g of each powder sample was taken and spread evenly and thinly on a petri dish whose weight had been predetermined. A humidifier was allowed to equilibrate to 100% relative humidity by leaving distilled water in it for 72hours. The samples were then placed in the humidifier sealed, and left for seven days. The samples were then reweighed, the weight gained expressed as percentage being the sorption capacity for each sample.

5. Determination of powder properties of CAC and CMCC powders.

(a). Angle of repose was determined using the funnel method. The average of three determinations was taken.

(b) Powder flow rate was determined by using the flow meter (Erweka). The average of three determinations was taken.

(c) Carr's index determination for each powder sample was determined using same methods as those of Audu-Peter *et al.* (2002).
(d) True density of ach powder sample was determined according method of Okhamafe *et al.* (1991) and Audu-Peter *et al.* (2002).

Results and Discussion

The result of the chemical identification tests performed on the powder samples all gave positive identification and tests for possible contaminants were negative. The tests showed that CAC and CMCC were totally free from presence of lignin as a result of lignin solubilizing treatment with 3.5%v/v nitric acid. They were also free of tannins and other pigments as a result of the treatment with alkali, acid washings and bleaching. The treatment with 2% NaOH and 2% sodium sulphite were also intended to digest the cob fibres (Okhamafe *et al., 1991*), loosening them for further action by subsequent chemical reactions. Further treatment with 17.5%NaOH was for dissolving β- and γ-cellulose, being soluble in the solvent while α- cellulose was left intact as it was not soluble in the 17.5%NaOH solution (Okhamafe *et al,* 1991). The final residue obtained was therefore cob alpha cellulose (CAC) as identified by the chemical tests. Positive identification was also made on CMCC as microcrystalline cellulose, with Avicel® as the standard which was positively identified by the test. The larger mean particle size of powders (Table 1) observed with CAC may largely be as a result of unhydrolysed bonds in the cellulose structure. The hydrolyzing of these bonds gave rise to CMCC, which has smaller particle sizes, and are more spherical in shape than CAC. The mean particle sizes for CMCC and Avicel are comparable which is significant for the local production of microcrystalline cellulose, as they are likely to have comparable powder properties. A plot of the particle size distribution Vs frequency (Fig.1) shows that CAC has a wider distribution of particles than CMCC and Avicel this can be attributed to the more amorphous and longer size of particles of CAC than CMCC particles (Musa, 1996) and CAC is more fibrous than CMCC particles (Okhamafe *et al.* 1991, Staniforth, 1993). The orientation of the particles of CAC on the micrometer stage of the microscope may have led to larger average sizes than the more uniform particles of CMCC and Avicel. CMCC has a normal distribution curve while Avicel® has a positively skewed one.

The higher values of moisture content for CAC maybe attributed to the larger sizes of its particles than the other two. These structures when hydrolyzed as in CMCC led to their breakdown and reduction in the moisture in the microcrystalline cellulose. Presence of moisture in powders for tableting is desirable (Rees and Shotton, 1971, Rees and Hersey, 1972) and formulation scientist generally consider 3% moisture content as adequate (Okhamafe *et al.*, 1991). The high values of sorption capacity of CAC may also be due to its amorphous and fibrous texture (Musa, 1996). Moisture sorption capacity of a pharmaceutical excipient is critical as it serves as an indication of the excipient affinity for water, which can be desirable if it will be used as a disintegrant and undesirable if it will affect the chemical stability of a hydrolysable constituent, like aspirin. CAC will have better disintegrant properties than CMCC and Avicel®; but Avicel® will make a better diluent for drugs sensitive to moisture.

A good pharmaceutical powder for direct compression is expected to have high bulk density; an indication of its packing geometry. A powder has bulk density by virtue of its occupying a certain volume, and on vibration, the bed mobilizes the particles so that on stopping the vibration the bed come into static equilibrium but occupies a different spatial volume from the initial one, changing the bulk volume to tapped bulk volume. Avicel® with bulk density of 0.266g/cm^3 and

tapped density of 0.374g/cm^3 has the best property here. True density of a powder does not change, but it indicates how easily the particles will flow. The heavier the particles the more difficult will it flow and this can be observed from the result where Avicel® with the least true density of 1.408g/cm^3 has flow rate of 0.987g/s. The anomaly observed with CMCC (True density, 1.473g/cm^3 and flow rate at 0.807g/s) and CAC (true density 1.503g/cm^3 and flow rate at 0.908g/s) need to be further investigated. But it may be attributed to their particles sizes where CAC has larger size than CMCC.

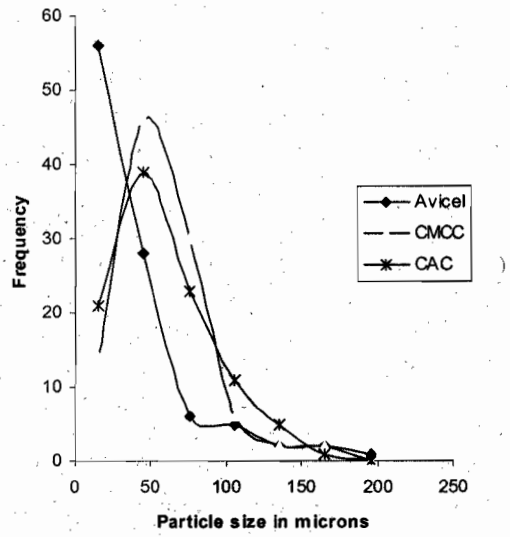

Fig.1. Frequency Vs particle size distribution of Avicel, CMCC and CAC powders.

Table 1. Mean size, standard deviation (SD) and standard error of the mean (SEM) value for CAC, CMCC and Avicel® powders.

	CAC	CMCC	AVICEL®
Mean particle size (μm) =$\sum FX / \sum F$	58.40	38.90	39.20
Standard deviation, SD = $\sqrt{(\sum FX^2 - \sum FX^2 / n/n-1)}$	34.40	20.10	37.20
Standard error of the mean, SEM = SD/ $\sqrt{n}$	3.95	2.01	3.70

Table 2. Properties of CAC, CMCC and Avicel® powders

Properties	CAC	CMCC	AVICEL®
Bulk density (g/cm^3)	0.244	0.219	0.266
Tapped density (g/cm^3)	0.356	0.348	0.374
Carrs' index (%)	37.10	37.06	28.88
Flow rate (g/s)	0.908	0.807	0.987
Angle of repose (°)	47.54	41.54	45.93
True density (g/cm^3)	1.503	1.473	1.408
Hausners' index	1.46	1.59	1.41
Packing fraction	0.15	0.15	0.19
Bed porosity (%)	85	85	81
Moisture content (%)	3.1	2.9	2.9
Moisture sorption capacity (%)	23.4	22.1	11.4

The values for bed porosity gives indication on how easily the powders settle after vibration and both CAC and CMCC have bed porosity of 85%, which compared favourably with values for Avicel® at 81%.

Carr's index, angle of repose measurement and Hausners index are indices for predicting the flow properties of powders. Higher values indicate poor flow and since all three powders show high values, poor flow was observed and workers like Tsai *et al.* (1998) advice the use of larger particle sizes of microcrystalline cellulose or granulating it when it is to be used in direct compression. The packing fraction of a powder is the ratio of its bulk density to the true density and the value has an inverse relationship with particle sizes and it is affected by shape of powder particles. Porosity is an important factor in tableting as it affects die filling and subsequently uniformity of content from batch to batch.

References

Audu-Peter J.D., Ojile J., Bhatia P.G. and Bukar B.B. (2002); Evaluation of properties of direct compression paracetamol tablets containing only microcrystalline cellulose derived from maize cobs. *W. Afr. J. Biol. Sci.* 43-50

Brain T.K. and Turner R. (1975) The phytochemistry of plants. Wright Scientechnica Bristol. pp 267-280.

Bolhius G.K and Lerk C.F. (1973) *Pharm Week.blad* 108:469. From Rubinstein (1993), Tablets, In: Pharmaceutics, The Science of Dosage Forms (Aulton, M.E. Ed) ELBS with Churchill Livingstone Ch.18 pp 304-321.

British Pharmacopoeia (BP 1980) Vol. I and II. Pharmaceutical Press, H.M S.O., London.

Evans W.C. (1997) "Cell differentiation and Ergastic cell contents" and "carbohydrates" In: Trease and Evans Pharmacognosy. 14th Ed. W.B Saunders Co. Ltd., London.

Gibbon D. and Pain A. (1985). Crops of the Drier Regions of The Tropics. Intermediate Tropical Agric Services. Longman, Singapore Pub (Pte) Ltd pp 79-83.

Musa A.N (1996) Physicochemical and tabletting properties of cellulose (treated and untreated) derived from sugar cane. M.Sc. Thesis, Department of Pharmaceutics and Pharmaceutical Microbiology, Ahmadu Bello University Zaria.

Okhamafe A.O; Igboechi A.C. and Obaseki T. (1991). Cellulose extracted from groundnut shells and rice husks; Preliminary Physicochemical characterisation. *Pharmacy World Journal* 8 (4): 120-123.

Okhamafe A O. Ejika E.N; Akinrinola F.F and Ubuane-Insedgbo A. (1995). Aspects of the tablet disintegrant properties of cellulose derived from bagasse and maize cobs, *J. West Afr. Pharm* 9 (1); 8-13.

Rees J. E. and Shotton E. (1971) *J. Pharm. Sci.* 60; 704. From: Bangudu A.B. (1985) Production of paracetamol tablets by mass screening method. Ph. D. Thesis, Department of Pharmacy, Chelsea College University of London.

Rees J.E and Hersey J. (1972) *Pharm. Acta Helv.* 47: 235. From: Bangudu A.B. (1985) Production of paracetamol tablet by mass screening method. Ph.D. Thesis, Department of Pharmacy, Chelsea Collage, University of London.

Swinyard E.A and Lowenthal W. (1990) In: Remington's Pharmaceutical Sciences, A.R. Gennaro, (Ed) 18th Ed. Mack Printing Co; U.S.A. pp 1035-1319.

Tsai T., Jen-sen W., Hsiu-O H. and Ming-Thau S. (1998). *J. Pharm. Sci.* 87(1), 117-122