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Characterization of extracted alpha cellulose from *Manihot esculenta* (cassava) peels as formulation aid for paracetamol tablet

Ebhodaghe Augustine EBOSELE [1], Johnbull Aiwaguore OBARISIAGBON [1, *], Alebiowu GBENGA [1], Tunde OWOLABI [2] and Collins Ovenseri AIREMWEN [3]

[1] *Department of Pharmaceutics and Pharmaceutical Technology, College of Pharmacy, Igbinedion University, Okada, Edo State, Nigeria.*
[2] *Department of Pharmacognosy, College of Pharmacy, Igbinedion University, Okada, Edo State, Nigeria.*
[3] *Department of Pharmaceutics and Pharmaceutical Technology, Faculty of Pharmacy, Cyprus International University, Nicosia, Cyprus.*

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Abstract

Background: Alpha-cellulose as a pharmaceutical excipient can be employed as a binding agent in tablet formulation. Cassava peels husk are waste agricultural material and is being investigated for its α-cellulose for use in oral solid pharmaceutical formulation.

Purpose: The objective of this study is to extract α-cellulose from cassava peels agro-industrial waste using a standard method, subject the extracted cassava peels cellulose and determine its phytochemical and physicochemical properties of formulated granules of paracetamol.

Methods: Alpha-cellulose from cassava peel was extracted using alkali method. The fresh peels were prepared, milled into slurry and de-watered to obtain a coarse wet material, dried, milled and sieved to obtain very fine powdered particles. Phytochemical analysis and characterization of extracted α-cellulose powder were determined. Differential scanning calorimetry (DSC), scanning electron micrographs (SEM), and x-ray diffractogram (XRD) analysis of extracted cassava peel cellulose were done. Paracetamol granules was prepared by wet granulation using varying concentrations of the binders.

Results: DSC and XRD results revealed a semi-crystalline material with amorphous and crystalline regions. while the SEM showed a lumpy structure and reticular shape.

Pre-compression evaluations of the formulated batches of paracetamol granules showed that cassava peels cellulose (CPC) have similar flow properties with those of microcrystalline cellulose BP (MCC) and acacia BP (ACA) powders respectively. The granules have free flow properties with average angles of repose (CPC ≤ 27.6^0 MCC ≤ 26.9^0 and ACA ≤ 25.6^0), compressibility index (CPC ≤ 25, MCC ≤ 23 and ACA ≤ 21.7%), Hausner's ratio (CPC ≤ 1.32, MCC ≤ 1.31, and ACA ≤ 1.27) respectively.

Conclusion: The extracted cassava peel α-cellulose was found ideal as pharmaceutical excipient binder in oral solid dosage forms.

Keywords: *Manihot esculenta*; α-Cellulose; Binder; Tablet; Acacia; Wet granulation, Microcrystalline, Acacia.

* Corresponding author: Johnbull Aiwaguore Obarisiagbon

1. Introduction

Developing countries are highly reliant on importation of pharmaceutical raw materials and finished products (Saigal *et al*., 2009), hence the search for natural local and effective excipients that are readily available, safe and economical. Pharmaceutical industries in developing countries have recognized the importance of using local and natural occurring materials (Adedokun and Nkori 2014). One category of such excipients that has enjoyed wide and considerable use in the pharmaceutical and food industries is Cellulose. Alpha-cellulose obtained as a pulp from fibrous plants is treated with mineral acids to obtain microcrystalline cellulose (MCC) which has been used as pharmaceutical excipient because of its good flowability, compatibility and compressibility. Binding agents impact cohesive qualities to powdered materials, and are employed in pharmaceutical tablet formulations to provide adequate mechanical properties by promoting the bond existing between the different components of a powder mix in a formulation thereby enhancing the strength of the eventual tablet produced (Ogunjimi and Alebiowu, 2010; Adetogun and Alebiowu., 2009, Toneja *et al* 1999). Cassava (*Manihot esculent*) is the fifth most abundant starch crop produced in the world, the third most important food source for inhabitants in the tropical areas (Mandal and Chakra; 2011), and Nigeria is far the largest cassava producing country in the world with about 60 million tonnes production as of 2018, (FAOSTAT, 2019, Heliyon, 2020). Cassava bagasse and cassava peels are produced in large quantities by industrial tapioca starch. The bagasse is usually used as animal feed whereas cassava peel is very rarely used and are wasted. The peels of cassava contribute 15% of the tubular cassava root weight and the abundant production of this crop has resulted in the yield of a huge amount of peels that are discarded as waste. Therefore, the cassava peels have potential as large source of α-cellulose.

2. Materials and methods

2.1. Materials

Cassava peels, Sodium hydroxide pellets/ crystals, Bodi®, Sodium Hypochlorite, Paracetamol powder BP, Microcrystalline cellulose BP (MCC), Acacia gum BP, Corn starch BP and Lactose BP powders. All other chemicals and reagents used were of analytical grade.

2.2. Methods

2.2.1. Collection and Identification

Fresh cassava peels were collected from local cassava processing plant at Okada in Ovia North East Area of Edo State, Nigeria. The cassava peels were identified and authenticated by Dr. Adebayo of the Department of Pharmacognosy, College of Pharmacy, Igbinedion University, Okada, Edo State, Nigeria with Haberium Number IUO/11/036.

2.2.2. Extraction of cassava peel α-cellulose

Cassava peels were washed thoroughly and milled into slurry, dewatered, pulverized and was sundried for 72 hours. The coarse powder was milled and sieved to obtain very fine powder particles. Alpha-cellulose was isolated using alkaline method (Widiarto et al 2019). About 400 g of dried powder was refluxed with 8 liters of 4% sodium hydroxide for 2 hours at 90 ^{0}C with constant stirring for delignification (removal of lignin and β-and γ-cellulose), the mixture was bleached with 4 liters of 4% sodium hypochlorite for 1 hour at 80 ^{0}C with constant stirring (Alalor and Edo 2020).

The mixture of mucilage was passed through a muslin cloth. The residue obtained was washed with distilled water until a neutral pH, and was oven dried at 60 ^{0}C for 1 hour, milled and sieved to obtain fine powder.

Test for cellulose: The method of Eraga *et al*., (2016) was adopted in which the extracted powder (10 mg) was dispersed in 2 ml of iodinated zinc chloride solution and colour change noted.

2.3. Phytochemical analysis and characterization of extracted α-cellulose powder

Extracted cassava peels cellulose was subjected to phytochemical analysis in line with Obarisiagbon *et al*, (2023) method.

2.3.1. Physicochemical properties of extracted cellulose (CPC)

Assessment of organoleptic properties: Organoleptics of extracted cassava peel cellulose odour, taste, colour and texture were examined using sensory organs and observations recorded (Nnamdi *et al*, 2009).

2.3.2. Solubility of extracted cellulose

About 1g of extracted cellulose CPC was dispersed in 20 ml distilled water in a test tube at room temperature. The dispersion was intermittently shaken for the period of 24 hours and then filtered using a pre-weighed filter paper. The result of the residue on the filter paper was air dried and weighed. The solubility was calculated as the percentage different between the initial weight of the cellulose sample and the final weight on the filter paper residue using the formula

$$wt - wf / wo \times 100 \quad \text{---------- 1}$$

2.3.3. Melting point

Cellulose powder was packed into a one end sealed capillary tube and tapped on hard surface to form a column at the bottom of the capillary tube and inserted into the heating block of a Gallenkamp melting point apparatus (model), Melting point of sample CPC was determined and recorded.

2.3.4. Swelling capacity/index

The method of Okhamafe *et al* (1991), and used by Eraga *et al* (2016), was adopted for the determination of the swelling capacity of the extracted cellulose.

$$\text{The swelling index was calculated using the formula.} = (v\_2\text{-}v\_1)/v\_1 \times 100 \quad \text{---------- 2}$$

Where v1 = powder tapped volume, v2 = volume of the sediment (Eraga S.O *et al*, 2016).

Micromeritic of α-cellulose extracted from cassava peels

2.3.5. Bulk density

Extracted cellulose (20 g) was weighed into a 100 ml measuring cylinder. The volume occupied by the powder was record as bulk volume (vb). Mean of triplicate values of bulk density was calculated using equation below.

$$\text{Bulk density (g/cc)} = (\text{Mass of powder (w)})/ (\text{Bulk volume (vb)}) \quad \text{---------- 3}$$

2.3.6. Tapped density

The measuring cylinder was tapped on a flat surface about 100 times to a constant volume (vt), and the mean of triplicate values of tapped density was recorded (Obarisiagbon and Uhumwangho, 2018).

$$\text{Tapped density (g/cc)} = (\text{mass of powder (w)})/ (\text{tapped volume (vt)}) \quad \text{---------- 4}$$

Carr's (compressibility) index

Carr's (compressibility) index of extracted cellulose powder was evaluated using equation (Carr, 1965).

$$\text{Carr's index} = (\text{Tapped density-Bulk density})/ (\text{Tapped Density}) \times 100 \quad \text{---------- 5}$$

2.3.7. Hausner's ratio

The ratio of the tapped density to the bulk density of the extracted cellulose powder calculated using equation

$$\text{Hausner's ratio} = (\text{Tapped Density})/ (\text{Bulk Density}) \quad \text{---------- 6}$$

2.3.8. Flow Rate

Extracted cellulose powder (20 g) was weighed into a glass funnel with orifice of 0.75 cm fixed on a retort stand. The orifice was then open at time 0 second and the time taken for the entire mass powder to pass through the funnel orifice was recorded. Mean values of triplicate determinations were recorded. The flow rate was calculated using equation

$$\text{Flow rate (g/s)} = (\text{Weight of powder})/ (\text{Time}) \quad \text{---------- 7}$$

2.3.9. Angle of repose

The angle of repose was determined by allowing extracted powder (30 g) to flow through funnel with orifice 0.7cm placed at a fixed height onto a flat surface to form a cone-like heap. The height of the heap was measured and the angle of repose (θ), was calculated using equation

$$\theta = 〖\tan〗^{(-1)} h/r \text{ ----------8}$$

2.3.10. High resolution analysis of sample (CPC)

High resolution analysis of extracted α-cellulose (CPC) were determined under the following: Differential scanning calorimetry (DSC), Scanning electron microscopy (SEM), and X-ray diffraction (XRD) and results printed and recorded.

Table 1 Formula for paracetamol tablet 500 mg/tablet of different binders

Formulation	**Bacth cpc i**		**Batch cpc ii**		**batch cpc iii**		**batch cpc iv**	
	QTY\TAB(MG)	**QTY \ 200 TAB(G)**	**QTY\TAB (MG)**	**QTY\200 TAB (G)**	**QTY\TAB (MG)**	**QTY\200 TAB (G)**	**QTY\TAB (MG)**	**QTY\200 TAB (G)**
Paracetamol BP	500	100	500	100	500	100	500	100
Corn starch BP	60	12	60	12	60	12	60	12
Lactose BP	20	4	20	4	20	4	20	4
Extracted Cassava Peel cellulose w\v		5%		75%		10%		12.5%
Dry corn starch 5%w\w	30	6	30	6	30	6	30	6
Magnesium stearate BP 1% w\w	12	2.4	12	2.4	12	2.4	12	2.4
Compression Weight	620	0.62	620	0.62	620	0.62	620	0.62
INGREDIENT FORMULATION II	**BATCH MCC I**		**BATCH MCC II**		**BATCH MCC III**		**BATCH MCC IV**	
	QTY\TAB(MG)	**QTY \ 200 TAB(G)**	**QTY\TAB (MG)**	**QTY\200 TAB (G)**	**QTY\TAB (MG)**	**QTY\200 TAB (G)**	**QTY\TAB (MG)**	**QTY\200 TAB (G)**
Paracetamol BP	500	100	500	100	500	100	500	100
Corn starch BP	60	12	60	12	60	12	60	12
Lactose BP	20	4	20	4	20	4	20	4
Micro crystalline Cellulose BP % w\v		5%		7.5%		10%		12.5%
Dry Corn Starch BP 5% w\w	30	6	30	6	30	6	30	6
Magnesium stearate BP 1% w\w	12	2.4	12	2.4	12	2.4	12	2.4
Compression Weight	20	0.62	620	0.62	620	0.62	620	0.62

Ingredient formulation III	BATCH ACA I		BATCH ACA II		BATCH ACA III		BATCH ACA IV	
	QTY\TAB(MG)	QTY \ 200 TAB(G)	QTY\TAB (MG)	QTY \ 200 TAB(G)	QTY\TAB (MG)	QTY \ 200 TAB(G)	QTY\TAB (MG)	QTY \ 200 TAB(G)
Paracetamol BP	500	100	500	100	500	100	500	100
Corn starch BP	60	12	60	12	60	12	60	12
Lactose BP	20	4	20	4	20	4	20	4
Acacia powder BP % w\v		5%		7.5%		10%		12.5%
Dry corn starch BP 5% w\w	30	6	30	6	6	30	6	30
Magnesium stearate BP 1% w\w	620	0.62	620	0.62	620	0.62	620	0.62
Compression Weight	620	0.62	620	0.62	620	0.62	620	0.62

Key CPC = cassava peel cellulose, MCC = microcrystalline cellulose, ACA = acacia powder

2.4. Preparation of paracetamol granules

The excipients and paracetamol powder were carefully weighed and sieved into a mixing bowl, and different binders' concentrations (CPC, MCC, and ACA) were separately added and dry mixed. Wet granulation method was used to wet mass the mixed powders, dried in hot air oven at 60oc for 1 hr and screened through 20 mesh sieve size and stored in an air tight container. Same procedure was repeated for the remaining formulations of microcrystalline cellulose (MCC) and acacia (ACA).

2.5. Physicochemical analysis of paracetamol granules

Physicochemical properties of paracetamol granules were evaluated. The granules flow rate, angles of repose, bulk and tapped densities, Carr's (compressibility) index and Hausmer's ratio were evaluated in triplicate and the average values recorded as shown in Table 4.

3. Results

Table 2 Organoleptic/physical properties of extracted cellulose (CPC)

Properties	Extracted cellulose
Extract yield (%)	12.03
Appearance / colour	Light brown
Texture	Smooth
Taste	Tasteless
Odour	Odourless
Solubility in water	Insoluble
Melting point (^{0}c)	205-268
Swelling capacity (%)	6.25

Table 3 Phytochemical analysis of extracted cellulose (CPC)

Phytochemical constituents	Extracted cellulose
Cellulose	+ ve
Saponins	— ve
Flavonoids	+ ve
Alkaloids	+ ve
Tannins	— ve
Cardiac glycosides	+ ve
Steroids	+ ve
Terpenoids	+ ve
Anthraquinones	— ve
Cyanogenetic glycosides	± ve

Key: + ve (present), - ve (absent), ± ve (present but not pronounced)

Table 4 Heavy metal analysis of extracted cassava peels α-cellulose (CPC)

As (µ/kg)	Cd (ug/kg)	Cr (ug/kg)	Cu (ug/kg)	Pb (ug/kg)	Ni (mg/kg)	Zn (mg/kg)
15.43 WHO/FAO (value = 20.0)	12.52 WHO/FAO (value = 22.0)	30.81 WHO/FAO (value = 23.0)	14.22 WHO/FAO (value = 730.0)	21.11 WHO/FAO (value = 30.0)	22.60 WHO/FAO (value = 670.0)	19.50 WHO/FAO (value = 995.0)

Key: As (Arsenic), Cd (Cadmium), Cr (Chromium), Cu (Copper), Pb (Lead), Ni (Nickel), Zn (Zinc)

Table 5 Micromeritic properties of paracetamol granules

Formulation Batches	Binder Conc. (% w/v)	Flow Rate (g/sec)	Angle of repose (°)	Bulk density (g/ml)	Tapped Density g/ml	Carr's Index (%)	Hauser's ratio
CPC-I	5.0	4.10 ± 3.00	27.60 ± 1.5	0.67 ± 0.02	0.89 ± 0.04	25.00 ± 1.24	1.32 ± 0.08
CPC-II	7.5	3.78 ± 0.12	27.43 ± 0.76	0.65 ± .01	0.81 ± 0.05	18.84 ± 6.60	1.23 ± 0.20
CPC-III	10.0	2.66 ± 0.20	26.70 ± 0.17	0. 67 ± .01	0.84 ± 0.03	20.0 ± 1.56	1.25 ± 0.09
CPC-IV	12.5	3.07 ± 0.17	26.8 ± 1.01	0.67 ± 0.03	0.82 ± 0.18	18.48 ± 5.48	1.22 ± 0.09
MCC- I	5.0	3.80 ± 0.18	26.9 ± 0.31	0.65 ± 0.05	0.86 ± 0.08	23.63 ± 1.80	1.31 ± 0.16
MCC- II	7.5	4.58 ± 1.08	25.64 ± 0.30	0.68 ± 0.09	0.82 ± 0.04	17.02 ± 1.80	1.20 ± 0.08
MCC- III	10.0	5.44 ± 1.21	26.50 ± 1.22	0.63 ± 0.20	0.80 ± 0.80	22.0 ± 0.08	1.27 ± 0.05
MCC- IV	12.5	4.52 ± 0.15	26.01 ± 0.35	0.63 ± 0.12	0.77 ± 0.15	18.80 ± 0.56	1.23 ± 0.07
ACA- I	5.0	4.47 ± 0.28	25.49 ± 0.47	0.69 ± .06	0.88 ± 0.06	21.50 ± 1.10	1.25 ± 0.07
ACA- II	7.5	5.65 ± 1.07	24.28 ± 0.55	0.73 ± 0.06	0.85 ± 0.07	15.55 ± 2.25	1.16 ± 0.09
ACA- III	10.0	4.48 ± 0.35	26.12 ± 0.38	0.68 ± 0.28	0.87 ± 0.05	21.68 ± 1.12	1.27 ± 0.05
ACA- IV	12.5	6.03 ± 1.50	25.64 ± 0.31	0.70 ± 0.04	0.86 ± 0.09	18.30 ± 1.87	1.22 ± 0.09

Key: CPC = cassava peel cellulose, MCC = microcrystalline cellulose, ACA = acacia powder

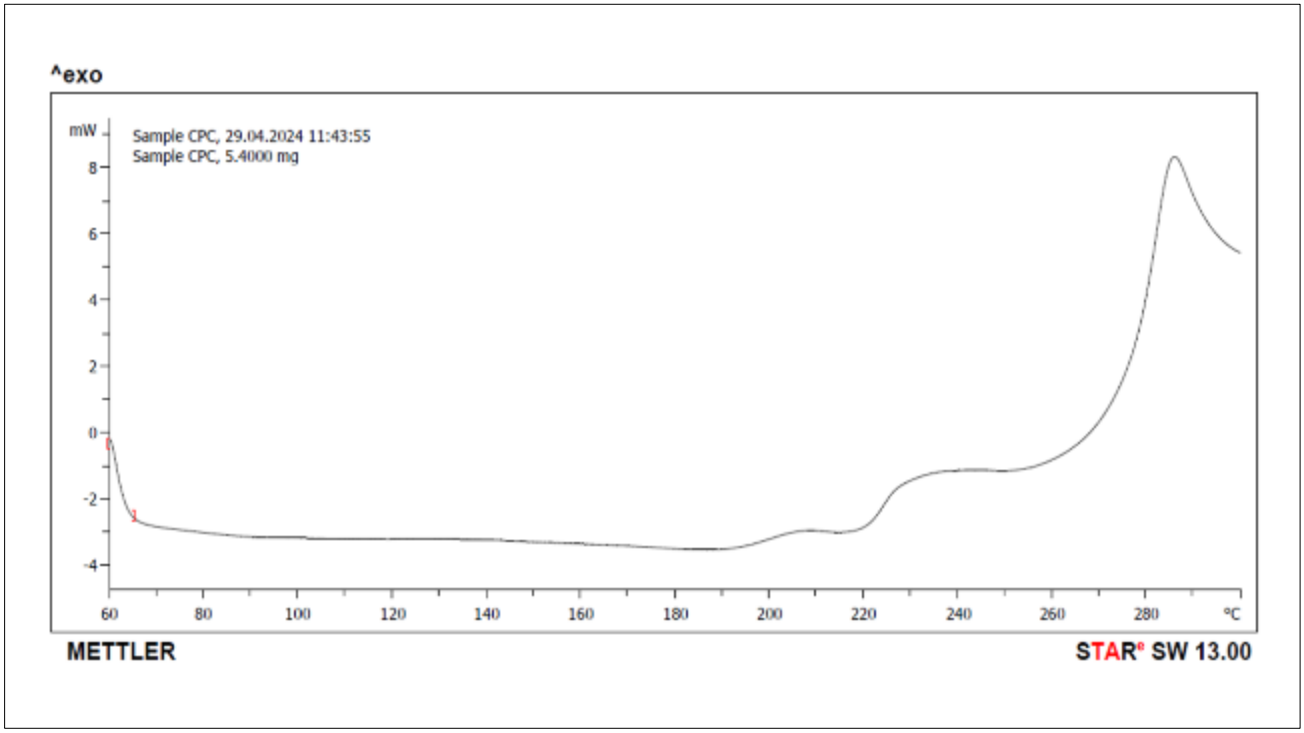

Figure 1 Differential scanning calorimetry (DSC) of cassava peel celulose (CPC)

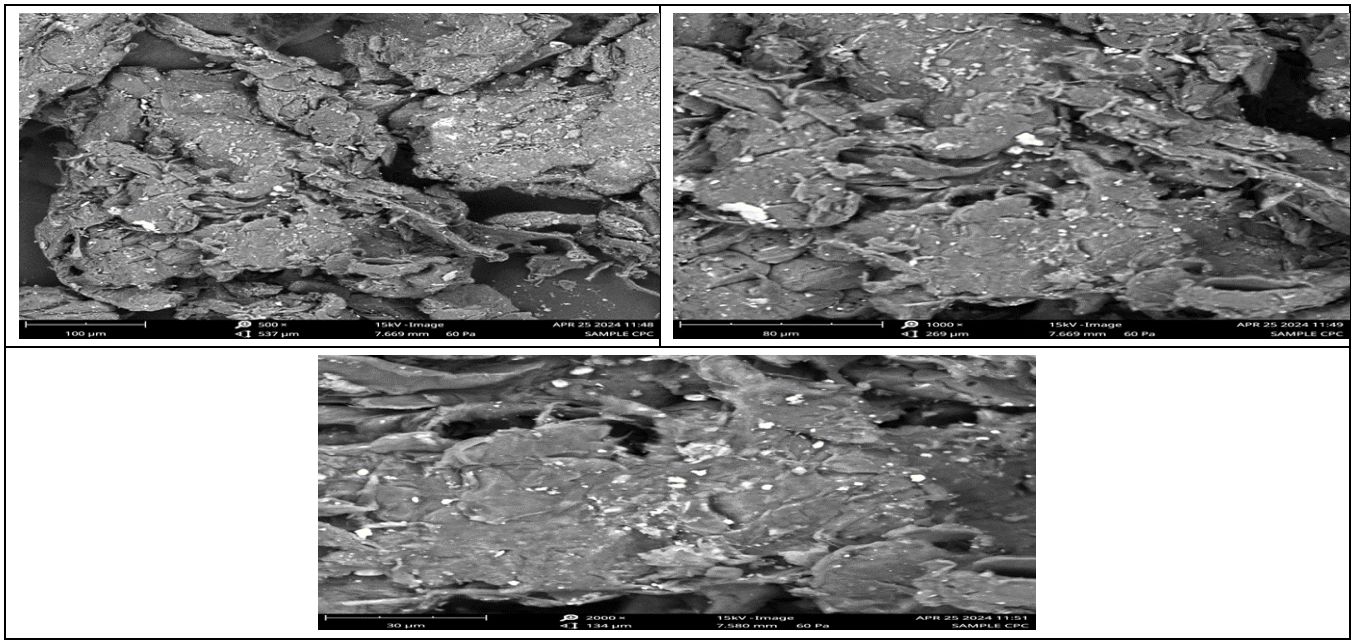

Figure 2 Scanning electron micrographs of extracted (SEM) cellulose powder (Magnification: A(500x), B(1000x), C(2000x)

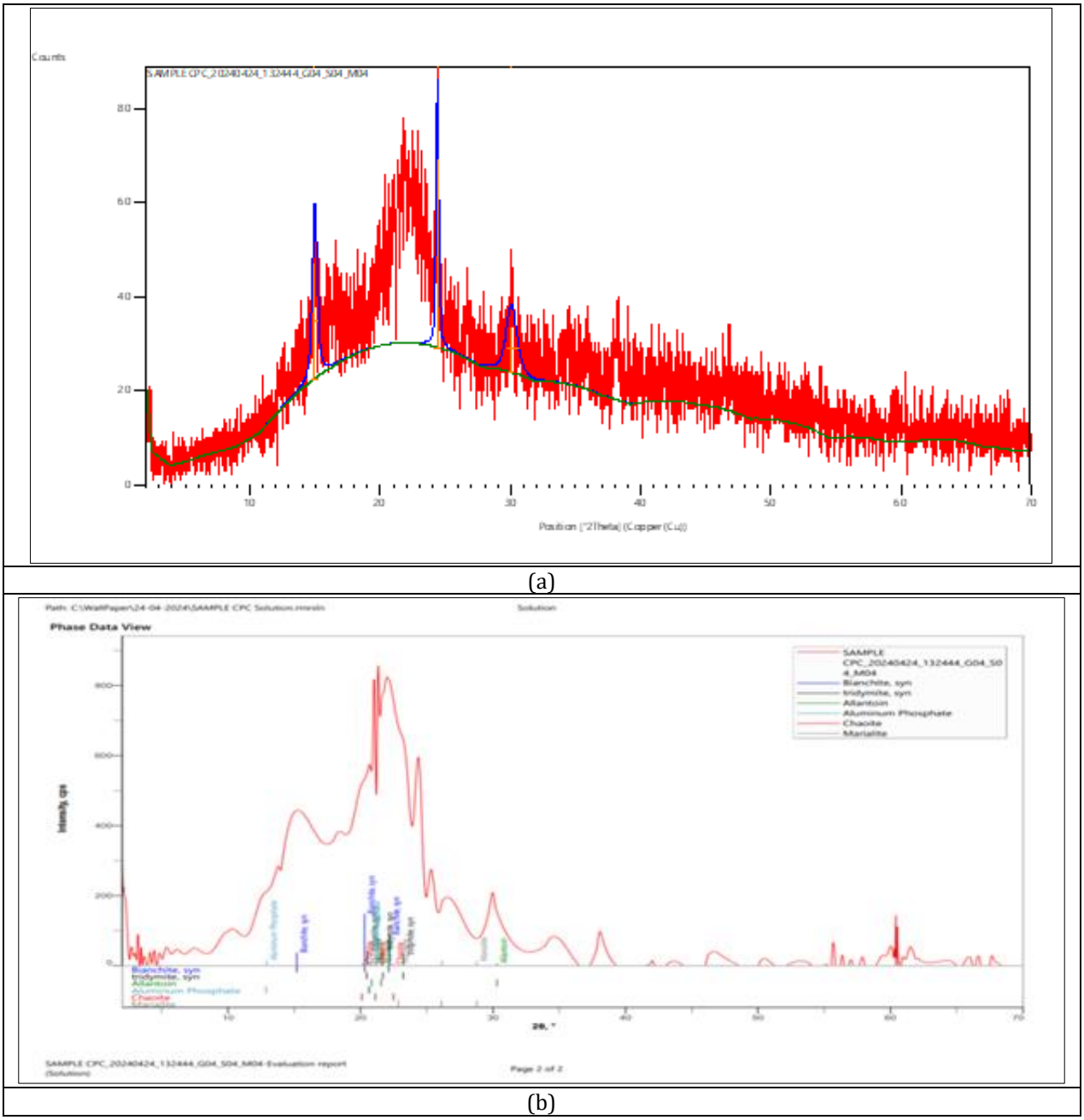

Figure 3 (a)(b). X-ray Diffractogram of cassava peel cellulose (CPC)

4. Discussion

Table 1 shows the yield of extracted cassava α-cellulose to be 12.03%. The low yield may be due to the experimental conditions such as reaction temperature, reaction time, agitation, concentration of sodium hydroxide, concentration of acid for hydrolysis and muslin cloth (the pores size) used. However the percentage yield was relatively similar to work reported by (Widarto *et a,l* 2019 and Leite *et al*, 2017) with 17.8% and 10.5% respectively. Organoleptic properties are reported in Table 2 and also melting point which ranged between 205-268 ^{0}C similar to earlier reported work by Galiwango *et al.,* (2019) indication that cellulose decomposed at temperature between 200-300 ^{0}C the swelling capacity was found to be 6.25%.

Phytochemical analysis (Table 2) of the extracted α- cellulose powder showed presence of flavonoids, alkaloids, cardiac glycosides, steroids, terpenoids and cyanogenic glycosides. These results are similar to those obtained from previous research by Olaniyan and Ajayi, 2021. Results of analysis of heavy metals (Table 3), were similar to previous studies by Mensah *et al*., 2009 and were all below permissible limits recommended by WHO/FAO. Result also showed that

extracted cassava cellulose exhibited a melting point temperature range of 205 – 268 ^{0}C which is similar to research by Galiwango *et al*, (2019) in which cellulose was reported to decomposed at temperature between 200 to 300 ^{0}C. The micromeritic properties of paracetamol granules are shown in Table 4.

Cassava peels α-cellulose (CPC) exhibited excellent to passible flow characteristics, with angle of repose, Carr's index and Hausner's ratio values of 29.710 ± 0.530, 21.2% ± 4% and 1.27 ± 0.07 respectively. The relative high bulk and tapped densities (0.75 ± 0.01 and 0.98 ± 0.05 g/cc) of the cassava cellulose agreed with previous research reported by Riatong *et al*., 2014, Ding *et al*., 2020).

These values show that CPC powder had less porosity and therefore will be less compressible according to studies by Ding *et al*, (2020). Porosity may hinder densification of powder bed when external stress is applied. Higher values of bulk density also pre-supposes existence of materials with higher sphericity, low interstitial air content and high inter-particulate cohesiveness as opposed to coarse particles with more irregular shapes and more particulate-container adhesiveness. (Alalor and Edo 2020; Chineze *et al*., 2021, Okeke *et al*., 2021). Hence, extracted cassava peel α-cellulose would be an ideal binder for incorporation in powdered raw materials in finished products requiring compact packaging.

Thermal analysis of the extracted α-cellulose from cassava peel is shown in Figure 1. The thermogram exhibited a single sharp peak at about 288 ^{0}C with a smaller peak at 210 ^{0}C and 230 ^{0}C respectively. The major single sharp peak at about 288 ^{0}C is representative of the melting point of the cellulose. While the sharpness of the peak is a representation of the crystallinity of the cellulose powder materials, the width of the peak indicates the amorphousity of the powder material, hence confirming the semi-crystalline nature of the extracted cassava peels α-cellulose powder. The two minor peaks at about 210 ^{0}C and 230 ^{0}C are indicative of loss of water from the cellulose sample at about 210 ^{0}C and the melting point of the degradative product of the cellulose at 288 ^{0}C. The scanning electron microscopy (SEM) was used to determine the form and surface characteristics of the extracted cassava peel cellulose shown in Figure 2 at various magnifications (500X, 1000X and 2000X). The surface morphology of the sample CPC presented lumpish structure, which may be due to the strong intra-molecular hydrogen bond, which is in line with earlier research reported by Adewuyi and Vargas (2017). The reticular shape of the cellulose indicated that the process of purifying and bleaching had not broken the cellulose structure. X-ray diffraction pattern of the cellulose powder (CPC) exhibited the following peaks; the first prominent peak was at 2θ= 14.960. d-spacing = 5.92097 and of intensity 24.90 counts per second (cts), followed by 2θ= 24.370, d-spacing = 3.65189 and fo intensity 40.08 counts per seconds (cts) and 2θ = 30.06, d-spacing = 2.97283 and of intensity 9.78 counts per second (cts). Their Full width at half-maximum (FWHM) values indicative of crystallinity of the powder sample showed that the first and second peaks width are small, indicate that the materials is crystalline while the peak value which is large suggests the material is amorphous, confirming the semi-crystallinity nature of the extracted cellulose. These results are in agreement with previous studies on the crystallinity. (Martens *et al.*, 2018; Dome *et al.*, 2020).

5. Conclusions

Cellulose from cassava peels was successfully extracted with percentage yield of 12.03%,

swelling capacity of 6.25% and with good powder flow properties. Pre-compression evaluations of formulated batches of paracetamol granules, showed that all batches of CPC have similar properties with those of MCC and ACA standard powder materials respectively.

The conversion of cassava peel into industrial pharmaceutical raw material such as α-cellulose will salvage the environmental nuisance associated with waste as well as adding value to the crop, economic empowerment to the farmers, and individual entrepreneur and increase the national economic base.

The study has developed a potential pharmaceutical-grade α-cellulose for tablet formulation as a natural binder from plant waste materials.

Compliance with ethical standards

Disclosure of conflict of interest

The Author declares that he has no conflicts of interest for this article.

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