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Research Article

THE PHYSICOCHEMICAL CHARACTERISTIC OF MICROCRYSTALLINE CELLULOSE, DERIVED FROM SAWDUST, AGRICULTURAL WASTE PRODUCTS

*OYENIYI, Y.J[1] AND ITIOLA, O.A[2]

[1]Department of Pharmaceutics, & Pharmaceutical microbiology, Faculty of Pharmaceutical Sciences, Usmanu Danfodiyo University, Sokoto, State Sokoto Nigeria. [2]Department of Pharmaceutics, & Industrial Pharmacy, Faculty of Pharmacy, University of Ibadan, Oyo State, Nigeria. Email: DrJimiOyeniyi@gmail.com

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ABSTRACT

This study was devoted to the extraction and purification of microcrystalline cellulose, (MCC) from Sawdust, an agricultural waste product, readily available in most developing countries at no cost.

Microcrystalline cellulose is an important ingredient in both pharmaceutical and food industries. Alkali delignification of sawdust, followed by bleaching and acid depolymerisation was employed. The microcrystalline cellulose obtained, was thereafter evaluated for its physicochemical characteristic, in comparison with commercially available Avicel PH 101.

The percentage yield of MCC from this waste product was 68% W/w, this is relatively high enough to stimulate large scale commercialization of the extraction of MCC.

Keywords: Sawdust, Microcrystalline cellulose and physicochemical characterization.

INTRODUCTION

In the world today, any green product, which will ensure the utilization of waste product, for the production of valuable material (resources recovery) and the conservation of the energy in the industrial process with the view of reducing carbon emission and global warming, is a welcome development.

Sawdust is an agricultural waste products obtained in the process of smoothing plant and timber. Plant cell wall is the backbone structure of all plant materials, and cellulose is the chief constituent of plant cell wall and perhaps the most abundant organic matter available in the world today.[1]

Cellulose was discovered in 1838 by Anselme Payne, who isolated it from plant material and determined its chemical formula.[2,3,4]

Cellulose is a liner polysaccharide consistency of several D-glucose units linked together by ß, 1-4 glycosidic bond.[5]

it is tasteless, odorless, white crystalline material, they are about 2000 to 4000 glucose units all linked by ß ,1,4 glycoside bond hence the chain length is not constant.[6] Microcrystalline cellulose can be biodegrade to its constituent glucose units via acid hydrolysis at high temperature and through enzymatic processes.[7,8] Microcrystalline cellulose today, had revolutionaries tableting technology because of its unique compressibility and carried capacity. It exhibit excellent property as excipients for solid dosage form as its compact well under minimum compressional pressure. It's safe and physiological inert.[9]

MATERIALS AND METHODS

Sawdust collected from saw mill in Kano. All other reagents and solvent used were of pharmaceutical or analytical grade.

Method of Preparation

a. Preliminary treatment and size reduction: the sawdust collected was dried at 60^{0}c for 24 hours. This was then milled with atlas alzcico milling machine and sieved using Endecott sieves attached for vibrator. Fraction retained by 250 micrometer sieve mesh was treated with 17.5% w/v sodium hydroxide solution in a 12-litre volume stainless steel bowl, immersed in a water bath maintained at 100^{0}c for 12 hour.

b. Bleaching with sodium hypochlorite: The residue which is the alpha cellulose in its crude form was then bleached with 100 ml of 1:1 dilution of 3.5% w/v sodium hypochlorite solution; this was conducted at 80^{0}c for 8 hours. This method was repeated until the material become milky white

c. Whitening with 20% v/v Hydrogen peroxide: The resulting alpha cellulose was further treated with 1L, 20% hydrogen peroxide at 40^{0}c for 2 h this was also repeated until material became snow white. The obtained cellulose was rinsed, filtered, present and dried at room temperature for 48 hours and then dried in a Gallenkamp oven at 60^{0}c for 1 hour.

d. Production of MCC: To five hundred millilitre of 2.5M hydrochloride acid, heated to 105^{0}c, 500g of extracted cellulose was added to the boiling acid and left for 15 minute. The resulting crystalline cellulose was collected by filtration, which was then washed with aqueous ammonia solution and de- ionized water. MCC obtained was then dried at room temperature to a constant weight.

e. Determination of yield: The obtained microcrystalline cellulose were weighted and the yield was calculated using equation (1)

Yield (%) = A / B x 100 ……..1

A (mg) = Weight of obtained microcrystalline

B (mg) = Weight of alpha cellulose

f. Particular size analysis: Using a light microscope fitted with graticle the particle size of fifty particle were determined. The average particle size of MCC was however calculated statistically.

g. Flow Rate of MCC Powder: Ten gram of MCC was passed through the erweka flowability tester. The time taken for 10g of the material to flow was recorded. The same procedure was repeated twice and the average flow rate calculated from the date obtained.

h. Bulk density & Tapped density: Using a 100ml capacity measuring cylinder and fifty gram of obtained MCC the bulk and tapped volume of MCC were determined. Bulk and tapped density of obtained MCC were calculated using equation 2 and 3

BD = 50/BV …… (2)

TD = 50/TV……..(3)

BD = Bulk density

TD = Tapped density

BV = Bulk volume of Mcc

TV = Tapped volume of Mcc

i. Carr's Index and Hausner Ratio Determination: Data values obtained from bulk density and tapped density from BD and TD above were used to calculate the Carr's index and hausner ratio, equation 4,and 5

Carr's index = Compressibility index

$$= 100 \times \frac{(TD-BD)}{TD} \quad \ldots\ldots\ldots\ldots\ldots (4)$$

Hausner ratio = TD /BD …….. (5)

j. Angle of Repose determination: This was determined following standard U.S.P 2010 method.

k. Moisture content: An evaporated dish containing 10 grams of MCC was heated to 105^{o}c in a gallenkamp oven, until such a time that a constant weight was obtained. The average for three readings was obtained.

MC = 100 (iw-fw/fw) ……(6)

Chemical Evaluation of MCC

The following tests were conducted on the produced MCC, to confirm the identity of extracts.

A. Test for the Presence of Lignin

To 100 mg of obtained MCC placed on a glass slide, and moistened with concentrated hydrochloric acid, two drops of phloroglucinol was added and heated, until the liquid content was completely evaluated. Slide was thereafter examined under light microscope for any coloration.

B. Test for the presence of sugar

Standard B.P test for free reducing sugar was conducted on the extract.

C. Test for the presence of starch

To 0.2g of o obtained MCC few drops of N/50 iodine solution were added, followed by addition of conc. (66.67%v/v) of sulphuric acid. Any change in colour and swelling was noted.

D. Confirmation Test for cellulose

To 50gm of MCC, placed in a test tube, 5ml of 5% W/V of potassium hydroxide solution was added, heated and observed for any canary yellow colouration.

E. Total Ash Determination

1g of sample was placed in a tarred dish, this was thereafter placed in hot oven maintained at 450^{o}C for 3hr. the residue left after 3hr was collected and transferred into a desiccator. The weight of the residue was also noted.[12]

F. Elementary Analysis of Ash Residue

Know weight of the ash residue was mixed with few drops of Conc trioxonitriate iv acid until a slurry is formed. The slurry was thereafter dispersed in 100ml of de ionized water, the concentration of elements present were determined using atomic absorption spectrophotometer.[13]

RESULT

Yield 87% w/w

The yield of microcrystalline cellulose from crude sawdust was 87% w/w, this is relatively high enough to stimulate large scale industrial processing of microcrystalline cellulose from sawdust.

Average Particle Size 62 µm

Sawdust microcrystalline cellulose contained particles of various sizes as show in table I below; the calculated average particle size of 62 micrometer can be consider adequate as most commercially available microcrystalline cellulose posses particle size of between 60 - 70 µm.

Table 1: Particular size distribution of sawdust MCC

Size range (µm)	% frequency	class mark	fixi	%cum. Freq.
1-50	60	25.5	1530	60
51-100	22	75.5	16661.0	82
101-150	9	125.5	1129.5	91
151-200	5	175.5	8776.5	96
201-250	3	225.5	676.5	99
251-300	1	275.5	275.5	100
Total	**100**	**6150**		

X = mean particles size = $\frac{6150}{100}$ = 61.5µm

Table 2: Particles size distribution of Avicel®

Size range (µm)	% frequency	classmark	fixi	%cum. Freq.
1-50	60	25.5	1530	60
51-100	20	75.5	1510	80
101-150	9	125.5	1129.5	89
151-200	5	175.5	877.5	94
201-250	5	225.5	1127.5	99
251-300	1	275.5	275.5	100
Total		**64500**		

X = mean particles size = $\frac{64500}{100}$= 64.5µm

Flow properties of SDMCC and Avicel®

The flow properties of pharmaceutical powders are must often adjudged by various parameters such as bulk densities, tapped densities, Carr's indices, as well as the follow rate, angle of repose, and Hausner ratio. Table 3 show the values obtained for sawdust microcrystalline cellulose as well as that for avicel®

Identification test for Cellulose: The test conducted on the extract is positive as the blue coloration observed indicated that the residue obtained after the extractive process is cellulose.

Chemical test for Sugar: The test resulted negative, since the extract fails to produce brick red coloration that may have indicated the present of sugar.

Chemical test for Starch: The result is negative since no colour change was observed, a canary yellow coloration will had indicate the presence of starch in the extract.

Chemical test for lignin: Lignin was also observed not be present in the extracted microcrystalline cellulose, this is a good indication of the success of the extractive process.

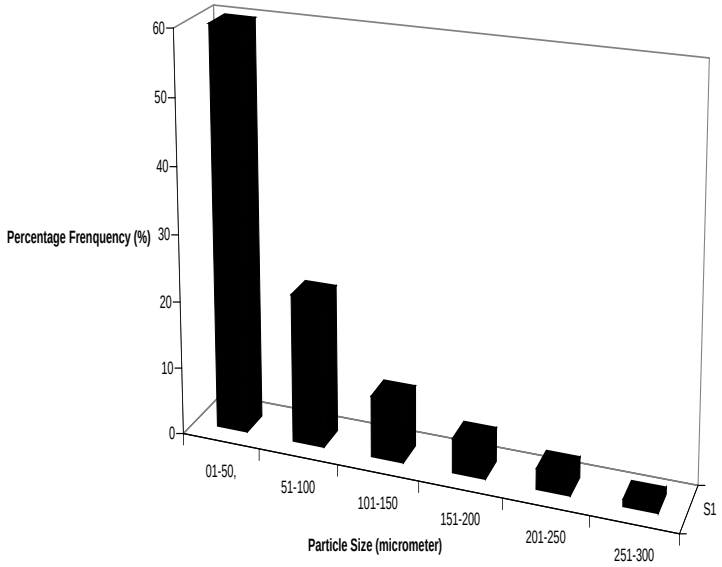

Fig. 1: Percentage Frequency vs Particle size of (SD) MCC

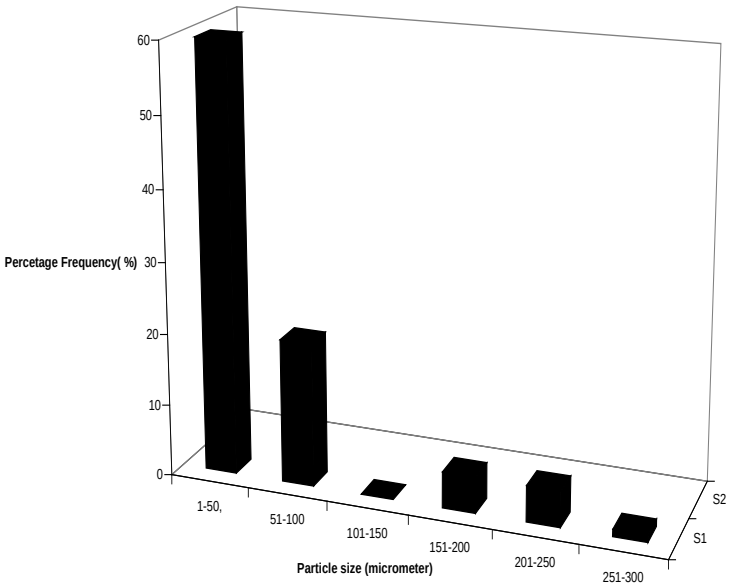

Fig. 2: Percentage Frequency vs Particle size of Avicel®

Table 3: Flow properties of SDMCC and Avicel®

Properties	SDMCC	Avicel
Bulk density	0.30	0.31
Tapped density	0.33	0.34
Carr's index	9.1	8.8
Flow rate g/s	1.00	1.02
Angle of repose (°)	26	25
Hausner ratio	1.29	1.22
Moisture content	2.0% w/w	2.0%

Table 4: Chemical and identification tests conducted on extracted sawdust microcrystalline cellulose

Test	Result
1 identification test for cellulose	(positive)
2 chemical test for the presence of sugar	(negative)
3 chemical test for the presence of starch	(negative)
4 chemical test for the presence of Lignin	(negative)

Total ash residue 67% w/w

The total ash left after the exposure of sawdust microcrystalline cellulose to temperature of 450 ^{o}C was 67% w/w and the elementary composition of the ash residue is shown in table 5

Table 5: Total Ash Elementary Content

Elements	Weight/1gr
K^{+1}	$2.0.\ x10^{-2}$
Na^{+1}	$6.3\ x\ 10^{-3}$
Fe^{+2}	$4.15\ x\ 10^{-4}$
Zn^{+2}	$9.0\ x\ 10^{-5}$
Ca^{+2}	$7.0\ x\ 10^{-5}$

DISCUSSION

Comparative Particle size analysis of SDMCC, and Avicel

The extracted sawdust MCC and Avicel shows similar particle size characteristics.

Generally both SDMCC and Avicel contain few longitudinal fibrous structures, with most of the particle (75%) have sizes lower than 100 µm the mean particle size of SDMCC was 61.5µm while that of Avicee was 64.5µm

Comparative flow Properties of SDMCC & Avicel

Both SDMCC and Avicel a commercial grade show similar flow properties

Three commonly employed methods of testing flow properties of pharmaceutical powders are;

- angle of repose
- compressibility index
- flow rate through an orifice

The angle of repose for SDMCC was 26^0 while that of Avicel was 25^0 pharmaceutical powders with angle of repose value between $25\text{-}39^0$ are consider excellent U.S pharmacopoeia 2000.

Also the compressibility index of SDMCC was 9.1% while that of Avicel was 8.8%.

Generally, the pharmaceutical powder with compressibility index of less than 10% is considered excellent.

Both powders process excellence compressibility and flow properties that is necessary for excellent tablet compression, thus these are ideal excipients that can be use in direct compression tablet production.

Chemical properties

All the chemical tests carried out on the sawdust MCC shows the presence of cellulose and absence of other cells contents such as lignin, sugar and starch, which if present will constitute impurities in the extract.

The extractive process is simple and inexpensive, meaning that the many unemployed youths can be train on how to produce MCC from sawdust, thus supporting the poverty alleviation programme of central government of Nigeria and other developing nations.

In developing countries like Nigeria, local production of MCC should be encourage from this agricultural waste product such as sawdust. The extraction of MCC from sawdust will generate huge foreign exchange for the country, as MCC is in high demand by both pharmaceutical and food companies manufacturing industries.

The observed close physical and chemical similarities of both sawdust microcrystalline cellulose and Avicel commercial grade microcrystalline cellulose is a good indication to both pharmaceutical and food manufacturing industries that sawdust microcrystalline cellulose can be a good substitute of Avicel in all pharmaceutical and food formulations.

CONCLUSION

Sawdust MCC, posses, both physical and chemical properties that are similar to a commercial available microcrystalline cellulose (Avicel). Both Sawdust MCC and avicel flow properties are excellent thus both can be interchangeable be use as direct compression excipients. The extraction and purification process is simple and inexpensive

REFERENCES

1. A. Payen (1838) “Memoire surla composition da tissue proper des plantes et du Ligneux” memoir on the composition of the tissue of plans and woody material, comptes rendus, vol 7 pags 1052-1056.
2. Crawford, R.L (1969) Lignin biodegradation and transformation Newyork. John Wiley & Sons ISBN 0-471-05743-6
3. Drydan, C and Reid, J.D, (1940) Textile colorist, 62,43
4. Okhamafe, A.O Igbobechi, A.C and Obasek, T.O (1991) Cellulose extracted from groundnut preliminary physico chemical characterization. Pharmacy world Journal 8 (4) 120-123.
5. Slavin, J.L Braver, P.M. and Marlett, J.A., (1981). Neutral detergent fiber, hemcellulose and cellulose digestibility in human subjects. The Journal of Nutrition 111 (2); 287-97 PMID 6257867.
6. Young, Raymond (1986) cellulose structure modification and hydrolysis New York; Wiley ISBN 0471827614
7. Cross, C.F, Bevan E.g cellulose, and outline of the chemistry of the structural elements of plants Longmans, Green & Company, London.
8. Necessitates in: Remington – The science & Practice of pharmacy, Editors Gennaro, A.R el, al, Lippincott, Williams & Wilkins U.S.A Pp 1042.
9. Reily (Jr) W.J. (2000) Microcrystalline cellulose, Ch 55 Pharmaceutical
10. Sun, X.F XU, F, Sun, R.C Fowler, P and Baird, M.S Characteristic of degraded cellulose obtained from steam – exploded wheat straw. Carbohydrate Res. 2005, 340:97-106
11. United State Pharmacopoiea 2010 vol. i,ii,iii
12. Ohwonuworhua, F.O., Kunle, O.O. and Ofoefule, S.I.(2004) Extraction and Characterization of microcrystalline cellulose derived from Luffa Cyludrica plant. Afr.J.Pharm.Res.Develop. 1 (1) 1-6.
13. The international Pharmacopeia (1979) Vol 1, 3rd ed. General methods of analysis. WHO publication p161