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Extraction and Processing of Pharmaceutical Grade Microcrystalline Cellulose from *Dracaena arborea* Stem

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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ABSTRACT

Cellulose is an abundant renewable biodegradable polymer regarded as a promising feedstock for chemical productions with its versatility evaluated as a useful structural and functional material for pharmaceutical and industrial applications. It is a straight chain polymer which appears in cell walls of most plant and consists of *D. glucose* units, with absence of coiling or branching and can be derived from variety of sources including: annual plants, microbes, animals etc. Three basic types of cellulose often exist in nature as - alpha (α), beta (β) and gamma (Y). Microcrystalline cellulose (MCC) sourced from cellulose, occurs as a purified and partially depolymerized alpha cellulose from plant parts such as *D. arborea* stem possibly by severe acid or alkaline hydrolysis. *D. arborea* plant is a palm – like tree of 1.5m height with several branches, often used as a boundary mark, a non-selective habitat and belongs to the family Asparagaceae, sub family Nolinoideae. With alkali hydrolysis of the *D. arborea* stem, the percentage yield of MCC from the alpha cellulose is 54.32%. Physicochemical analysis of the MCC reveals it to have a pH of 7.80 and physicotechnical analysis resulted in values as recommended in the official monogragh. Proximate principles of the extracted MCC, depicted percentage fiber content as 65.78% and low lipid and protein content as 0.6 and

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0.4% respectively. Elemental analysis shows the composition of sodium and iron as 41% and 35% respectively but absence of lead and other deleterious materials. FTIR analysis suggests the presence of carbonyl groups, 6- membered cyclic ring (aromatic structure) with ortho and meta - OH substitution and long aliphatic chains. The x-ray diffraction study gave a percentage crystalinity index of 6.02 at $2\theta = 22$ and $2\theta = 34$.

Keywords: D. arborea stem; microcrystalline cellulose; extraction; processing; excipient.

1. INTRODUCTION

Cellulose, the most abundant renewable and bio degradable polymer is a promising feed stock for the production of chemicals, serving as materials for applications in various industries and the versatility of cellulose has been re-evaluated as a useful structural and functional material for pharmaceutical and industrial applications [1].

Cellulose can be derived from variety of sources including woods, annual plants, microbes and animals. It serves as a structural material within the complex architectures of the plant cell walls with variation in contents and is a naturally occurring material that may contain bi-products leading to application problems and difficulties in the chemical modification reactions although in recent times, cellulose isolation and purification is yielding materials of high purity and viability [2].

Cellulose is derived from D-glucose units and is a straight chain polymer unlike starch, absence of coiling or branching occurs and the molecules adopts an extended and rather stiff rod like conformation aided by equatorial conformation of the glucose residues [3].

Cellulose is taste less, odorless and hygroscopic with contact angles 20 – 30° and it is insoluble in water and most organic solvents, chiral and biodegradable and can be broken down chemically into its glucose units by treating with concentrated acids or bases at high temperature [4].

Cellulose in the cell wall of most plants likely exists together with encrusting substances such as lignin, hemicellulose and pectin, which though might be removed by straining under pressure, use of sodium sulphite and sulphurous acid and by the Kraft's process [5].

Three basic types of cellulose are commonly available and includes:- alpha (α) beta (β) and gamma (Y) celluloses. They could differ in their solubility behaviors especially when in contact with 17.5% sodium hydroxide, the α- cellulose are insoluble, β cellulose is soluble and can be precipitated out of the solution by mineral acids while the Y is soluble but cannot be precipitated out of solution [6].

Cellulose is insoluble in aqueous solution and most common solvents. This poor solubility is attributed mainly to the strong inter molecular hydrogen bonding between the individual chains but in spite of the poor solubility characteristics, it is useful in a wide range of applications. The chemical modifications of the celluloses are preferred to improve process ability and to produce cellulosic derivatives that could be tailored for specific industrial and pharmaceutical applications.

Cellulosic materials could occur in various reproducible, recyclable, and biocompatible forms and are used in various biomedical applications such as blood purification membranes and the likes [7].

Microcrystalline cellulose (MCC) is a purified and partially depolymerized α- cellulose derived from plant parts otherwise termed as natural fibers. The MCC could occur as a white odorless, tasteless and crystalline powder composed of porous particles. It is often derived from special grades of purified α- wood cellulose by severe acid/alkaline hydrolysis to remove the amorphous cellulose portions yielding particles consisting of bundle like microcrystals and a better understanding of the chemical and physical properties of these natural fibers is necessary for processing natural fibers – reinforced composites such as cellulose [8].

Cellulose from the reinforced sources, appears as a semi solid crystalline polysaccharide which consists of D- glucopyranosyl unit linked together by β- (1-4) glycosidic bonds. It also consists of three OH groups at C-2 and C-3 positions of

Plate 1. *Dracaena arborea* (Willd.) link

secondary hydroxyl groups while that at the C-6 positions of primary hydroxyl group forms intra – and inter – molecular hydrogen bonds that could allow for the creation of highly ordered three dimensional crystal structures [9].

1.1 *Dracaena arborea* (Willd.) Link, Plant as a Cellulose Derivative

D. arborea is a palm – like tree of about 1.5m in height and 2.3m in girth. It has several branches with leaves aggregated near branched ends. It has an inflorescence and pendulous appearance shortly branched via individual flowers of creamy – white petals.

The plant, is planned as a boundary mark used as an ancient landmark. The stem is smooth with nodal points evident along its length. It belongs to the family Lilliaceae formerly Asparagaceae. It has a non- selective habitat but commonly found around humid environment, upland forests and near stream and thrives well in loamy soil [10].

The research was borne out of the desire to unravel the diverse possibilities to getting MCC, regarded as a primary raw material in most pharmaceutical and chemical industries from a new plant source rather than the synthetic sources.

The research also aims at extracting α- cellulose adopting the alkali hydrolysis method from a new plant source (*D.arborea* stem) with subsequent isolation of MCC, then characterization and screening of the MCC obtained for its suitability and application as excipient in the pharmaceutical industries.

2. MATERIALS USED

Sodium Hydroxide pellets (Avondale lab England), Sodium hypochlorite 3.5% (Hypo multipro Enterprises, Nigeria), n- hexane (Kermel, China), D.arborea stem (Choba Rivers state, Nigeria), Litmus paper (Lab tech D/2, England), Muffle furnace (Sheffield, England).

2.1 Methods

2.1.1 Extraction of microcrystalline cellulose (MCC)

The extraction of MCC, was carried out using the stem shoot of *D. arborea* plant after identification and authentication applying the alkaline hydrolysis method.

2.1.2 Isolation of α- cellulose and MCC by alkaline hydrolysis

2.2 Preliminary Treatment and Size Reduction

The stem bark was peeled, and exposed inner part of the stem was chopped into smaller bits and immersed in water to remove sand and other extraneous and particulate matters.

2.3 Initial Delignification Stage

A 1.606-kilogram quantity of the fiber, was macerated in 200 ml of 0.25 M sodium hydroxide solution contained in a glass container, and heated at 80°C for 4 hours to De- lignify the fibers.

The fibers present, was washed several times with distilled water until neutral to litmus paper and excess moisture squeezed through a muslin cloths.

2.4 Pre-Bleaching with Sodium Hypochlorites

The pre-lignified moist fibers was heated over a water bath at 80°C in 100 ml of an aqueous solution of sodium hypochlorite (1:1) for 1hour to effect some degree of bleaching. The bleached mass, was rinsed severally with distilled water and squeezed through a muslin cloth to remove excess moisture.

2.5 The Second Delignification Process

The bleached mass was again subjected to delignification process by heating over the water bath at 80°C for 1hr using 17.5% v/v sodium hydroxide solution, which was prepared by dissolution of about 170 g weighed quantity of sodium hydroxide in 1litre of water

2.6 Final Bleaching with Sodium Hypochlorite

Further bleaching of the formed moist mass was carried out (after washing with distilled water) by heating the material after immersing in a 1:1 sodium hypochlorite for 1 hr. The bleaching was done severally until a milky white material was obtained.

The resultant alpha - cellulose, was repeatedly washed with the distilled water until neutral to litmus paper. The excess moisture, was squeezed out with the muslin cloth and the material in the form of small lump dried in a hot air oven at 60°C for 2 hrs.

2.7 Final Treatment with an Acid to Obtain the MCC

A 213.0 grams quantity of the alpha cellulose obtained was placed in a flask and hydrolyzed with 1.5 M H_2S0_4, at a temperature of 105°C for 15 minutes.

The acid mixture was then poured into a container of cool distilled water and stirred vigorously. The MCC obtained was filtered through a number 1 Whatsman filter paper and washed continously with distilled water until it became neutral to litmus paper.

Thereafter, the obtained MCC crystals was dried in a hot air oven at 60°C for 60 minutes, then passed through, a 500 µm sieve and later 250 µm sieve to obtain an MCC of such crystal size. The yield of the MCC obtained was there after determined and recorded.

2.8 Determnation of Percentage Yield of Alpha (A) Cellulose

The α- cellulose was weighed and the yield was calculated using the equation

$$\%\text{Yield} = \frac{A}{B} \times 100$$

Where: A (g) = weight of alpha cellulose produced, B (g) = weight of initial fiber material

2.9 Determination of the Percentage Yield of Mcc from A- Cellulose

$$\text{Yield} = \frac{A}{B} \times 100$$

Where: A (g) = weight of microcrystalline cellulose produced, B (g) = weight of initial alpha cellulose

2.10 Physicochemical Properties of A-Cellulose

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

2.11 Test for Lignin

A 100 mg of MCC, was 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 evaporated. The slide was thereafter, examined under light microscope for any coloration.

2.12 Qualitative Determination of Cellulose

A 10 ml solution of iodinated zinc chloride was added to 10mg of MCC cellulose powder on a watch glass and observation made for possible color change.

2.12.1 Organoleptic test

The color, taste, and texture of the alpha cellulose produced were observed physically and by touch and the result recorded.

2.12.2 Solubilty test

One gram (1.0 g) of MCC was placed in a test tube and distilled water added. The test tube was shaken vigorously for 10 minutes and observed for extent of dispersibility of the micro crystalline cellulose

2.12.3 Swelling capacity

The swelling capacity which determines the extent of water retention of the MCC was determined adopting the process as outlined. The tapped volume (V_1) occupied by 5.0 g of the powdered alkali synthesized MCC, was noted. The powder was then dispersed in 85 ml of distilled water and made up to 100ml using same solvent then allowed to stand for 24 hours undisturbed. At the lapse of the time the volume of the sediment ($V_{2,}$) was estimated and the swelling capacity was computed using the relation:

$\frac{V2}{V1}$ Where: V_2 = Volume of sediments, V_1 = Tapped volume of 5.0 g of MCC powder

This test was also repeated for cornstarch powder.

2.12.4 Hydration capacity

This was determined to observe for extent of water absorption of the extracted MCC according to the method of Nguluka et al (2010). 1.0 g of the MCC powder (X) was placed in a centrifuge tube and covered with 10ml of distilled water. The tube was shaken intermittently for about 2 hrs and left to stand for 30 minutes before centrifuging at 3000 rpm for 10 minutes. The supernatant was decanted and the weight of the powder after water uptake and centrifugation (Y) was determined. Hydration capacity was calculated as:

$\frac{X}{Y}$ Where: X= Initial weight of microcrystalline cellulose before water update, Y= weight of MCC after water uptake. This was also done for cornstarch.

2.12.5 Moisture sorption capacity

The method of Oyeniyi et al (2012) was adopted with slight modification. Two grams (2.0 g) of the MCC powder (W), was weighed and put into a tarred petri dish. The sample was placed in a desiccator containing distilled water at room temperature and the weight gained (Wg) by the exposed sample at the end of the 5th day was recorded and the amount of water absorbed (Wa) was calculated from the weight differences as: Wa = Wg – W

2.13 Determination of Microcrystalline Cellulose True Density

The true density (Dt) of the cellulose powder was determined by the liquid displacement method using n-hexane as the immersion fluid and computed according to the following equations.

Dt = $\frac{Wp}{((a+Wp)b)} \times SG$

Where: Wp = the weight of powder. SG = the Specific gravity of solvent, a = the weight of bottle + solvent. b= is weight of bottle+ solvent + powder.

2.13.1 Porosity

The porosity (E), was calculated by the method as adopted by Ohovwurhua et al (2005) as:

E= $\frac{1-Bd}{Dt} \times 100$ Where: Bd = bulk density, Dt = true density of MCC cellulose

2.14 Physico Technical Properties of Extracted MCC

2.14.1 Bulk and tapped densities

A 20 g quantity (Wp) of MCC powder was gently poured through a short stemmed glass funnel into a 250 ml graduated cylinder. The volume occupied by the powder was taken as Vp. The powder was gently tapped on a wooden surface from a height of 7mm until no further change in volume was observed. This volume (VpT) was taken as the tapped volume. The bulk and tapped density were computed using the formula:

Bd = $\frac{Wp}{Vp}$ Td= $\frac{Wp}{VpT}$

Where: Bd is the bulk density, Td is the tapped density.

2.15 Carr's Compressibility Index and Hausner's Ratio

The Carr's index (CI) and Hausner's ratio (HR) were computed from the bulk and tapped densities as:

HR = $\frac{Td}{Bd}$ CI = $\frac{Td-BD}{Td}$ × 100

2.16 Angle of Repose and Bulkiness

Angle of repose was measured for the MCC and the method employed a funnel secured with its tip at a given height (h), 4 cm, above a plane paper placed on the horizontal flat surface. The powders were poured through the funnel until the apex of the conical pile touched the funnel. The diameters of the base of the powder heap was noted and the angle of repose (Θ) calculated using the formula

$(\Theta) = \text{Tan}^{-1}h/r$

r = radius of the powder heap base, h = height of the funnel tip from horizontal surface.

Bulkiness: This was calculated as the reciprocal of the bulk density.

2.17 Particle Size Analysis

This was determined using a sieve shaker, containing standard sieves arranged in a descending order according to their opening size. About 50 grams of the MCC powder was placed on the top sieve and shaken for 10 min. The weight of MCC retained on each sieve was determined and the average diameter calculated as reported by Ansel et al (2005) using the following equation:

Average diameter of the MCC particles = [∑ (% retained) × (mean aperture)]/100.

The results are presented in a graph of percent passing versus the sieve size. On the graph the sieve size scale is logarithmic. To find the percent of aggregate passing through each sieve, the percent retained in each sieve was first determined using the following equation,

$$\% \text{ retained} = \frac{\text{Amount retained (g)} \times 100}{\text{Weight of sample taken}}$$

The cumulative percent of aggregate retained in each sieve was calculated by adding up the total amount of aggregate retained in each sieve and the amount in the previous sieves. The cumulative percent passing of the aggregate was determined by subtracting the percent retained from 100%.

% Cumulative Passing = 100% - %Cumulative Retained.

The values were plotted on a graph with cumulative percent passing on the y-axis and logarithmic sieve size on the x-axis. After the plot, coefficient of curvature (Cc) and the coefficient of uniformity (Cu) for the sol (particles) was determined from the graph.

$$\text{Cc} = \frac{(\text{D30})^2}{\text{D60 X D10}} \text{ and } \frac{\text{Cu} = \text{D60}}{\text{D10}}$$

Where from the graph

D_{60} = particle diameter at 60% finer, D_{30} = particle diameter at 30% finer and D_{10} = particle diameter at 10% finer.

2.18 Proximate Analysis

A quantitative analysis of the MCC powder was carried out which helped to estimate the approximate amounts of components in the material. This helped to determine the levels of purity of the powder by giving an estimate of the actual fiber content. The following analysis, were carried out for the alpha cellulose powder.

2.19 Protein Content Determination

This was determined adopting the Kjedalhi method involving digestion, distillation and titration stages.

The % Organic nitrogen = $\frac{Titre\ value \times 1.4 \times 100 \times 100}{1000 \times 20 \times 0.1}$

Where titer value = the volume of acid used in the titration

1.4 = Nitrogen equivalent to the normality of 0.1N HCl used in the titration.
100 = the total volume of digest dilution, 100 = percentage factor.
1000 = Conversion factor from gm to mg, 20 = integral volume of Digits analyzed or distilled, 0.1= the weight of sample in grams digested, 6.25 = % Nitrogen

2.20 Carbonhydrate Content Determination

This was determined using Cleg Anthrone method.

$$\%CHO \text{ as glucose} = \frac{25 \times Absorbance\ of\ sample}{Absorbance\ of\ standard\ glucose} \times 100$$

2.21 Moisture Content Determination

This was determined by application of the air oven method

$$\%Moisture = \frac{Weight\ of\ fresh\ sample - Weight\ of\ dry\ sample}{Weight\ of\ sample} \times 100$$

2.22 Lipid Content Determination

This was carried out by adopting the soxhlet extraction method

$$\%\ Lipid = \frac{Weight\ of\ flask\ and\ extract - Weight\ of\ empty\ flask}{Weight\ of\ sample\ extracted} \times 100$$

2.23 Ash Content Determination

The ash content was analyzed using the furnace method

$$\%\ ash = \frac{Weight\ of\ crucible + ash\ sample - weight\ of\ crucible}{Weight\ of\ sample} \times 100$$

2.24 Elemental Analysis

Sample was ashed in a muffle furnace at a temperature of 650°C for 3 hrs. The ash sample was dissolved in 10ml concentrated hydrochloric acid and was heated on an electro-chemical heater hotplate. The solution of the ash was diluted to 50 ml with distilled water. The solution was analyzed for metal ion by atomic absorption spectrophotometer based on their absorption wavelengths as 283.3 nm for lead (pb), 766 nm potassium (K), 213.8 nm zinc (Zn), 589 nm sodium (Na) and 248.3 nm iron (fe).

Functional groups detection and possible interaction studies

This study was enabled using the Fourier transform infrared spectroscopy (FTIR) where the surface of each sample was characterized using perk in – elner spectrum 1000 fourier transform infrared (FTIR) spectrophotometer. Each sample was examined four times (4x) at a resolution of 4 cm – 1 between 4000 and 650 cm – 1.

2.25 X-Ray Diffraction Assay

2.25.1 X-ray diffraction (XRD)

The diffraction patterns were obtained using a high resolution Rigaku X-ray diffractometer with a scanning rate of 2°/min. The diffraction patterns were recorded using Cu-Kα radiation (wavelength 1.541 Å) operating with Ni filter from 0° to 80° of 2-theta (scanning angle) at an accelerating 340 Progress in Environmental Protection and Processing of Resource voltage of 40 kV and the current of 25 mA. The diffraction patterns were intended to identify structural changes in crystalline and amorphous phases. The sample were pressed into pellets (25 mm in diameter) by compression of 0.25 g in a mold under pressure of 50 Mpa. The degree of crystallinity could be relatively expressed by the crystallinity index (CrI). The value of CrI was calculated from the diffraction patterns based on the following equation [11].

$$\mathrm{CrI} = \frac{[(\mathrm{I002} - \mathrm{Iam})]}{\mathrm{CI002}} \mathrm{x}\ 100$$

Where, I002 is the intensity of the peak representing the crystalline part (at about 2θ=22°) and Iam is the intensity corresponds to the peak of the amorphous phase (at about 2θ=33°).

3. RESULTS

3.1 Percentage Yield

3.1.1 Yield of alpha cellulose (alkaline hydrolyzed)

Weight of initial fiber materials =1606 g, Weight of obtained alpha cellulose=213g

Percentage yield of α-cellulose =13.26%

3.2 Determination of Percentage Yield of MCC

3.2.1 MCC Yield for alkaline hydrolyzed

Weight of MCC obtained =115.7 g, Weight of Obtained Alpha cellulose=213 g

Percentage yield of MCC=54.32%

3.2.2 Qualitative determination of cellulose/confirmatory test

A violet – blue coloration was observed.

3.2.3 Organoleptic test

The MCC, obtained was brownish yellow in color, tasteless, fine and smooth

Plate 2. The pictures of *D. arborea* Fibers After further size reduction

Plate 3. Isolated alpha celulose from *D. arborea* plant

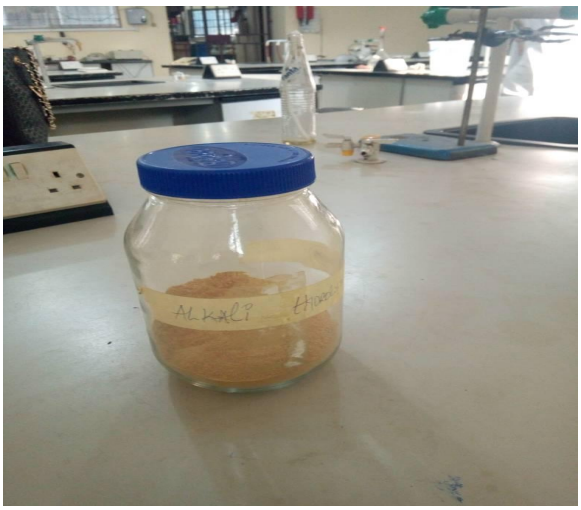


Plate 4. Picture showing the MCC obtained using Alkaline hydrolysis method

Table 1. Physicochemical properties of the extracted MCC powder

Parameters	MCC powder
pH	7.80
Bulk density (g/ml)	0.33±0.01
Tapped Density (g/ml)	0.46 ±0.12
Swelling Capacity (ml)	1.143±1.22
Hydration Capacity(gm)	3.60±0.85
Moisture sorption capacity(gm)	0.10
Carr' index (%)	27.73±0.02
Hausner's quotient	1.38±0.013.
Angle of repose (Ɵ)	(28.37±0.01)^{Ɵ}

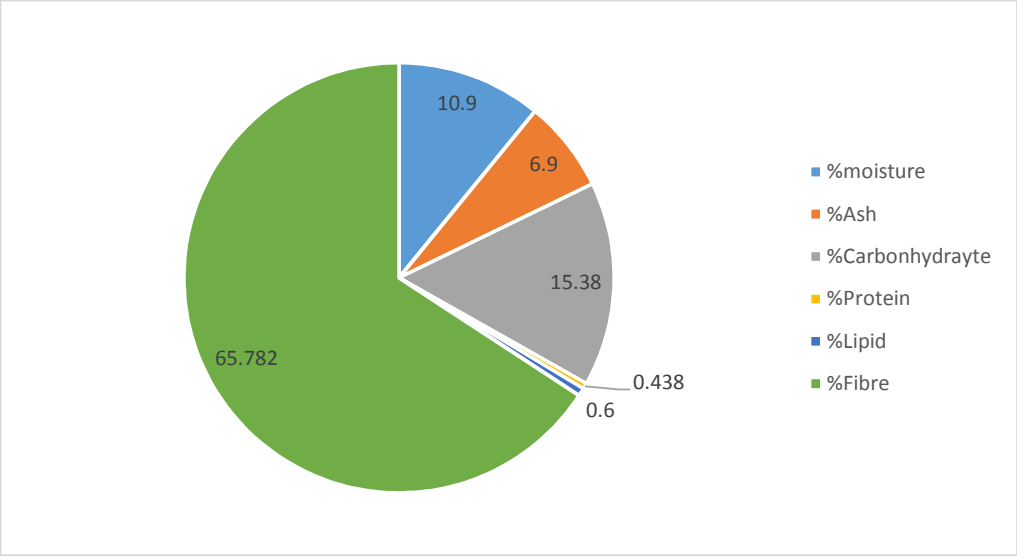


Fig. 1. Proximate analysis of MCC

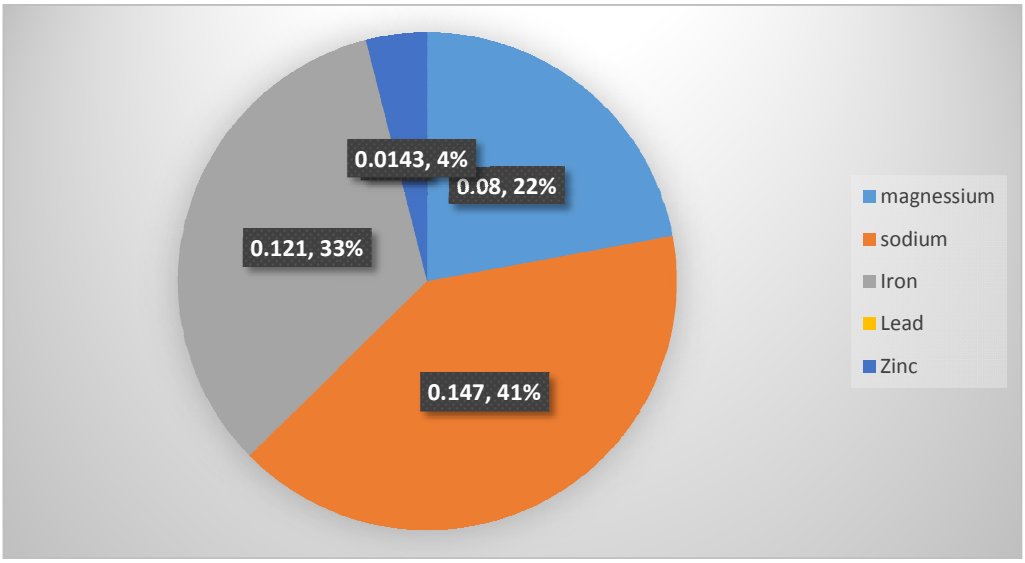


Fig. 2. Elemental analysis of MCC

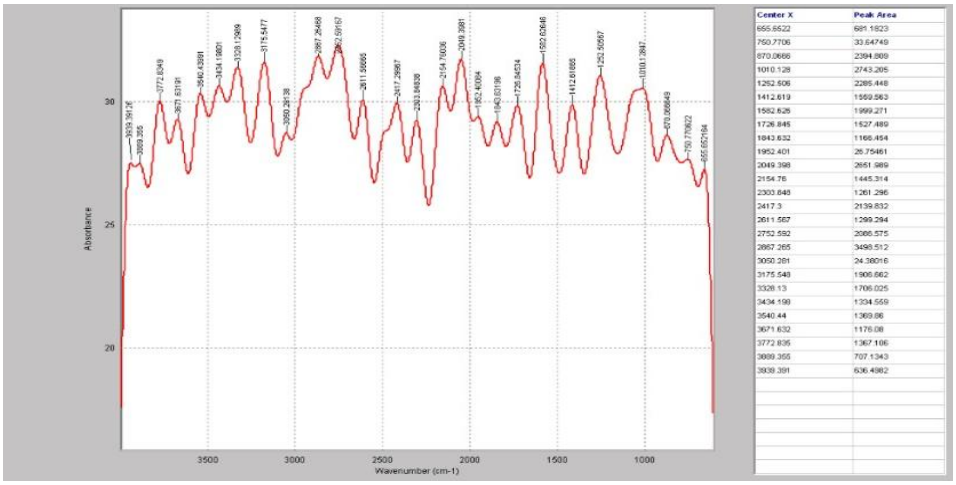
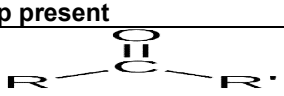
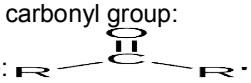
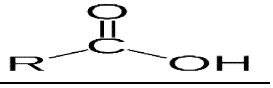
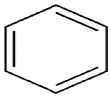
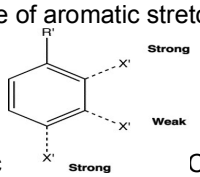
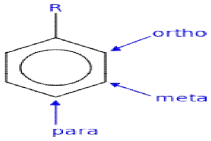
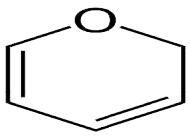


Fig. 3. Ftir assay conducted of the mcc isolate

Table 2. Functional groups detection of mcc obtained from *dracaena aborea link* from ftir analysis

Funtional group present	Wave-Length (Cm^{-2})
Carbonyl group: 	1650 - 1850
Simple carbonyl group:	1728
Ketone: 	(Intermediate between 1700 – 1750)
Aldehyde: 	
Presence of aromatic stretch: 	1412 - 1582
Aromatic C-H stretch: 	3000 - 3175
Ortho- substitution of aromatic ring: 	750
6-membered cyclic ring: 	1726
Long aliphatic chains:	2860 – 3050
(CH2 –CH2)n	1412
	750
Multiple Group (OH) stretch	3200 - 3600
Meta – OH group	3300 – 3700 strong and sharp band

3.3 Particle Size Analysis

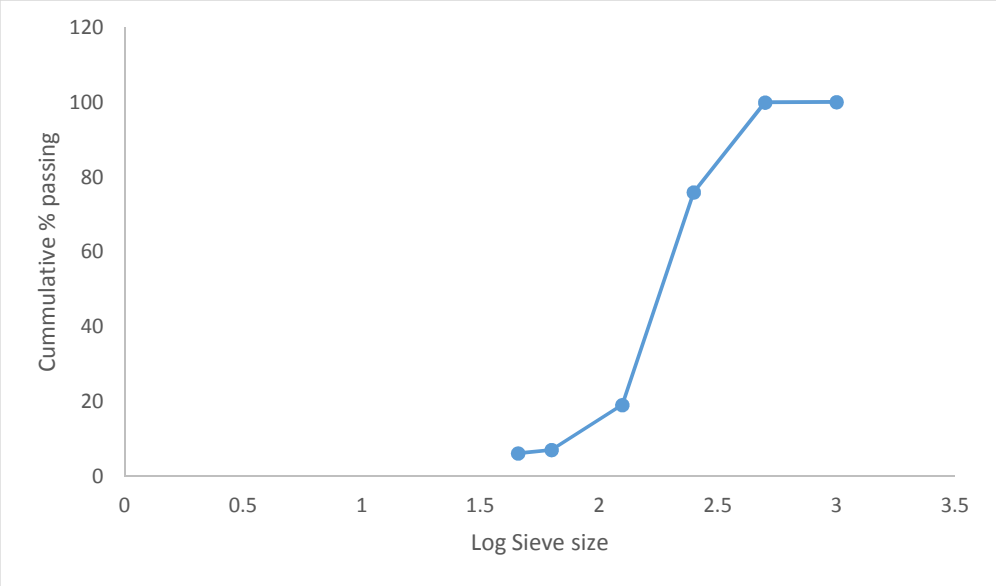


Fig. 4. Plot of cummulative % passing against log sieve size for the alkali extracted mcc from *d. arborea* stem

From the graph plotted and the calculation made, the coefficient of curvature (Cc) = 14.79µm and 21.37µm.

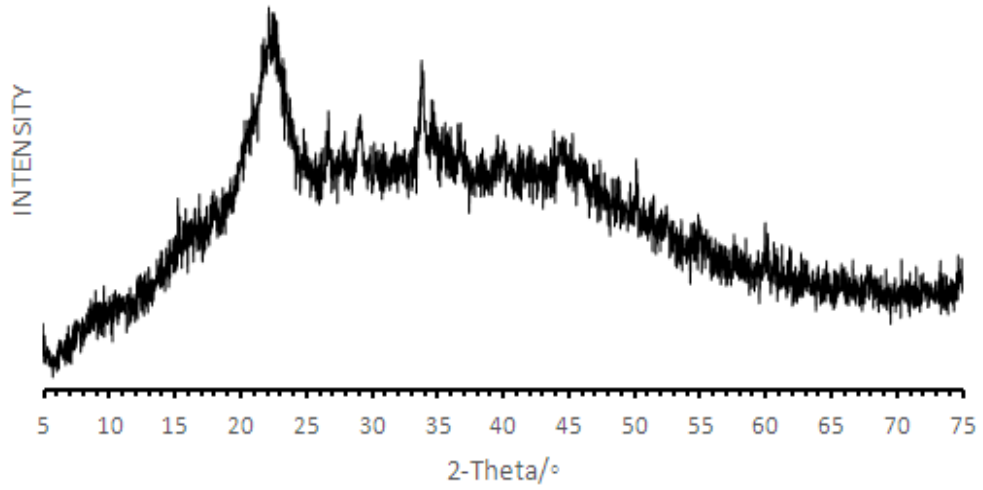


Fig. 5. X-ray diffraction of the extracted mcc from *d. arborea* stem

Percentage (%) Crystallinity index calculated = 6.02

4. DISCUSSION

From the study, the average yield of the alkaline hydrolyzed microcrystalline cellulose from the *Dracaena arborea* plant was 54.32%, this shows that the method adopted is very suitable in the extraction of microcrystalline cellulose from the *D.arborea* plant stem and the plant stem can be a prospective economic source of micro crystalline cellulose.

The organoleptic properties of the extracted MCC obtained from *D. arborea* plant shows a good property such as color, taste and texture which all conformed to the BP specification for microcrystalline cellulose, thus the organoleptic

properties of the MCC as obtained could be regarded as appropriate for usage in pharmaceutical oral fomulations.

The FTIR analysis is used to identify the various functional groups present in the sample. From the result obtained and the interpreted spectra, it shows a peak at (1650 – 1850 cm-^{2}), indicating the presence of a carbonyl group and peak at a point intermediate between (1700 -1750 cm^{-2}) showing presence of an aldehyde or ketone group. The peak at (3000 – 3175 cm^{-2}) shows the presence of an aromatic C-H stretch while that at 750 cm^{-2} shows the presence of an ortho-substitution of aromatic ring and that at 1726cm^{-2} shows the presence of 6- membered ring. The peaks at 2860 – 3050 cm-2, 1412 cm-2, confirmed the presence of long aliphatic chain, .that at 3200 – 3600 cm^{-2} depicts an -OH group stretch, while a strong and a sharp peak at 3300 – 3700 cm^{-2} shows the presence of meta- OH group. The various peaks however, points at the basic functional group moiety present in the cellulose structure and hence confirms the extract as a cellulose material.

For the solubility profile of the extracted MCC conducted, it was found to be insoluble in water and most other organic solvent such as methanol and chloroform. However, the solubility improved with the use of ionic solvent such as sodium hydroxide (NaOH) and potassium hydroxide (KOH). This conforms to the basic characteristics of microcrystalline cellulose, known to be insoluble in water and most common organic solvents but with improved solubility in an ionic solvent.

For the basic characterization of the MCC extract, it was observed that the bulk density of the alkaline hydrolyzed MCC was 0.33±0.01 g/ml and the tapped density 0.46±0.12 g/ml. The bulk density of a powder depends primarily on the particle-size, shape and the tendency of the particles to adhere to one another. The particles of MCC, may pack in such a way as to leave large gaps (pores) between their surfaces, resulting in a light powder or powder of low bulk density.

Angle of repose is a characteristic property relatively associated to inter particulate friction or resistance to movement between particles. The angle of repose is not an intrinsic property of the powder rather it could be much dependent upon the method used to form the powder cone. The value of angle of repose obtained for alkaline hydrolyzed MCC was 28.20°and the flow rate 0.68 g/sec, after adopting the Cone method for the determination. This obtained angle of repose for the sample analyzed indicates an excellent flow property reference to the British pharmacopoeia (BP) standard.

The compressibility index and Hausner's ratio are determined by measuring both the bulk and tapped volume of a powder. The Hausner's ratio is indicative of inter particulate friction, while the Carr's index shows the aptitude of a material to diminish in volume and become compressible. As the value of these indices increased, their flow ability decreases and from the data analyzed, the alkali hydrolyzed MCC had a fair flow.

In general, however, Hausners ratio greater than 1.25 indicates poor flow, and Carr's compressibility index below 16% indicates good flowability while values above 35% indicate cohesiveness. From the result the extracted MCC, has Hausner's ratio of 1.35 and Carr's compressibility index of 20%. With this index, it can be adjudged that the powders are poorly flowable, however, the powders are not too cohesive since the Carr's index is below upper limit [12].

Swelling is widely accepted as an indication of tablet disintegration ability and can be assessed by the determination of hydration capacity, swelling capacity and moisture sorption profile. From the results, the swelling capacity for extracted MCC, is 1.143 cm, hydration capacity 3.60 g and moisture sorption capacity is 0.10 g. The theory of disintegration is guarded by the fact that penetration of water (or any liquid) must precede disintegration, hence with result obtained, the swelling capacity of the microcrystalline cellulose is assumed to be appropriate and within acceptable limit for its possible use as a pharmaceutical excipient.

The moisture sorption capacity of a material measures the sensitivity of the material to moisture. It has been reported that the crystalline portion of cellulose does not absorb water and that the extent of water adsorption by cellulose should thus be proportional to the amount of amorphous cellulose present.

From the result obtained, it was observed that the moisture sorption capacity of the powder is 0.10 g and this could be attributable to the amount of amorphous cellulose likely present in

the cellulose fibrils. Aside from this, the study of water sorption is also of importance as it often reflects on the relative physical stability of powders when stored under humid conditions. Basically the moisture sorption capacity of the cellulose powder showed that the powders are sensitive to atmospheric moisture and should therefore be stored in an airtight container [13].

Bulkiness or bulk is an important consideration in the packaging of powders and it is known to increase with decrease in particle size. The bulk of alkaline extracted cellulose powder was calculated as 44.55 cm. However, the bulk densities of the powder is 0.33 g/ml, this shows that it will require large volume container to accommodate small weight of powder.

Carmella (1991), pointed out that a direct correlation exists between the degree of crystallinity of cellulose and its true density when determined in a non-polar liquid. Consequently, the true density values for the cellulose material suggests that the powder might have a slightly elevated degree of crystalinity with true density as calculated giving a value of 0.183 although in this case the correlation was not well achieved based on outcome of x-ray analysis implicating large amount of amorphous powder following the alkaline hydrolysis process.

From the study, it was observed that alkaline extracted MCC, had a pH of 7.405, which is within the neutral pH range hence the extracted MCC from *D. arborea* stem could be compatible as admixture for various systemic pharmaceutical formulations without imparting any incomptibility effect.

In the proximate analysis carried out, it was observed that the percentage moisture content of the MCC is 10.90%. This could be attributable to presence of numerous large pores, which could entrap moisture and lead to increased moisture absorption. However, the moisture content of this sample is above the official limits 8% stated in British pharmacopoeia and this possibly could be as a result of the high amorphous content of the powder.

Cellulose is known to be the chief constituents of plants cell wall and enclose varying amount of other substances as proteins, lipids and carbohydrate, hence presence of these substances within the cell. The analysis reveals that average carbohydrate content of the MCC is 15. 38%, the protein content 0.438% and the lipid 0.60%. These proximate parameters are chiefly employed in the food industry to quantify the nutritional values of a food substance hence the low level of protein and lipid will enhance the formulation of a stable pharmaceutical product slightly free from microbial influence. The outcome of this analysis has also paved way for further research on the profiling of the phytochemical constituents of the stem of *D. arborea* plant. Cellulose is known to be high in fiber content, thus the high percentage fiber content of the sample (68.78%) is an indication of large presence of MCC from the stem of *D. arborea* plant which could be of a great relevance in the Pharmaceutical, industrial and manufacturing sectors.

The elemental analysis result reveals cellulose constituent as having no lead or other deleterious material and this confirms the plant cellulose extract as safe for use in the human physiological system. However, the presence of magnesium, iron, sodium and zinc in considerable amount verifies the plant cellulose as useful in enhancing the metabolic and other physiological functions of living organisms if incorporated as excipients in internally prospected products. The x-ray results reveals the alkali extracted MCC as scantly crystalline but predominantly amorphous as the percentage crystallinity index was estimated at 6.02 at 2θ= 22 and 2θ= 34. Although defined peaks and detailed interpretation of the resulting graph is lacking due to shortfall in analytical tools useful for such purpose. Sequel to the observation about the low percent crystalline index of the MCC, water penetration, and moisture absorbability might increase as decreased crystallinity tends to increase hydrolysis [14]. Therefore controlling the crystal state is most important for controlling resultant product qualities.

5. CONCLUSION

From the study, *D. arborea* stem is verified to have high yield of microcrystalline cellulose though further step in the extraction process (alkaline/acid treatment) should be encouraged while adopting the alkali hydrolysis method to obtain high percentage crystallinity index of the MCC.

Adequate and exhaustive extraction procedure along with proper treatment of the plant cellulose will also contribute immensely to quality achievement and suitability of the extracted MCC from natural source (*D. arborea* plant stem), as a

pharmaceutical excipient especially following its consideration as a binder or disintegrant comparable to synthetic materials like avicel. Adherence and improvement on the quality development procedure of the MCC will further assist in boosting the economy of the developing countries, as there will be a resultant reduction in the much dependence on imported synthetic microcrystalline cellulose like avicel and allied materials.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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