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COMPARATIVE INVESTIGATIONS ON LOCALLY PROCESSED ETHYL CELLULOSE AND ETHOCEL®

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Abstract: **Sawdust is an industrial waste by-product obtained in the process of smoothing wooden planks. It is readily available and at low prices. Alpha cellulose was extracted from saw dust powder using a standard extraction method. Thereafter the alpha cellulose was subjected to the process of ethylation using diethyl sulphate at 80°C for 24 hrs. The derived ethylcellulose was evaluated for its physicochemical properties such as particle size, bulk density, flow rate, Carr`s index, etc. Results indicate that particle size, flow rate, etc. were adequate for its use as a pharmaceutical excipient. These physicochemical characteristics were found to be comparable to that of a commercial excipient, Ethocel®. This study has established the possibility of obtaining ethylcellulose, a pharmaceutical excipient from sawdust, an industrial waste product.**

Keywords: **Cellulose, Ethylcellulose, flow properties.**

1. INTRODUCTION

Cellulose is the backbone structure of plants, and it is the chief constituent of the plant cell walls. Cellulose is the most abundant and widely used organic material in the world, with a worldwide consumption that is higher than steel, coal, or sugar (Ott e*t al*, 1954). Three basic types of cellulose are available i.e. alpha, beta, and gamma celluloses. They differ in their solubility behaviour in 17.5% w/v of sodium hydroxide (Cross, 1895).

While alpha cellulose is insoluble in 17.5% w/v NaOH solution, beta cellulose is soluble and can be precipitated out of solution by the addition of a mineral acid; gamma cellulose is soluble in 17.5% w/v of NaOH solution but cannot be precipitated out of solution. From alpha-cellulose, numerous derivatives of cellulose can be obtained (Okhamafe *et al*, 1992).

Ethyl cellulose is a derivative of alpha cellulose in which some of the hydroxyl groups on the repeating glucose units are converted into ethyl ether groups. It is an inert, free-flowing, white, hydrophobic powder. It is also a tasteless, odorless, noncaloric polymer used as a tablet binder and for film coating of tablets (Tyler *et al*, 1981). Because of its water retarding properties, it is used to control the release of active pharmaceutical ingredients in tablet production.

2. MATERIALS AND METHODS

Materials

Wood shaving was obtained from a local furniture company (Sokoto Furniture Ltd) within the Sokoto metropolis, Sokoto North Sokoto State Nigeria, Ethocel®, (BDH England), and all other reagents are analytical grades.

Method

Collection and drying of sawdust

One Kilogram of wood shaving was collected and dried at 60^{0}C for 24 hours in the Gallenkamp hot oven. This was later milled with the Alzico milling machine and sieved using a Nesty Endecott sieves arranged on a vibrator.

Extraction of alpha-cellulose from sawdust

400g fraction of milled wood shaving retained on a 250-micrometer sieve was treated with 17.5 w/v sodium hydroxide in a 12-litre stainless steel bowl immersed in a water bath maintained at 100 ^{0}C for 12 hrs. This was to remove all other plant cell constituents by solubilization in 17.5% w/v sodium hydroxide solution, the residue was alpha-cellulose that is not soluble in 17.5% w/v sodium hydroxide solution. This was then bleached with 100ml of 1:1 dilution of 3.5% w/v sodium hypochlorite solution, the bleaching was conducted at 80 ^{0}C for 8 hrs. This step was repeated until the material became milky white. The resulting alpha-cellulose was then treated with 50 ml of 20 % v/v hydrogen peroxide at 40 ^{0}C for 2 hrs. The cellulose was washed, filtered, pressed and dried at room temperature for 48 hrs.

Production of Ethyl Cellulose

Ethylation was done with 100g of alpha-cellulose extracted above by treating the same with excess diethyl sulfate in 10% w/v sodium hydroxide medium at an elevated temperature of 80 ^{0}C for 24 hrs. The ethylcellulose formed was thoroughly washed with 500 ml of methanol. The washing process was repeated until all impurities were washed off. The obtained ethylcellulose was filtered, and dried at room temperature (25 ^{0}C) for 24 hrs. The percentage yield of locally produced ethylcellulose (WSEC) was determined using the following equation.

$Y = 100 \times W_2/W_1$ (%) …. equation 1

While Y = percentage yield

W_2 = final weight of WSEC

W_1 = Initial weight of alpha cellulose

Identification test for ethyl cellulose

A 5.0 g quantity of WSEC was dissolved in 95 g of 80:20 w/w mixtures of toluene and ethanol. The resulting solution was heated for 10 min. The same procedure was repeated for Ethocel® (a commercial-grade ethylcellulose) which served as the reference material for comparison.

Particle Size Determination of ethylcellulose:

The particle size of ethylcellulose was determined using 0.02g of the material mounted in 10 ml of water and observed light microscope fitted with graticule (magnification×10). One hundred particles were selected at random and their diameters were measured by varying the field of view.

Sieve Analysis of Ethylcellulose: Using the sieve analysis method, a set of Endecott sieves was stacked in the following sieve mesh in descending order. 1000µm, 500µm, 300µm, 250µ, 180µm, 90µm, and collecting pan. A known weight, (300g) was placed on the top most sieve and the rest of sieves placed on the shaker which was vibrated for 30 min. The weight of ethylcellulose retained by each sieve was determined. The same Procedure was repeated for Ethocel® for comparison.

Flow Rate

Ten grams (10g) of the obtained ethyl cellulose was passed through the Erweka flowability tester. The time taken for the ten grams of the materials to flow through the tester was recorded. This procedure was repeated two more times and the average time was obtained. The same procedure was repeated for Ethocel® for comparison.

Bulk and tapped densities (BD & TD)

A 50g of the ethyl cellulose was transferred into a 100ml capacity measuring cylinder and the bulk volume was recorded. The measuring cylinder was tapped 59 times on the hard top of the laboratory table. The tapped volume was also noted. The procedure was repeated twice and the average was determined.

Bulk density (BD) = W/BV ………… equation 2

Tapped density (TD) = W/TV………. equation 3

Where W = Weight of ethylcellulose

BV= Bulk volume

TV =Tapped volume

The Carr's index which is the percentage difference between the tapped and bulk

densities were determined using the equation below:

CI=100 (1-BD) / TD (%) …...equation 4

Angle of Repose

The angle of repose was determined using a 20 g sample of the powder, which was poured into a plugged glass funnel with the tip 10 cm above the flat surface of the bench. The powder was allowed to flow freely through the orifice of the funnel to form a heap whose height and diameter were determined. The angle of repose was calculated using the equation below.

Tan ϴ = H/R ……………..equation 5

Determination of moisture loss

About 30g of the obtained ethylcellulose was placed in an evaporating dish of known weight. This was later transferred into the Gallenkamp hot air oven and dried at 105 ^{0}C until a constant weight was obtained. The experiment was repeated twice and the average weight was determined. The percentage moisture loss was computed using Equation 6

below:

MC = 100 (IW – FW) IW …. equation 6

where

MC = Moisture Content

IW = Initial weight

FW = Final weight of the sample

The same procedure was repeated for Ethocel® for comparison.

Test for Lignin

About 100mg of WSEC which was on a glass slide were added Conc. HCL and warmed gently until the liquid content dried off. Two to three drops of phloroglucinol were added and the glass slide was examined under a light microscope.

Test for Sugar

400mg of the ethylcellulose was dispersed in 4 ml of distilled water in a clean boiling test tube which was boiled for 5 min in a water bath and then allowed to cool thereafter. To one portion of the boiled sample, 5ml of equal volume of a mixture of Fehlings solutions A and B was added and boiled for a few min.

Test for the presence of starches

To some 50mg of WSEC placed in a test tube 5 ml of KOH 5 % w/v was added and then the resulting mixture was heated gently for a few min. This was observed for any colour change.

Reaction to litmus

A 5 % w/v aqueous suspension of SDEC was prepared and treated with blue litmus paper to determine the acidity.

Solubility test

One gram of WSEC was added to one litre of water. The degree of solubility was noted. The same procedure was carried out with one litre of chloroform as another solvent.

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3. RESULTS

The result of the physicochemical characteristics of sawdust wood shaving derived ethylcellulose (SDEC) and that of commercially available ethylcellulose, Ethocel® is shown in Table I.

Table 1: Physicochemical characteristics of SDEC and Ehocel®

SN	Properties	WSEC	Ethocel
1	Colour	Off white	Off white
2	Odour	Odorless	Odorless
3	Taste	Tasteless	Tasteless
	Identification test for the Presence of		
4	Reducing Sugar	Absent	Absent
5	Lignin	Absent	Absent
6	Starch	Absent	Absent
7	Aqueous Solubility	Insoluble	Insoluble
8	Solubility in chloroform	Soluble	Soluble
9	Reaction of Litmus	Neutral	Neutral
10	% Yield	88	NA

Table 2: Microscopic Particle Size determination of SDEC

Size Range (µm)	% frequency F_j	Class Mean X	$F_i x_i$	% Cum Freq
1-150	60	25.5	1530	60
51-100	27	75.5	2038.5	87
101-150	10	125.5	1255.0	97
151-200	1	175.5	175.5	98
210-250	1	225.5	225.5	99
251-300	1	275.5	275.5	100
301-350	0	325.5	0	100
	100		**5500**	

Mean particle size = $\sum F_i x_i / \sum F_i$ = 5500/100 = 55µm

Table 3: Microscopic Particle Size determination of Ethocel®

Size Range (µm)	% frequency F_j	Class Mean X_i	$F_i x_i$	% Cum Freq
1-150	65	25.5	1657.5	65
51-100	25	75.5	1887.5	90
101-150	6	125.5	753	96
151-200	1	175.5	175.5	97
210-250	1	225.5	225.5	98
251-300	1	275.5	275.5	99
301-350	0	325.5	325.5	100
	100		5300	

Mean particle size = ∑ Fixi / ∑Fi = 5300/100 = 53µm

Table 4: Comparative flow properties of SWEC and Ethocel ®

Flow Properties	WSEC	Ethocel
Bulk density (g/cm^3)	0.77	0.75
Tapped density (g/cm^3)	0.90	0.86
Carr's index (%)	14.44	15
Flow Rate (g/s)	1.22	0.99
Angle of repose (°)	22,01	25
Hausner ratio	1.17	1.16
Mean particle size (µm)	55.00	53.00
Moisture Content (%w/w)	3.00	2.95

4. DISCUSSION

The identification tests revealed the presence of ethylcellulose, and impurities such as reducing sugars, starch lignin, and other impurities were absent (Table 1). The product is neutral to litmus as shown; the product was insoluble in water but soluble in chloroform, suggesting high substitution of the ethoxy group. This present one way to to differentiate high-grade ethylcellulose (containing a high ethoxy group of about 46% or more) from Low-grade ethylcellulose which is water-soluble and lower degree of substitution, (Kumar et al,m 2014).

The solubility of ethylcellulose can be used to distinguish between low-grade ethylcellulose and high-grade ethylcellulose, as the low-grade ethylcellulose is readily soluble in water as against high-grade ethylcellulose. However, it is only the high-grade ethylcellulose that is suitable for controlled-release tablet formulation release formulations because of the water-retrading property (Reilly, 2000). The mean particle size of WSEC was 55µm while that of Ethocel ® was 53µm the two values were insignificantly differences ($P \leq 0.05$). The flow parameters of both Ethocel and WSEC are as in Table 4, which equally revealed similarities in their flow parameters such as the flow rate, angle of repose, and the carr's index.

5. CONCLUSION

The result of the preliminary physicochemical evaluation reveals the possibility of producing ethylcellulose locally from wood shaving an agricultural waste product for both pharmaceutical and food industries. This will reduce capital flight and provide gainful employment for the citizens

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