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Comparative Physico-Chemical Properties of Microcrystalline Cellulose (MCC) from *Dracaena arborea* Stems Processed by Acid and Alkali Hydrolysis

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Abstract. Cellulose, an abundant renewable biodegradable polymer, is often recognized as a potential feedstock for chemical productions with its versatility being extended as a useful structural and functional material for pharmaceutical and industrial applications. It is a straight chain polymer constituent in cell walls of most plants consisting of D-glucose units devoid of coiling or branching. Three basic types of cellulose existing in nature are alpha (α), beta (β), and gamma (ϒ). Microcrystalline cellulose (MCC), which occurs in the form of purified and partially depolymerized α-cellulose from plants such as *D. arborea* stem, was derived by severe alkaline and acid hydrolysis. The MCC derived was of the percentage yield, 54.3 and 61.05%, and pH of 7.80 and 6.80 respectively, from the two hydrolytic pathways. Physico-technical analysis resulted in values similar to those recommended in the official monograph. Proximate principles of the extracted MCC depicted similar percentage fiber, lipid and protein content, as 65.78%, 0.6 and 0.4% respectively. Elemental analysis also showed similar composition of sodium and iron content as 41% and 35% respectively with absence of lead and other deleterious materials. FTIR analysis suggested the presence of carbonyl groups, 6- membered cyclic ring (aromatic structure) with ortho and meta - OH substitution and long aliphatic chains. Micromeritic analysis of the MCC gave average particle size of 112.46µm, coefficient of curvature (Cc) and coefficient of uniformity (Cu) as 1.14 and 1.23 for acid hydrolysed and average particle size of 358µm, Cc (1.003) and Cu (1.204) for alkaline hydrolysed. The x-ray diffraction study gave a percentage crystallinity index (CI) of 9.09 at 2θ = 22 and 2θ = 34 especially for alkaline hydrolysed although that of the acid hydrolysed was not determined but the percentage CI was suspected to be higher based on the particle size index.

Key words: *D. arborea* stem, hydrolysis, microcrystalline cellulose, extraction, processing

Introduction

Cellulose is the backbone of plants and the chief constituents of plant cell wall. It is insoluble in water and most common solvents and this poor solubility is attributed primarily to the strong intermolecular hydrogen bonding between the individual chains. Chemical modifications of cellulose are often performed to improve processability and to produce cellulose derivatives (cellulosic) which can be tailored towards specific industrial applications. Cellulose can be broken down chemically into glucose units by treating with concentrated acids (acid hydrolysis) and bases (alkaline hydrolysis) at high temperature (Lerouxel et al., 2006).

Cellulose is virtually the most abundant renewable and biodegradable polymer and is a promising feedstock for the production of chemical and for application in various industries.

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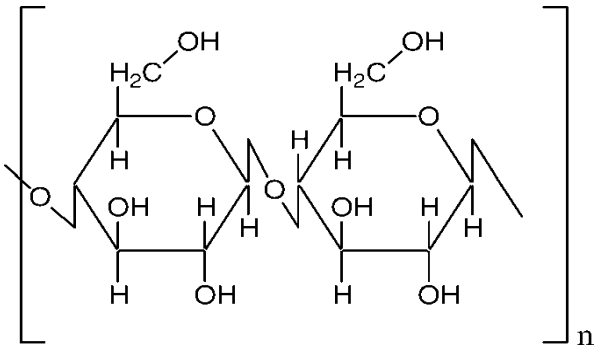
It can be derived from variety of sources such as woods, annual plants, microbes and animals (Carpita, 2011).

Microcrystalline cellulose (MCC) is a derivative of cellulose obtained by the chemical modifications of the cellulose present in both the gymnosperms of conifers, other soft and hard wood cotyledons although with considerable differences in terms of the chemical compositions or proportions of hemicellulose, fibers, mineral and acid contents (Lupidi et al., 2023).



Note: n = number of repeated crystalline cellulose long continuous chain

Figure 1: Molecular structure of cellulose

Several different crystalline structure of cellulose exists corresponding to the location of hydrogen bonds between and within strands. Cellulose produced by bacteria and algae is enriched in α-1 while cellulose of higher plants consists mainly of β-1.

Production and Use of Cellulose

The Kraft process is used to separate cellulose from lignin, another major components of plants matter. Other major constituents of cellulose include: paper, paperboard, card stock that are used for textiles made from cotton, linen, and other plants fibers. Cellulose can be converted into cellophane (a thin transparent film), and into rayon an important fiber that has been used for textile.

Cellulose is used to make water miscible adhesive and binders such as methyl cellulose and carboxy methylcellulose which are used in wall paper paste. Microcrystalline cellulose and powdered cellulose are useful as active fillers in tablets and as thickeners and stabilizers in processed foods, and also in the laboratory as the stationary phase for thin layer chromatography. Cellulose consists of crystalline and amorphous regions. By treating it with strong acid, the amorphous regions can be broken, thereby producing nano crystalline cellulose, a novel material with many desirable properties. Recently, nano crystalline cellulose was used as the filler phase in bio based polymer materials to produce nano composites with superior thermal and mechanical properties (Awa, Shinzawa, & Ozaki, 2015).

Assaying a Cellulose Containing Material

Given a cellulose-containing material, the carbohydrate portion that does not dissolve in a 17.5% solution of sodium hydroxide at 20ºC is alpha cellulose, which is true cellulose. Acidification of the extract precipitates β cellulose. The portion that dissolves in base but does not precipitate with acid is ϒ cellulose (Zhang, Hong, & Ye, 2009).

Chemistry of Microcrystalline Cellulose

The microcrystalline cellulose has a chemical formula of $(C_6H_{10}O_5)_n$. it is insoluble in water, ethanol, ether and dilute mineral acids and slightly soluble in sodium hydroxide solution. It has a pH range of 5.0-7.5 and its loss of weight should not be less than 8%**.**

Pharmaceutical microcrystalline cellulose has a good dilution potential and compressibility. It is a special form of cellulose consisting of Fibril by which individual crystal is held together by hydrogen bonding.

Uses of Microcrystalline Cellulose

Microcrystalline cellulose maybe used as an excipient in pharmaceutical granule/tablet formulation involving either dry compression or wet granulation technique as a secondary binder and can be used to granulate both soluble and insoluble powders. The granules compress quickly and produce tablets that generally do not harden with age. The fast wicking action of microcrystalline cellulose promotes rapid and even wetting of the powder mix. This is particularly useful in high moisture granulations as it binds with the excess moisture thereby keeping the granules dry and free flowing (Chaerunisaa, Sriwidodo, &Abdassah, 2019).

Microcrystalline cellulose is today widely used as pharmaceutical excipient acting as a disintegrant in various forms of tablet formulations. It speeds up tablets disintegration and provides the highest level of disintegration force at low concentration, utilizing dual disintegration mechanism of wicking and swelling for more rapid release of the active ingredients and despite the tablets hardness, it does not hinder the release of the active ingredient from the powder matrix.

Microcrystalline cellulose can also be used as a glidants and also as a binder where it can also be termed as substances that glue products together and cause them to form granules and also used as solutions or dry powders depending on other ingredients in the formulation and method of preparation. Microcrystalline cellulose is used as a wet binder and in wet granulation, it may be used as a secondary binder and can be used to granulate both soluble and insoluble powders. The fast wicking action of microcrystalline cellulose promotes rapid and even wetting of the powder. This is particularly useful in high moisture granulations as it binds the excess moisture keeping the granules dry and free flowing (Crouter & Briens, 2014).

***D. arborea* Stem**

Figure 2: Mature *D. arborea*

This plant has visible stem and belong to the family, Liliaceae (Asparagaceae), genus dracaena and common name is mart plant or boundary plant.

The plant has an inflorescence and pendulous appearance, shortly branched with individual flowers of creamy-white petals. It is planted as a boundary mark used as an ancient landmark. The stem is smooth with nodal points evident along its length. It has a non-selective habitat but commonly found around humid environment, upland forests and near streams and it thrives better in a loamy soil (Blick & Burns, 2009).

Materials

D. arborea stem (Choba, Port Harcourt), the chemicals used were of analytical grade and includes: Potassium dihydrogen sulphate, Sodium hydroxide pellets (Avondale lab. Limited. England), Sodium hypochlorite 3.5%w/v (Hypo Multipro Enterprises, Nigeria), n-hexane (Kermel, China), weighing balance (W and T Avery Limited, England), Bench centrifuge (Gallenkamp, England), drying (Oven) (New Life Medical, England), Litmus paper (Labtech, code D/2), Muffle furnace (Sheffield, England), Analytical balance (OHAUS, China), Desiccators, Pycnometer, Water bath (HH-1, HH-4, HH-6, HH-8 (Techmel and Techmel, USA). Set of sieves, Sieve shaker, X-ray diffraction machine.

Methods

Extraction of MCC, from the stem shoot of *D. arborea* plant was done following, acid and alkaline hydrolysis methods.

Alkaline Hydrolysis

Preliminary Treatment and Size Reduction

The stem bark was peeled and the inner part of the stem was cut into smaller bits and immersed in water to remove sand and other particulates matters.

Initial delignification stage

A 1.61 kilogram quantity of the resultant fiber was macerated in the 200ml of 0.25M sodium hydroxide solution in a conical flask and heated at 80^{o}C for 4hours to de lignify the fibers.

The fiber generated was then washed severally with distilled water until neutral to litmus paper and excess moisture squeezed through the muslin cloths.

Pre-bleaching with sodium hypochlorites

The pre-lignified moist fibers were heated over a water bath at 80^{o}C in 100ml 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 the muslin cloth to remove the water.

The second delignification process

The bleached mass was then subjected to a second delignification process by heating over the water bath at 80^{o}C for 1hr using 17.5% v/v sodium hydroxide solution. The 17.5% sodium hydroxide was prepared by dissolution of 170g weighed quantity of sodium hydroxide in 1litre of water

Final bleaching with sodium hypochlorite

Further bleaching of the formed moist mass was carried out (after washing with distilled water) by heating the material using a 1:1 ratio of the material to sodium hypochlorite for 1hr. This was done severally until the material became milky white indicating α-cellulose.

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 lumps finally dried in a hot air oven at a temperature of 60^{o}C for 2hrs.

Final treatment with an acid to obtain the MCC

A 213 grams' quantity of the alpha cellulose obtained was placed in a flask and hydrolysed with 1.5M H_2S0_4 at a boiling temperature of 105^{o}C for 15minutes.

The acid mixture was then poured into a container with distilled water and stirred vigorously. The MCC obtained was filtered through a number 1 Whatman filter paper and washed with distilled water until it became neutral to litmus paper (Ordu & Udenze, 2021).

After this, the MCC crystals obtained was dried in an oven at 60^{o}C for 1 hr. Further sieving was done by using a 250 µm sieve to obtain an MCC crystal of such size range. The yield was then calculated and recorded.

Acid Hydrolysis Method

An 81.0ml of concentrated sulphuric acid (H_2SO_4) was measured and transferred into a 1000ml measuring cylinder containing 700ml of a distilled water and made up to volume to form a 1.5M H_2SO_4.

The plant material about 800g was then weighed and placed on a heating glass container and the prepared solution of the acid carefully introduced into each of the containers. The mixture was heated to 100^{o}C for 2hrs then filtered using a fourfold muslin cloth and the remaining liquid carefully squeezed out.

The obtained lump was washed thoroughly using distilled water until neutral to litmus paper.

Pre-bleaching process

A 500ml of sodium hypochlorite solution mixed with equal volume of water in a measuring cylinder to obtain a 1:1 mixture and transferred into the container housing the plant extract. It was then heated up to 100^{o}C for 1hr, cooled, washed severally with distilled water until neutral to litmus paper.

Treatment with an alkali

The lump obtained was introduced into the heating container. A 20.5% of caustic soda prepared by dissolving 205g of sodium hydroxide in 1litre of water then transferred into the bottles and heated at 80^{o}C over a water bath for 2.30hr. The sample was cooled and washed thoroughly with distilled water until neutral to litmus paper.

Final bleaching with sodium hypochlorite

The recovered moist mass was bleached with 500L of sodium hypochlorite in equal volume of water for 30minutes. The resulting milky coloured sample was filtered, washed thoroughly with distilled water until neutral and then dried in an oven to obtain crystals

The crystals obtained were made to pass through a 500µm sieve aperture' and thereafter through 250µm sieve to obtain a 250µm microcrystalline cellulose (Adeleye et al., 2022). Then the percentage content of MCC obtained was calculated, and then sample characterized and result recorded.

Percentage Yield Determination of Alpha Cellulose Obtained Following Alkaline and Acid Hydrolysis Respectively

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

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

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

Phythochemical Assessment of MCC

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

Test for lignin

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

Qualitative determination of cellulose

A 10ml solution of iodinated zinc chloride was added to 10mg of MCC powder on a watch glass. The colour change was observed and noted.

Organoleptic test

The colour, taste, and texture of the MCC produced were observed physically and the result noted.

Solubility test

One gram (1g) of MCC was placed in a test tube, distilled water and other solvents added and the content shaken vigorously and observed for dispersibility. All the tests were carried out on both the alkaline and acid hydrolysed MCC.

MCC powder characterization

The physicochemical and physicotechnical properties of the microcrystalline cellulose isolated were comparatively evaluated.

Bulk and tapped densities

A 2.0g quantity (Wp) of MCC powder was gently poured through a short stemmed glass funnel into a 250ml graduated cylinder. The volume occupied by the powder was taken as Vp. The powder was 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:

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

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

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:

$$\text{HR} = \frac{Td}{Bd}$$

$$\text{CI} = \frac{Td - BD}{Td} \times 100$$

Swelling capacity

The swelling capacity of the MCC was determined by noting the tapped volume occupied by 5g of the powder (alkali and acid hydrolysed MCC) as Vx. The powder was then dispersed in 85ml of distilled water and the volume made up to 100ml with water. After 24 hours of standing, the volume of the sediment, Vv was estimated and the swelling capacity was computed (Chandra & Samsher, 2013).

$$\frac{Vv}{Vx}$$

Where: Vv = Volume of sediments, Vx = Tapped volume of MCC powder.

Hydration capacity

Here 1.0g of the MCC powder (Y) was placed in a centrifuge tube and dispersed with 10ml of distilled water. The tube was shaken intermittently for 2hrs and left to stand for 30 minutes before centrifuging at 3000rpn for 10minutes. The supernatant was decanted and the weight of the powder after water uptake and centrifugation (X) was determined (Tomer et al., 2001). Hydration capacity was calculated as:

$$\frac{X}{Y}$$

Where: X= weight of MCC before water update, Y= weight of MCC after water uptake.

Moisture sorption capacity

Two grams (2g) of the MCC powder (w) was weighed and put into a tarred petri dish. The sample were then placed in a desiccator containing distilled water at room temperature and the weight gained (Wg) by the exposed sample at the end of a five-days was recorded (Roja, Moren, & Lopez, 2011).

The amount of water absorbed (Wa) was calculated from the weight differences as:

$$Wa = Wg - W$$

Determination of MCC 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 a pycnometer. 0.5 g quantity of cellulose powder was placed in a dry pre weighed pycnometer and the rest filled with 50 ml n-hexane (SG 0.86) as the immersion fluid, the weight of the pycnometer filled with only liquid has previously been established and density of the powder was computed according to the following equation (Ohwoavworhua & Adelakun, 2005):

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

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.

Powder porosity (e)

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

$$E=\frac{1-Bd}{Dt} \times 100$$

Where: Bd = bulk density, Dt = true density of MCC cellulose.

Loss on drying

The powder sample 3 g was transferred into a Petri dish and then dried in an oven at $105^{O}C$ 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.

Angle of repose

Angle of repose was measured for both the alkali and acid hydrolyzed MCC. The method employed a funnel secured with its tip at a given height (h), 4cm, 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 diameter of the base of the powder heap was noted and the angle of repose calculated using the formula:

The angle of repose (θ) was calculated using the relation;

$$\theta = Tan^{-1}\frac{h}{r}$$

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

Bulkiness

This was calculated as the reciprocal of the bulk density.

Particle size analysis

This was determined using a sieve shaker, containing standard sieves arranged in a descending order according to their aperture 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 using the following equation (Hindi, 2017):

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

The results are presented in a graph of the sieve size versus cumulative percent passing. 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:

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

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 percent cumulative passing of the powder aggregate was determined by subtracting the percent retained from 100%:

$$\% \text{ Cumulative Passing} = 100\% - \% \text{ Cumulative Retained}$$

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

$$Cc = \frac{(D_{30})^2}{D_{60}xD_{10}} \quad \text{and} \quad Cu = \frac{D_{60}}{D_{10}}$$

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

Proximate analysis

A quantitative analysis of the MCC powder was determined to assist in making estimate of the approximate amounts of substances within the material including, determination of the levels of purity of the powder by giving an estimate of the actual fiber content from the α-cellulose powder of both hydrolytic processes (alkaline and acid hydrolysis)

Protein content determination: This was carried out using Kjeldahl method which involved such stages as: digestion, distillation and titration.

The distillate was titrated with standard 0.1 N Hydrochloric acid solution back to purple from greenish. The volume of hydrochloric acid added to effect this change was recorded as titer value (Horio, Yasuda, & Matsusaka, 2014):

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

Where: titer value = the volume of HCl used in titrating the ammonium distillate.

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 gram to milligram, 20 = integral volume of digits analysed or distilled, 0.1= the weight of sample in grams digested, 6.25 = % Nitrogen.

Carbonhydrate content: Cleg Anthrone method was adopted and here, glucose solution of 0.1ml was prepared and treated as the sample with anthrone reagent. Absorbance of the standard glucose was read and the value of carbohydrate in glucose was calculated using the formula below:

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

Moisture content: This involved the adoption of air- oven method where, 1g of the sample was weighed into a clean dried porcelain evaporating dish. This was placed in an oven maintained at a temperature of 105^{o}C for 6hours. The evaporating dish was cooled in a desiccator to room temperature and then reweighed.

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

Lipid content determination: This was by using soxhlet extraction method

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

Ash content: By use of the Furnace method, 1g of the dried sample was weighed into a porcelain crucible which was previously preheated and weighed. The crucible was inserted

into a muffle furnace and regulated to a temperature of 650^{o}C for 3hrs and allowed to cool to room temperature and re-weighed**.**

Calculation

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

Metal Analysis

Sample was ashed in a muffle furnace at a temperature of 650^{o}C for 3hrs. 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 50ml with distilled water. The solution was analysed for metal ion by atomic absorption spectrophotometer (AAS) based on their absorption wave lengths.

Lead ion: Lead ion was analysed at 283.3nm wave length. The concentration of lead ion in the sample was extrapolated from the standard graph of lead ion plotted. The concentration was expressed in mg/l or ppm and from the equipment corrections were made necessary in units of choice.

Potassium ion concentration: 766 wavelengths were selected.

Zinc ion: Sample preparation was as above. 213.8nm wavelength was selected. The sample solution was aspirated and the concentration of zinc ion in the sample was extrapolated and recorded in mg/l or ppm.

Sodium ion: 589nm wavelength was selected. The sample was aspirated into the instrument and its concentration obtained by extrapolation from the standard graph in mg/l or ppm.

Iron ion: 248.3 wavelength was selected. The sample was aspirated into the instrument and the concentration of Fe (ion) in the sample was obtained by extrapolation from the standard iron ion graph in ppm or mg/l.

Fourier-Transform Infra-Red Spectroscopy

The surface of each sample was characterized using Perkin-Elnerspectrum 1000 Fourier transform infra-red (FTIR) spectrophotometer. Each sample was Exposed 4 times at a resolution of 4cm-1 between 4000 and 650cm-1.

X- Ray Diffraction Assay of the MCC Obtained

Diffraction pattern were obtained using Philip X-ray diffractometer. The diffraction patterns were recorded using CU – kα radiation at 40 kV and 25Ma. The sample were pressed into pellets (25mm in diameter) by compression of 0.25g in a mold under pressure of 50Mpa. The crystalline index (CrL) was calculated according to formulae:

CrL = a/b signifying the ratio of peak intensity at 2Θ = 22 and 2Θ = 34 which are determined from the base line (valley).

Results

Table 1

***D. arborea* stem extract**	**Alkaline hydrolysis**	**Acid hydrolysis**
Weight of fibre (g)	161	161
Weight of α-cellulose (g)	213	284
Yield of α-cellulose (%)	13.26	17.68
Weight of MCC (g)	115.7	173.4
Yield of MCC (%)	54.32	61.05

Plate 1: Isolated alpha cellulose from *D. arborea* plant

Plate 2: MCC from Alkaline Hydrolysis Plate 3: MCC from Acid Hydrolysis

Table 2: Physicochemical characteristics of the obtained MCC

Property	**Alkaline hydrolysed MCC (A)**	**Acid hydrolysed MCC (B)**
Organoleptic	Brownish yellow, Tasteless, powdery and smooth	Brownish yellow, Tasteless, very powdery and smooth.
Identification	Violet-blue coloration	Violet-blue coloration
Organic impurities	No red colour with acidified phloroglucinol	No red colour with acidified phloroglucinol
Starch and Dextrin	No blue/ reddish brown ppt. with Iodine solution	No blue/ reddish brown ppt. with Iodine solution
pH	7.80	5.45
Water solubility	Insignificant	Insignificant

Table 3: Micromeritic properties of the MCC powder

Parameters	**A**	**B**
Bulk density (g/ml)	*0.33 (0.28)	*0.20 (0.02)
Tapped Density (g/ml)	*0.46 (0.01)	*0.29 (0.01)
True density (g/ml)	2.00 (0.21)	1.86 (0.23)
Porosity (%)	8.40 (0.32)	7.85 (0.16)
Carr's index (%)	20.20	31.03
Hausner's Ratio	1.35	1.45
Angle of repose (^{0})	28.20 (1.47)	43.53 (1.63)
Swelling Capacity (ml)	1.14 (0.20)	4.20 (0.18
Hydration Capacity (gm)	3.60 (0.06)	6.80 (0.04)
Moisture sorption capacity (%)	0.25 (0.18)	0.10 (0.05)
Loss on drying (%)	9.0 (0.08)	9.6 (0.12)
Average particle size (μm)	358	112.5

Note: *all initial value is the mean and std deviation is in parenthesis, number of replicate=3

Proximate Analysis

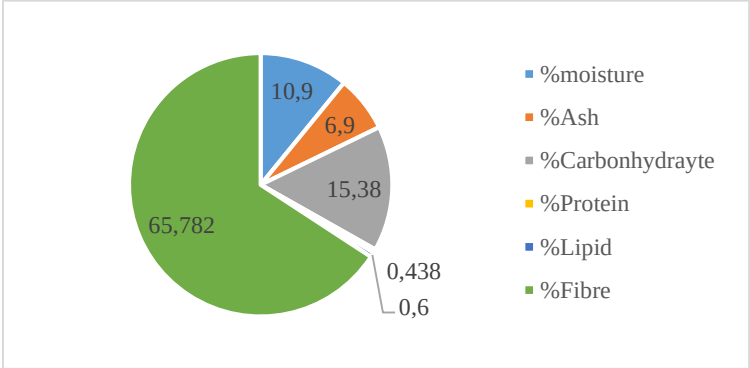


Figure 3: Proximate Analysis of Extracted *D. arborea* cellulose

Elemental Analysis

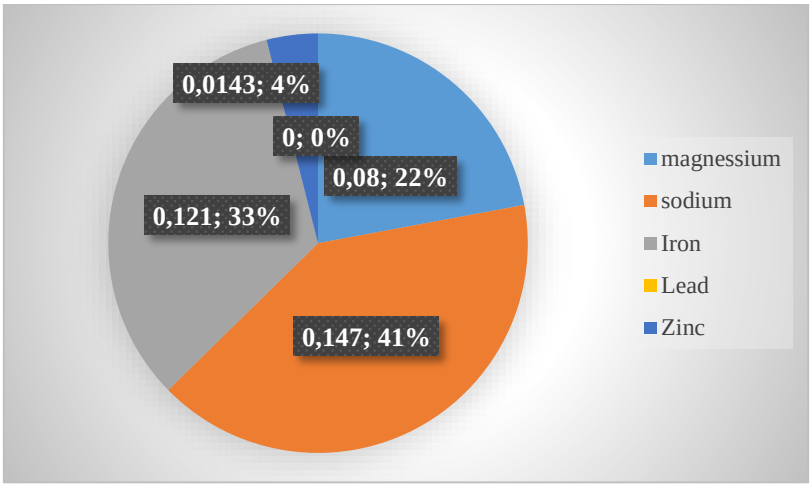


Figure 4: Elemental analysis of Cellulose from *D. arborea* stem

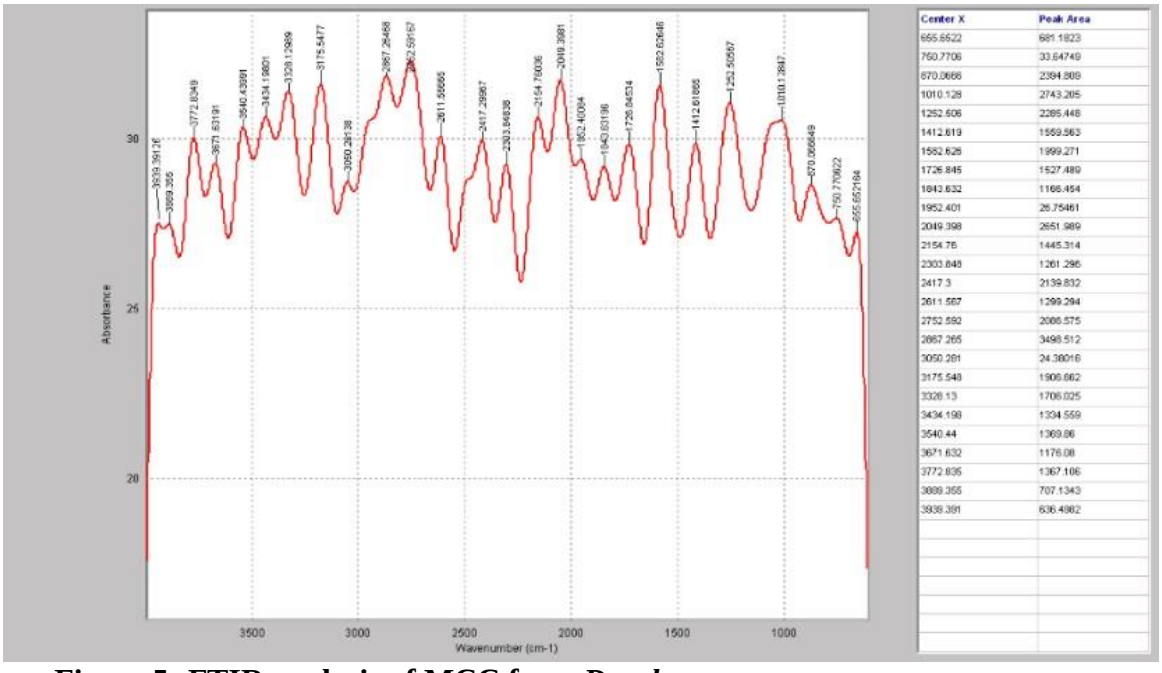


Figure 5: FTIR analysis of MCC from *D. arborea* stem

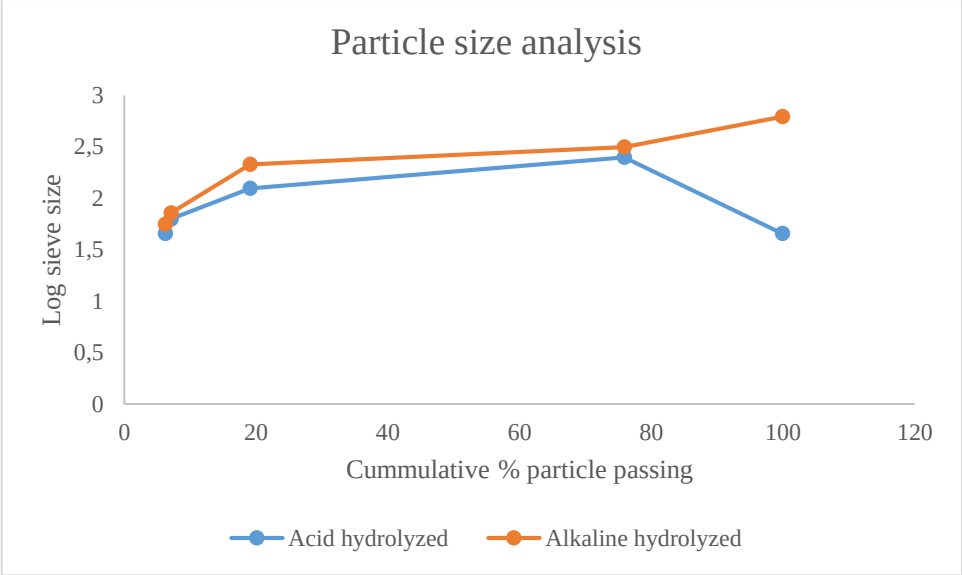


Figure 6: Plot of log sieve size against cumulative % particle passing

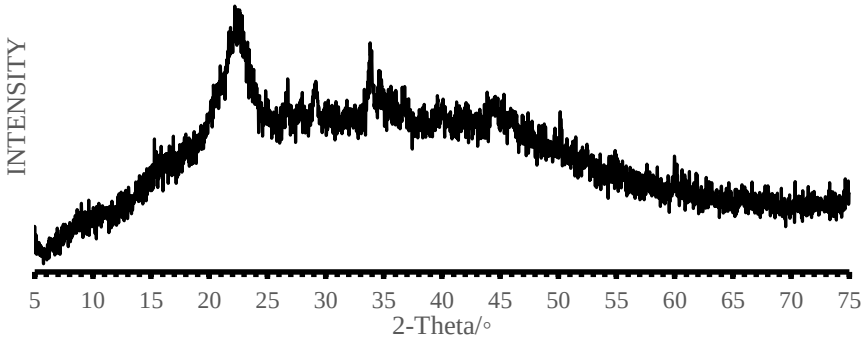


Figure 7: X-ray diffraction of MCC (alkaline hydrolysed)

Discussion

From the study, the average yield of the acid hydrolysed microcrystalline cellulose from the *Dracaena arborea* plant was 61.05% while that from the alkaline hydrolysis gave 54.32%, this shows that the acid hydrolysis method impacts higher efficiency in the extraction of microcrystalline cellulose from the plant stem.

The organoleptic properties of the extracted MCC obtained from *D. arborea* plant shows a good property with regard to colour, taste and texture and these all conformed to the BP specification for microcrystalline cellulose. Thus the MCC obtained could be recognized to be useful for pharmaceutical application as they seem not to be affected by the type of hydrolytic process.

FTIR analysis is often used to identify the various functional groups present in the sample. From the interpreted spetra, there is a peak at (1650-1850cm-^{2}), indicating the presence of a carbonyl functional group, that, intermediate between (1700-1750cm^{-2}), shows presence of an aldehyde or ketone functional group. Also the peak at (3000-3175cm^{-2}) depicts the presence of an aromatic C-H stretch, at 750cm^{-2} it shows the presence of an Ortho substituted aromatic ring. While the peak at 1726cm^{-2} shows presence of 6 member ring. More so, there is presence of long aliphatic chain confirmed by peak at 2860-3050cm^{-2},

1412cm^{-2}, and 750cm^{-2}. However, the peak at 3200 – 3600cm^{-2} shows an -OH group stretch, while a strong and a sharp peak at 3300 – 3700cm^{-2} shows the presence of meta OH group. These however points at the basic functional groups present in the cellulose structure and hence confirms the extract as a cellulose material.

The extracted MCC conducted, showed insolubility in water and most other organic solvents but the solubility improved with the use of ionic solvent such as sodium hydroxide (NaOH) and potassium hydroxide (KOH). This therefore conforms to the common characteristics of MCC which is known to be insoluble in water and most common organic solvents but solubility improves in an ionic solvent (Chandra & Samsher, 2013).

Micromeritic properties of the extracted MCC, shows, the bulk densities for sample A and Sample B as 0.33g/ml and 0.20g/ml, while the tapped densities were 0.46g/ml and 0.29g/ml respectively. 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 may pack in such a way as to leave large gaps between their surfaces, resulting in a light powder or powder of low bulk density. Thus, the difference in bulk densities between the sample A(alkali hydrolyzed) and Sample B(acid hydrolyzed) may be attributed to any of the factors outlined (Hindi, 2017).

Angle of repose on the other hand is related to the inter-particulate friction or resistance to movement between particles. Angle of repose is not an intrinsic property of the powder i.e. it is very much dependent upon the method used to form the cone of powder. The angle of repose of alkaline hydrolysed MCC is 28.20^0, while that of the acid hydrolysed MCC was 43.53^0. More so, the flow rate for alkaline hydrolysed cellulose was 0.68g/sec but sample B was not flowable hence its angle of repose was determined by Cone method. The angle of repose for sample A and B indicated an excellent and passable flow respectively as shown in the results of Table 3.

The compressibility index and Hausner's ratio are determined by measuring both the bulk volume 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**.** As the value of these indices increases, their flowability decreases. From the data analysed, the sample A had a good flow characteristic while the sample B is non- flowable.

In general, however, Hausner's ratio greater than 1.25 indicates poor flow, and Carr's compressibility index below 16% indicates good flow while values above 35% indicate cohesiveness (Rana et al., 2022). With the result obtained, sample A has Hausner's ratio of 1.35 and 1.45 while Carr's compressibility index was 20% and 31.03% for samples A and B respectively. Thus, following the results as obtained, it can be said that the powders obtained are poorly flowable, since their flow rate was above 16% although the samples are not too cohesive as the Carr's index is below 35%.

Swelling, generally accepted as an indication of tablet disintegration ability can be assessed by the determination of hydration capacity, swelling capacity and moisture sorption profile. From the results, the swelling capacity for sample A is 1.143ml, hydration capacity 3.60g and moisture sorption capacity, 0.10%, while sample B has swelling capacity of 4.20ml, hydration capacity, 3.80g and moisture sorption, 0.18%. The theory of disintegration is guarded by the fact that penetration of water (or any liquid medium) must precede its action. Hence with this result, the swelling capacity of the microcrystalline cellulose could be taken as satisfactory.

The degree of crystallinity of a polymer is defined as the fraction of the sample which is crystalline and increasing the crystallinity increases hardness and density as the degree of crystallinity has big influence on hardness, density, transparency and diffusion. The moisture sorption capacity of a material measures the sensitivity of that material to moisture. It has been reported that the crystalline portion of cellulose are largely impermeable to pollutants

and moisture due to densely packed polymer chains thus the sorption of polymers should be proportional to the amount of amorphous cellulose present as lower crystallinity index (a quantitative indicator of crystallinity) has a better water absorption ability (Mihranyan et al., 2004).

From the figures, it is seen that the moisture sorption capacity of sample A (0.25g) is lower than that of sample B (0.10g) and this could be due to the amount of amorphous cellulose likely present in the cellulose fibrils. Also study of water sorption is of importance since it reflects the relative physical stability of hard granules when stored under humid conditions (Teraoka et al., 2009), as moisture sorption capacity of cellulose powders showed that they are sensitive to atmospheric moisture and therefore, their storage should be in an airtight container.

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 sample A is observed to be lower (44.55cm) while that of sample B is higher (75.3cm). However, the bulk densities of Sample A and B were (0.33g/ml and 0.20g/ml) respectively, this shows that sample A will require lower packing volume but thicker packaging material than sample B.

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 of Sample B (1.86) was comparable to sample A (2.00) as seen in the result presented. Direct correlation exists between the degree of crystallinity of cellulose and its true density especially when determined in a non-polar liquid (Nakai et al., 1977). Consequently, the true density values for both cellulose materials suggests that sample B have a slightly elevated degree of crystallinity when compared with sample A, which seem to be more amorphous.

Variation in pH was observed between the two batches where batch A had value of 7.40, while that of sample B was 6.98. This difference may not be unconnected to the method/protocols used for their extraction. Although, the values obtained are still within the acceptable range hence both samples could be used in various formulations necessary for both systemic and external applications without harm.

Cellulose is known to be the chief constituents of plants cell wall, and it encloses varying amount of elemental and proximate principles. The average carbohydrate content of the extract was 15.38%, protein (0.438%), lipid (0.60%) and fiber (65.78%) an indication of high yield and good quality MCC and this could be of immense importance in the industrial/ manufacturing sector especially as the constituents could be chiefly employed in the food industry to quantify the nutritional values of a food substance, more so, as the low level of protein and lipid will ensure a stable formulation.

Conclusion

This research centred on the isolation of microcrystalline cellulose from *D. arborea* plant stem using two methods (alkaline and acid hydrolysis) and comparing their efficiency and yield.

The micromeritic results reveals, that the MCC from the acid hydrolysis method is more efficient, save time to obtain and gave a much higher yield than that of the alkaline. However, the powder flow of the acid hydrolysed was reduced probably as a result of very fine particles appearing like nano crystalline particles after the final hydrolysis although both hydrolytic pathways have proved the production of good quality cellulose albeit with varying characteristics.

The flow properties of a material are known to be essential in determining their suitability of as a direct compression agent and both extracts showed a good prospect.

Following the observations and results, the MCC obtained from the *D. arborea* plant stem based on the two extraction procedures, could be of great potential and may serve as substitute for the standard pharmaceutical binders and disintegrants in fast dissolving solid dosage formulations in the pharmaceutical formulations.

This finding is therefore suggestive of further research on the profiling of the phytochemical constituents of *D. arborea* plant stem for more insight towards its relevance and industrial applications.

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