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THE EFFECT OF PARTICLE SIZE OF GROUNDNUT HUSK FOR THE PRODUCTION OF MICROCRYSTALLINE CELLULOSE (MCC) FROM GROUNDNUT HUSK (GH)

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ABSTRACT: *Microcrystalline cellulose (MCC) is an important product used in the pharmaceutical, food, cosmetics and wood pulp industries. Agricultural waste, now the recent and most commonly used material for the production of MCC has found significance in the market due to the high content of cellulose and availability. In this work, microcrystalline cellulose was produced from various selected sizes of groundnut husk to obtain an optimum particle size for the production of GH-MCC at fixed temperature, time and concentration. Alkali treatment was used followed by bleaching in this process. The FTIR indicates extensive removal of lignin and hemicellulose, while the physicochemical analysis conforms to standards. The percentage yield obtained were 32%, 32%, 32%, 28%, 32%, 44%, 28%, 32%, 52%, 36%, 56%, 48% AND 40% for the particles sizes 3.35mm, 2.80mm, 2.36mm, 2.00mm, 1.70mm, 1.40mm, 1.19mm, 1.18mm, 1.00mm, 0.560mm, 0.500mm, 0.425mm and 0.212mm respectively.*

KEYWORDS: Groundnut husk (GH), microcrystalline cellulose (MCC), physicochemical analysis

INTRODUCTION

Nigeria is one of the African countries of the world with various oil seeds notably groundnut, oil-palm, soybean and cotton seeds. Been the fourth largest producer of groundnut in the world and the highest producer in Africa with 3.8 million metric tons (FAO, 2011) .The Northern part of Nigeria grows about 88% of the total groundnut production. The producing states includes Kano, Kaduna, Taraba, Bauchi, Bornu, Adamawa, Niger, Jigawa, Benue, Zamfara, Kebbi, Sokoto, Yobe, Plateau, Nassarawa and Gombe states (NAERL, 2011). However, the management of this agricultural waste is less effective which constitute to environmental issues. Therefore by utilizing the potential benefits of these crop residues useful products such as microcrystalline cellulose (MCC) can be produced.

The chemical composition of groundnut husk is 18.7% of hemicellulose fiber, 35.7% cellulose, 30.2% lignin and 5% of ash contents. Cellulose is the most abundant organic compounds in the world which is obtained from plants. It has several applications in industries such as veterinary foods, wood and paper, fibers and clothes, cosmetic and pharmaceutical industries. Pure cellulose is in different forms in the market with different mechanical and pharmaceutical properties whose grades influence their particle size and degree of crystallization. Cellulose serves as thickening agents and stabilizers, binders, fillers. MCC exhibits excellent properties in composite fabrication such as renewability, biocompatibility, biodegradability, high surface area for bonding with resins (Kamel, 2008) due to its and biodegradability. The process of extraction of microcrystalline

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cellulose utilizes the removal of hemicellulose and lignin which constitutes the matrix for cellulose micro fibrils. The isolation and characterization of microcrystalline cellulose from different sources like corn stalk (Reddy et al. 2005), rice husk (Reddy and Yang, 2005; Hanani et al., 2017), wood (Orts, 2005), sugar cane bagasse (Sun, 2004), cotton (Ohwoavworhua et al. 2005; Li *et al.*, 2019), coconut husk (Rosa, 2010), rice husk, sugar cane bagasse, corn cob, and cotton (John et al. 2011), sugarcane bagasse, rice straw, durra stalk and groundnut shell (Mohammed et al., 2015), Bamboo fiber (Lalduhsanga et al.,2013), oil palm (Loo et al.,2016),groundnut husk (Ohwoavworhua et al.,2009;Chukwuemeka et al.,2012; Rani et al., 2016) etc. have been previously reported. However, the effect of particle sizes on the production of microcrystalline cellulose from groundnut husk or other agricultural waste has not been reported. Therefore, the aim of this work is to study the effect of the particle sizes of groundnut husk for the production of microcrystalline cellulose production and obtain the optimum particle size for the optimization of process conditions in the production of MCC from groundnut husk. Also, Nigeria has abundant groundnut hush waste as source of cellulose of over 3.8 milliom MT (FAO, 11), this findings will help the reduce of celluloe importation and rather Nigeria can now develop an industry for the production of cellulose/ nanocrystalline cellulose and microcrystalline cellulose and as well import to other African country for pharmaceutical industries.

EXPERIMENTAL

Materials

Groundnut husk was obtained from Institute of Agricultural Research A.B.U Zaria, Nigeria and the specie and type of the groundnut husk was identified in Biological Sciences Department and Biochemical Laboratory as the raw material. The chemicals used were NaOH (BDH England), Ethanol, Nitric Acid, NaCLO. The result is shown in Table 1, all chemicals used are of reagent.

Particle size selection

The groundnut husk was washed with distilled water and sundried for three days to remove moisture. Dried groundnut husk were grounded using a ball mill and were sieved using the following sieve sizes: 3.35mm, 2.80mm, 2.36mm, 2.00mm, 1.70mm, 1.40mm, 1.19mm, 1.18mm, 1.00mm, 0.560mm, 0.500mm, 0.425mm and 0.212mm respectively as presented in Table 2.

Proximate Analysis:

1 g of the untreated groundnut husk was used to examine the percentages of Acid Detergent Fiber (ADF) and Neutral Detergent Fiber (NDF) to calculate percentages of cellulose, hemicellulose, lignin and ash content as shown in Table 3.

Production of Microcrystalline Cellulose

Each selected size were measured for 25 g of the husk and treated with NaOH (500 ml, 0.5M) for 2 hours at 92.1^0C with continuous stirring. The dark slurry obtained was filtered and washed several times with distilled water until pH 7 was recorded and then oven dried at 60^0C for 4hrs. The dried GH was refluxed with a mixture containing 20% (v/v) of nitric acid in ethanol in 500ml of distilled water set at a constant temperature of 94^0C in a thermostatic water bath. This treatment was carried out thrice as the colour changed from brown to orange in successive steps. The mixture

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was then filtered and washed with cold distilled water till the solution becomes neutral. The orange coloured residue was then bleached with sodium hypochlorite to obtain white cellulose. It was then oven dried at 60^0C to for 8hrs. Finally, the dried cellulose was stored in plastic containers. Finally, the dried microcrystalline cellulose was stored in plastic containers. The yield of the extracted cellulose were weight and recorded respectively. Photographs at different stages of the chemical treatments are shown in Figure 1.

(a) (b) (c) (d)

Fig.1. Photographs of (a) untreated groundnut husk GH (b) alkali treated (c) HNO_3/ ethanol refluxed (d) GH-MCC

Percentage Yield

Percentage yield was calculated using the following equation:

Percent yield (%) = $\frac{W3}{W1}$ X 100 .....Eq (1)

Where:

W3 is the weight of oven dried lignin free (pure cellulose) sample.

W1 is the weight of oven dried raw sample.

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR analysis was carried out using Shimadzu FTIR-8400S Japan spectrometer with scanning ranges of 4000.0 cm^{-1} to 500.0 cm^{-1} using Kbr pellet.

Bulk and Tap Densities

A 1.3 g quantity each of the powder samples was placed in a 50 ml clean dry measuring cylinder and the volume, V_o occupied by each of the samples without tapping was determined. After 100 taps occupied volumes, V_{100}, were determined. The bulk and tap densities were calculated as the ratio of weight to volume (V_0 and V_{100} respectively).

True Density

The true densities (Dt), of cellulose powders were determined by the liquid displacement method using xylene and pycometer. 0.5 g quantity of cellulose powder was placed in a dry pre weighed pycometer and the rest filled with 50 ml xylene (SG 0.86) as the immersion fluid, the weight of the

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pycometer filled with only liquid has previously been established and density of the powder was computed according to the following equation:

Dt = w [(a + w)-b] x SG.....Eq (2)
Where w is the weight of powder, SG is specific gravity of solvent, a is weight of bottle + solvent and b is weight of bottle + solvent + powder

The Carr's index and Hausner's ratio
These were determined from the values of the bulk and tapped densities results obtained above.

$$\text{Carr's Index} = \frac{Dtap - Dbulk}{Dtap} X\ 100 \ldots\ldots\ldots Eq\ (3)$$

$$\text{Hausner's Ratio} = \frac{Dtap}{Dbulk} \ldots\ldots\ldots Eq\ (4)$$

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

$$e = 1 - \frac{Dtap}{Dtrue} X\ 100 \ldots\ldots\ldots Eq\ (5)$$

Where D_{tap} is the tapped density, D_{true} is the true density and e is the Porosity. (chukwuemeka *et al.*, 2012)

pH determination:
1 g of the powder material was shaken with 50 ml of distilled water for 5 min and the pH of the supernatant liquid was determined using a pH meter.

Angle of repose
To obtain the angle of repose a long cylindrical tube, open at both ends, was used. The tube was perpendicularly placed on a clean cardboard paper and filled flat with the cellulose powder. The tube was then gradually lifted away, vertically, from the cardboard. The height *h*, and radius *r*, of the conical heap formed were measured, and then the angle of repose, θ, was calculated. Determination was done in triplicate and the average taken (Ohwoavworhua,*et al.*. 2004).

$\theta = \tan{-1}\ (h / r)$ ..... Eq (6)

SEM
Particle morphology of the MCC samples was studied by using China JSM 7500 field SEM.

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X-ray diffraction Analysis (XRD)
X-ray diffraction patterns of the cellulose samples were obtained using an X-ray diffractometer (Philips X-ray Analytical). Samples for analysis were prepared by pressing the powder into the cavity of a sample holder and smoothing with a glass slide. They were scanned from 5-400 2q with a Cu anode X-ray operated at 40 kV and 40 mA in combination with a Ni filter to give a monochromatic Cu-Kα radiation (l = 1.5418 Å). The X-ray beam is directed at a sample and measuring the scattered intensity as a function of the outgoing directions. Once the beam is separated, the scatter also called a diffraction pattern, indicates the sample's crystalline structure. The crystallinity index (CI) was calculated using Equation 3.7, as proposed by Rani et al (2016)

$$CI = \frac{I_{002} - I_{AM}}{I_{002}} X\ 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots Eq\ (7)$$

where:
I_{002} = is the intensity of the peak (002) lattice diffraction at ($2\theta = 16 - 18^0$)
I_{AM} = the Iam is the intensity diffraction at (2θ degrees = $20\text{-}26^0$)

RESULTS AND DISCUSSION

The groundnut husk was identified and Table present the result.

Table 1: The Identification of the untreated Groundnut husk

Kingdom:	Plantae
Genus:	*Arachis*
Species:	*Samnut 10*
Binomial name	*Arachis hypogaea*

Particle size

The particle sizes of the groundnut husk were selected using an Endecotts sieve shaker as shown in Table 2.

Table 2.0: The selected particle sizes of Groundnut husk for the production of microcrystalline cellulose.

Particle Sizes (mm)	**3.35**	**2.80**	**2.36**	**2.00**	**1.70**	**1.40**	**1.19**	**1.18**	**1.00**	**0.560**	**0.500**	**0.425**	**0.212**
Sample ID	F_1	F_2	F_3	F_4	F_5	F_6	F_7	F_8	F_9	F_{10}	F_{11}	F_{12}	F_{13}

$F_1 – F_{13}$ Untreated groundnut husk

FTIR analysis

The FT-IR spectrum of the untreated-GH (F_0) is at the peak of 3333.10cm^{-1}, while treated GH-MCC of these particle sizes (F_1, F_4, and F_8) are in the region of 3425.69cm^{-1}, (F_2, F_9, F_{10} and F_{12}) are in the region of 3410.26cm^{-1} , and (F_3, F_5, F_6, F_7, F_{11} and F_{13}) spectra are 3417.98cm $^{-1}$. The spectra of Avicel PH 101 are 3275cm^{-1} respectively. However, these intensities for the GH-MCC spectra are stronger than that for Avicel PH 101and the untreated GH. The analysis of the spectra with reference to published data (Abdul Khalil et al. 2001) shows typical features of the cellulose with the characteristic intermolecular and intramolecular O—H stretching vibration band of the alcohol group for GH-MCC and Avicel PH 101 which correspond to the peak of reference 3200-3600 cm^{-1}. The untreated GH (F_0) and GH-MCC sizes (F_5) are in the region of 2924.18 cm^{-1}, (F_3 ,F_6, F_7, F_9, F_{11}, F_{13}) are at 2931.90cm^{-1}, (F_1, F_2, F_4, F_8, F_{10}, F_{12}) at 2939.61 cm^{-1} and Avicel PH 101 at the peak of 2887cm^{-1} are all assigned to C-H stretching vibration of the alkanes group (Lalduhsanga et al, 2013).This shows that the GH-MCC intensities are stronger than that of Acel PH 101. However these spectra all correspond to the peak reference of 2970-2850cm^{-1} respectively. Both untreated GH and the GH-MCC sizes (F_0, F_1, and F4) are in the region of 2345.52cm^{-1}, GH-MCC size (F_5) is at 2630.95 cm^{-1}are assigned to C=O=C carbondioxide group. These spectra correspond to the peak reference of 2349cm^{-1}. However this intensity did not appear in the following GH-MCC sizes (F_2, F_3, and F_6-F_{13}) and the standard Avicel PH 101 respectively.

A small peak appeared in the untreated GH (F_0) at 1627.97 cm^{-1}. It is also seen in (F_1-F_{13}) at 1643.41 cm^{-1} and in Avicel PH 101 at the peak of 1638cm^{-1} respectively which corresponds to the peak of reference 1662-1626cm^{-1}. These spectra are assigned to H_2O due to the absorbed water. However, the GH-MCC intensities are stronger than that of the Avicel PH 101. The peak value of (F_0) and GH- MCC (F_2, F_4, F_7, F_{10}-F_{12}) are at the peaks of 1504.53cm^{-1}, GH-MCC (F_5, F_6 and F_8) are at 1512.24 cm^{-1} corresponding to the peak of reference 1550-1500cm^{-1} respectively. These spectra are assigned to the N-O nitro compound. However, this peak value disappeared in GH-MCC sizes (F_1, F_3 and F_9) and Avicel PH 101. The peak of (F_0) is at 1419.66 cm^{-1}, GH-MCC (F_4-F_8, F_{10}-F_{12}) is at the peaks of 1411.94cm^{-1}. It is observed that (F_1 and F_{13}) both have two peaks values appearing in them which are 1411.94 and 1489.10 cm^{-1}. Similarly two peaks values also appear in (F_3 and F_9) at 1419.66 and 1496.81cm^{-1}. Avicel PH 101 is at the peak value of 1425cm^{-1}.These spectra are assigned to C-H_2 bending of the alkane group which corresponds to the peak reference of 1600-1400cm^{-1}respectively. Therefore, peaks of (F_0, F_4-F_8 and F_{10}-F_{12}) shows lower intensities compared to Avicel PH 101. Two peaks appeared in (F_0) which are 1373.36 and 1327.07cm^{-1}, GH-MCC (F_5) at 1319.35cm^{-1} and Avicel PH 101 at 1363cm^{-1} are assigned to the O-H bending of the alcohol group. These peaks values correspond to the peak reference of 1390-1310cm^{-1}. However, these spectra disappeared in the following GH-MCC sizes (F_1-F_4 and F_6-F_{13}).It is observed that Avicel PH 101 shows a higher intensity compared to these peak values. The untreated GH (F_0) shows strong absorption frequency at 1728.28 cm^{-1} but absence in (F_1-F_{13}) and in Avicel PH 101.While another strong absorption appears in (F_0) at the peak of 1257.63 cm^{-1} and GH-MCC sizes (F_1 and F_5) at the peaks of 1226.77 cm^{-1}and 1273.06cm^{-1} respectively, both corresponding to C=O stretching vibration of acetyl group from lignin and hemicelluloses and C-O out of plane stretching vibration of phenyl group in lignin (Rani et al., 2016). However these

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two bands gradually disappeared during the chemical treatment of (F_1-F_4, and F_6-F_{13}). The absence of these two bands in the GH-MCC indicates the removal of lignin and hemicelluloses. Also this particular peak was not observed in the case of Avicel PH 101 indicating the purity of the Avicel standard. Peak associated to the –C–O–C– stretch of the β -1,4- glycosidic linkage in cellulose were observed at 1141.90cm^{-1}for GH-MCC (F_1-F_4 and F_6-F_{12}), 1157.33cm^{-1} for (F_5), 1149.61cm^{-1} for (F_{13}) and 1154cm^{-1} for Avicel PH 101 but absence in (F_0). This peaks value corresponds to the peak reference of 1300-1100cm^{-1}. However, Avicel PH 101 has a stronger intensity compared to the GH-MCC. Figure 2 shows the FT-IR of the various sizes of the produced GH-MCC.

It can be concluded that the FT-IR of the GH-MCC F_6, F_9, F_{10} and F_{11} have better result.

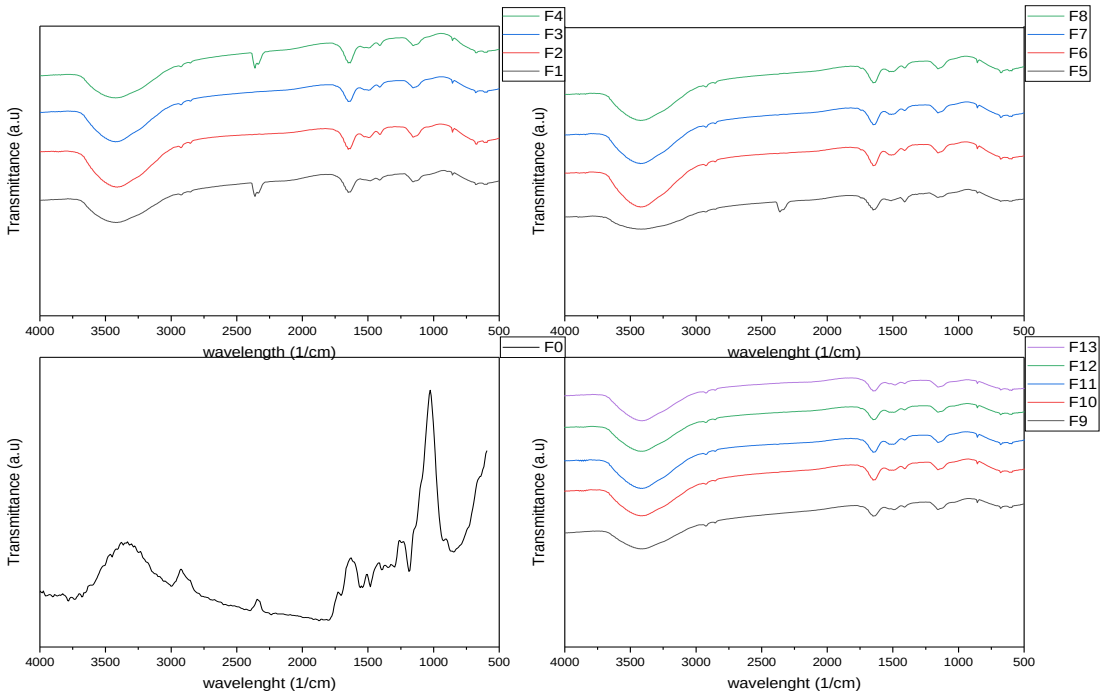

Figure 2: FTIR spectra of the various particle sizes of the microcrystalline cellulose produced (F_1-F_{13}) and the untreated groundnut husk (F_0)

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Table 3: The Physicochemical properties of Avicel PH 101, untreated - GH and GH-MCC

Powder property of Avicel PH101, untreated GH and GH-MCC

Sample ID	Particle Size (mm)	Bulk density (g/cm^3)	Tap density (g/cm^3)	True density (g/cm^3)	Carr's Index	Hausner ratio	Angle of repose	Percentage yield cellulose %	Porosity (%)	pH
F_1	3.35	0.144	0.163	0.831	11.35	1.13	33.69	32	80.37	6.0
F_2	2.80	0.144	0.163	0.844	11.53	1.13	32.30	32	80.64	5.6
F_3	2.36	0.144	0.163	0.866	11.53	1.13	38.24	32	81.19	5.6
F_4	2.00	0.163	0.185	0.875	11.89	1.14	34.23	28	78.85	5.7
F_5	1.70	0.163	0.217	0.877	24.89	1.33	36.70	32	75.24	5.6
F_6	1.40	0.163	0.185	0.874	11.89	1.14	32.57	44	78.84	5.8
F_7	1.19	0.163	0.185	0.881	11.89	1.14	32.37	28	78.99	5.6
F_8	1.18	0.144	0.163	0.880	11.53	1.13	37.46	32	81.48	5.7
F_9	1.00	0.163	0.185	0.877	11.89	1.14	32.72	52	78.90	5.8
F_{10}	0.560	0.217	0.260	0.865	16.53	1.21	33.25	36	69.94	5.8
F_{11}	0.500	0.163	0.185	0.880	11.89	1.14	39.11	56	78.97	5.8
F_{12}	0.425	0.163	0.185	0.877	11.89	1.14	33.68	48	78.91	5.8
F_{13}	0.212	0.325	0.433	0.878	25.02	1.33	36.90	40	50.69	5.8
AVICEL PH 101		0.32	0.45	1.56	23.73	1.31	31.61	-	79	5.0-7.5

Physicochemical properties

The physicochemical properties of the microcrystalline cellulose produced directly from the groundnut husk were discussed and presented in Table 3.0

Bulk density estimates the ability for material to flow. Table 1.0 shows that the bulk density of (F_1-F_3, F_8) are 0.144g/cm^3, (F_4- F_7, F_9, F_{11}-F_{12}) are 0.163g/cm^3, F_{13} is 0.325g/cm^3 and Avicel PH 101 is 0.32g/cm^3. However F_{13} has the highest bulk density and compared favourably with the standard. The bulk densities of the GH-MCC and the standard which is Avicel PH 101 are within the acceptable range which is 0.139–0.391g/cm^3.

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Tap density is the increased bulk density attained after mechanically tapping a container containing the powder sample. The tapped densities of the GH-MCC (F_1- F_3, F_8) are 0.163g/cm^3, (F_4, F_6, F_7, F_9, F_{11}, F_{12}) are 0.185g/cm^3, F_5 is 0.217g/cm^3, F_{10} is 0.260g/cm^3, F_{13} is 0.433g/cm^3 and Avicel PH 101 is 0.32 g/cm^3. Also F_{13} has the highest tapped density and compared favourably with the

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standard. The tapped densities of the GH-MCC and Avicel PH 101 are within the acceptable range which is 0.210–0.481g/cm3 (Handbook of pharmaceutical Excipient, 2006). In general, the higher the bulk and tapped densities, the better the potential for a material to flow and to re-arrange under compression (Okhamafe *et al*, 2012).

Tap density is the increased bulk density attained after mechanically tapping a container containing the powder sample. The tapped densities of the GH-MCC (F_1- F_3, F_8) are 0.163g/cm^3, (F_4, F_6, F_7, F_9, F_{11}, F_{12}) are 0.185g/cm^3, F_5 is 0.217g/cm^3, F_{10} is 0.260g/cm^3, F_{13} is 0.433g/cm^3 and Avicel PH 101 is 0.32 g/cm^3. Also F_{13} has the highest tapped density and compared favourably with the standard. The tapped densities of the GH-MCC and Avicel PH 101 are within the acceptable range which is 0.210–0.481g/cm3 (Raymond *et al.*, 2006). In general, the higher the bulk and tapped densities, the better the potential for a material to flow and to re-arrange under compression (Okhamafe *et al*, 2012).

Carr's compressibility index provides an estimate of powder compressibility while the Hausner index measures cohesion between particles. The values for both are inversely proportional to particle flow.

For Carr's index percentage, values in the ranges of 1-10, 11-15, 16-20, 21-25, 26-31, 32-37 and greater than 38 indicate excellent, good, fair, passable, poor, very poor and too poor flow properties of the material respectively (Okhamafe *et al*, 2012). GH-MCC (F_1,) is 11.35%, (F_2-F_3, F_8) are 11.53%, (F_4, F_6-F_7, F_9, and F11-F_{12}) are 11.89% and all these indicate good flowability. However (F_5) is 24.89% which indicate passable flow, (F_{10}) is 16.53 indicates a fair flow whereas (F_{13}) is 25.02% indicates a passable flowability. Avicel PH 101 has a 23.73% indicating a passable flowability.

For Hausner index, the values in the ranges of 1.00-1.11, 1.12- 1.18, 1.19-1.25, 1.26-1.34, 1.35-1.45, 1.46- 1.59 and greater than 1.60 indicate excellent, good, fair, passable, poor, very poor and very very poor flow properties of the material respectively. The GH-MCC of (F_1-F_3 and F_8) are 1.13 ratio indicating good flowability, (F_4, F_6-F_7, F_9 and F_{11}-F_{12}) are 1.14 also indicates good flowability. (F_5 and F_{13}) are 1.33 ratios which indicate a passable flowability, F10 is 1.21 ratio which indicate a fair flowability while Avicel PH 101 is 1.31 indicating a passable flowability. Therefore Avicel PH 101, GH-MCC (F_5 and F_{13}) both has a passable flow of property compared to (F_1-F_4 and F_6-F_{12}) which has a better flowability (Lalduhsanga *et al*, 2013).

The pH of the GH-MCC for F_1 is 6.0, (F_2-F_3, F_5 and F_7) are 5.6, F_4 is 5.7 and (F_9-F_{13}) are 5.8 respectively. Avicel PH 101 is within the range of pH 5.0- 7.5, therefore the pH of the GH-MCC from (F_1-F_{13}) is within the acceptable range (Handbook of pharmaceutical Excipient, 2006).

The angle of repose provides information on the powder flowability, it is a characteristic related to interparticulate friction or resistance to movement between particles. Angle of repose has a value in the ranges of 25-30, 31-35, 36-40, 41-45, 46-55, 56-65 and greater than 66 which indicate excellent, good, fair- aid not needed, passable – may hang up, poor- must agitate vibration, very poor and very very poor flow properties of the material respectively. GH-MCC (F_1, F_2, F_4, F_6, F_7,

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F_9, F_{10} and F_{12},) are 33.69, 32.30, 34.23, 32.57, 32.37, 32.72, 33.25 and 33.68 indicate good flow which conforms to the standard (Avicel PH 101) indicating a good flow also. However, (F_3, F_5, F_8, F_{11} and F_{13}) are 38.24, 36.70, 37.46, 39.11 and 36.90 also indicate a fair flow (USA Pharmacopeia)

The percentage yields of the groundnut husk microcrystalline cellulose (GH-MCC) for (F1-F3, F5, and F8) are 32% yield, (F_4 and F7) are 28%, F_6 is 44% yield, F_9 is 52% yield, F_{10} is 36% yield, F_{11} is 56% yield, F_{12} is 48% yield and F13 is 40% yield. Therefore (F_{11}, F_9, F_{12} and F_6) are the highest percentage yield 56%, 52%, 48% and 44% respectively.

The total porosity of a powder is made up of voids between the particles and pores within the particles. The results (Table 1.0) obtained for all the celluloses are similar indicating poly-sized particles and easily compressible powder during tableting. From F_1- F_3 and F_8 the percentage porosity is 80.37, 80.64, 81.19 and 81.48 slightly above Avicel PH 101 with 79% porosity. F_4 – F_7 are 78.85, 75.24, 78.84, 78.99 percentages, F_9 is 78.90%, F_{11} is 78.97% and F_{12} is 78.91% slightly below Avicel PH 101 and compares well with it. While F_{13} is 50.69% is smaller porosity.

X-ray diffraction Analysis (XRD)

Figure 4.2 shows the X-ray diffraction pattern of groundnut husk microcrystalline cellulose, (F_6, F_9, F_{11} and F_{12}). Samples F_6 exhibited a broad peak at $2\theta = 22.36^0$, which indicates the presence of crystalline material (Subramanian *et al*., 2005) and smaller prominent peaks at $2\theta = 16.57^0$ and 35.78^0. The peak at $2\theta = 22.36^0$ becomes more prominent and sharper as it reached MCC after various chemical treatments. Sample F_9 also exhibited a prominent crystalline peak at $2\theta = 22.38^0$ and lesser peaks at $2\theta = 16.64^0$ and 37.33^0. Sample F_{11} exhibited broad peak at $2\theta = 22.34^0$ and lesser peaks at $2\theta = 15.91^0$ and 38.07^0. Sample F_{12} shows a prominent peak at $2\theta = 22.32^0$ and lesser prominent peak at $2\theta = 14.16^0$ and 37.48^0 respectively. The two prominent peaks gave an insight on the crystalline nature of the GH-MCC, meaning that the GH- MCC is crystalline.The crystallinity index of GH-MCC samples "F_6, F_9, F_{11} and F_{12}" were calculated using the crystalline index (Bansal *et al.*, 2010), and the results were represented in Table 4.5. The crystallinity index of F_6, F_9, F_{11} and F_{12} were 88%, 89%, 91%, 92% respectively, indicating high crystallinity.

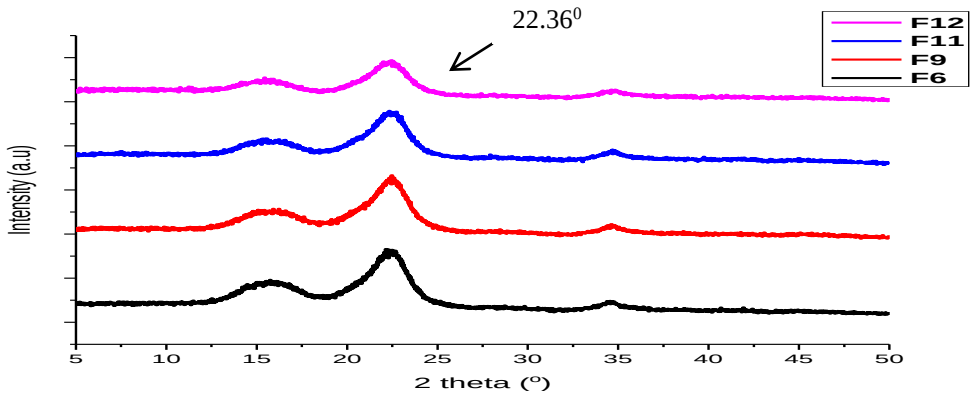

Figure 2: XRD pattern of GH-MCC of samples F_6, F_9, F_{11} and F_{12}

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Therefore, based on the FT-IR and the physicochemical analysis of the various sizes of the produced groundnut microcrystalline cellulose, (F_6, F_9, F_{11} and F_{12}) indicate favourable results that conform to the standard control, however, sample F_9 presents the optimal result,Table 4.0 shows the summary of these.

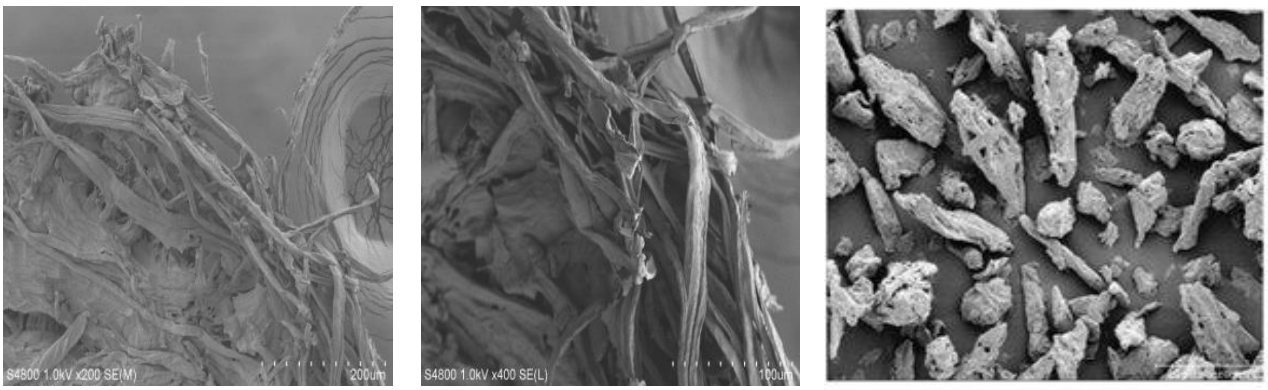
Table 4: Summary of the favourable selected sizes for GH-MCC

Powder property of Avicel PH101, untreated GH and treated GH

Sample ID	Particle Size (mm)	Bulk density (g/cm^3)	Tap density (g/cm^3)	True density (g/cm^3)	Carr's Index	Hausner ratio	Angle of repose	Percentage yield cellulose %	Porosity (%)	pH
F_{11}	0.500	0.163	0.185	0,880	11.89	1.14	39.11	56	78.84	5.8
F_9	1.00	0.163	0.185	0.877	11.89	1.14	32.72	52	78.90	5.8
F_{12}	0.425	0.163	0.185	0.878	11.89	1.14	33.68	48	78.91	5.8
F_6	1.40	0.163	0.185	0.874	11.89	1.14	32.57	44	78.84	5.8

The Scanning Electron Microscope

The morphology of GH-MCC powder after acid hydrolysis was studied through scanning electron micrograph and compared to the Avicel PH101. Fig.2 shows the SEM analysis of Sample F_9 with different magnification (Figure a, b, c and d). The particles size distribution of GH-MCC fibers is uneven and rod shaped compared to the flake shaped Avicel PH101 particles. Both the GH-MCC and Avicel PH101 were shown to exhibit rough surface. Figure 2 (a) shows magnification at 1.00k with the particle size of 50µm, figure (b) was magnified at 5.00k with the size of 10 µm, figure (c) at 200k magnification with the size of 200 µm, figure 2d is at 400k magnified with 100 µm size while Avicel PH 101 is magnified at 1000x with particle size of 50 µm.



(a) (b) (c)

Figure 2: SEM image of sample F_9

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Figure 2: SEM images of GH- MCC of Sample F_9 at different magnification (a) 200k x (a) 400k x and (c) Avicel PH 101

CONCLUSION

The specie and family of the groundnut husk used was Samnut 10 and *Arachis hypogaea*, the proximate analysis of the untreated groundnut husk indicated that hemicellulose was 9.48%, lignin 35.78%, alpha cellulose 44.16%, moisture content 2.3% and ash 6.1%. The Fourier Transform Infrared Spectroscopy (FTIR) confirmed the removal of hemicellulose and lignin from the GH-microcrystalline cellulose. The x-ray diffractometer (XRD) shows three prominent peak of crystallinity. The result indicated that the particle sizes selected for the production of microcrystalline cellulose from groundnut husk does not affect the yield, however, time, concentration and temperature does affect the yield. The alkali treatment at (500 ml, 0.5M) for 2 hours at 92.1^0C, hydrolysis using 20% (v/v) of nitric acid in ethanol in 500ml at 94^0C and bleaching at 95^0C for 15mins repeating trice shows that samples (F_6, F_9, F_{11} and F_{12}) indicate favourable results that conform to the standard control with the respective percentage yield 44%, 48%, 52% and 56%. However, the particle size of 1.00mm was found to be the optimal having a 52% yield of GH-MCC

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