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OPTIMIZATION OF PROCESS CONDITIONS IN THE MULTI – STAGE PULPING METHOD FOR THE PRODUCTION OF MICRO CRYSTALLINE CELLULOSE FROM GROUNDNUT HUSK

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ABSTRACT: *This study investigated the optimum processing conditions for obtaining the yield and purity of microcrystalline cellulose (MCC) powder obtained from groundnut husk (GH) using response surface methodology (RSM). Central composite design (CCD) was used to evaluate the optimum process conditions for producing MCC from alpha cellulose obtained from GH. The variables investigated for getting the optimum conditions were temperature (81.5 – 98.4^o C), hydrolysis time (0.5 – 2.5h) and the concentration ratio of the acid were (4 – 18.7g/ml). Groundnut husk of 1.00mm particle size was selected for the multi – stage pulping method using ethanol and nitric acid in ratio. The FTIR spectra indicated extensive removal of lignin and hemicellulose. The XRD shows that the material is crystalline in nature with two prominent peak, the pH is 6.0 and the physicochemical analysis confirms to the British Standard of Pharmacopeia. The Analysis of variance (ANOVA) showed that temperature was the most influential factor for hydrolyzing the amorphous sections of cellulose and improving purity. Under optimal conditions, the percentage yield and percentage purity of the alpha cellulose obtained were 73.79% and 88.08% respectively. The theoretical values for the percentage yield of the extracted alpha cellulose were close to the experimental one, resulting in small error percentages of 1.27% and 2.59%, respectively. Thus, it can be concluded that the RSM technique based on CCD design is suitable for optimizing the variables influencing the hydrolyzing of cellulose.*

KEY WORDS: Groundnut husk (GH), microcrystalline cellulose (MCC), groundnut husk (Arachis hypogaea)

INTRODUCTION

Cellulose is the most abundant organic compounds, a biodegradable and renewable structural plant polymer. It is a natural long chain polymer having a link with sugar ß-D-glucose which can be processed into microcrystalline cellulose (MCC), Nano crystalline cellulose (NCC) snd many more. It has a variety of applications in industries such as veterinary foods, wood and paper, fibers and clothes, cosmetic and pharmaceutical industries. Due to its advantage as thickening agents and stabilizers, binders, fillers, cellulose and cellulose based polymers have gained popularity and importance in pharmaceutical industries because of the new derivatives products and its application.

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Pure cellulose is available in different forms in the market with different mechanical and pharmaceutical properties whose grades influence their particle size and degree of crystallization. (Javad & Khosro, 2013)

MCC exhibits excellent properties in composite fabrication such as renewability, biodegradability, high surface area for bonding with resins (Zulaihani *et al*., 2016). The acid hydrolysis method is one of the main processes for the extraction of cellulose from cellulosic material. Due to the combination of ordered and disordered regions in cellulose chains, the disordered regions can be easily hydrolyzed by acid and the ordered parts are left as the remaining. Many researchers reported on using strong minerals acids such as sulphuric acid (H_2SO_4), hydrochloric acid (HCl) (Ohwoavworhu *et al*., 2005 & 2010; John *et al*., 2011; Chukwuemeka *et al*., 2012; Lalduhsanga *et al*., & Sirikalaya *et* al., 2013; Achor *et al*., & Li *et* al., 2014) and nitric acid in ratio to ethanol (Mohammed *et* al., 2015; Rani *et al*., 2016), to hydrolyze the amorphous regions. In this study, ethanol (C_2H_5OH) and nitric acid (HNO_3) in ratios has been chosen as an alternative for this work because it gives better and higher; yield, purity and crystallinity of cellulose as reported by (Zulaihani *et al*., 2016).

METHODOLOGY

The methodology used for the production of microcrystalline cellulose is discussed as follows

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 as presented in Table 1. The chemicals used were NaOH (BDH England), Ethanol, Nitric Acid, NaCLO.

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 2.

Production of Alpha Cellulose

The alpha cellulose were measured for 19 g and refluxed with a mixture of ethanol (C_2H_5OH) and nitric acid (HNO_3) in ratios within the concentration range of (4-18.7g/ml), a temperature range of (81.5 - 98.4^{0}C) and the time were (0.5 – 2.5h) in a thermostatic water bath. This treatment was carried out thrice as the colour changed from brown to orange in successive steps. The mixture 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

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oven dried at 60^0C to for 8hrs. The factors obtained from the Central Composite Design (CCD) were presented in Table 3. The yield of the extracted cellulose were weight and recorded respectively, the dried microcrystalline cellulose was stored in plastic containers. Photographs at different stages of the chemical treatments

Percentage Yield

Percentage yield was calculated using the following equation:

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

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

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

Powder Porosity

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

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

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).

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

SEM

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

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)

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$$CI = \frac{I_{002} - I_{AM}}{I_{002}} X\ 10 \ldots \ldots (7)$$

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

Groundnut Husk Identification

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*

Proximate Analysis

The Chemical composition of the untreated groundnut husk was investigated using a gravimetric method to determine the proximate properties. Table 2.0 presents the results obtained.

Table 2: Proximate Properties of Untreated Groundnut Husk

S/No.	Parameter	Value (%)
1	Ash	6.1
2	Hemicellulose	9.48
3	Lignin	35.78
4	Alpha Cellulose	44.16
5	Moisture Content	2.3

Actual Design of the Experiment

Research Surface Methodology was employed to maximize the production of alpha cellulose from groundnut husk by the optimization of the process conditions. The interaction among process variables was determined by statistical techniques. Central composite design was employed to investigate the effect of critical parameters i.e; temperature, time, and concentration of the process on the groundnut husk. The three variables and their value range were selected based on preliminary studied. Three dimensional surface were applied to investigate and validate the influence on process variables on the treatment of alpha cellulose. The reaction time (1-2h), temperature (80 - 98^{0}C) and sodium hydroxide concentration (0.25 - 0.90 g/ml) were the input variables as shown in Table 3.

Table 3: Summary of Factors input and the Responses of Upper and Lower Limits for the Multistage Pulping

Constraints						
Name	**Goal**	**Lower Limit**	**Upper Limit**	**Lower Weight**	**Upper Weight**	**Importance**
A:temp (^{0}C)	is in range	81.5	98.4	1	1	3
B:time (h)	is in range	0.5	2.5	1	1	3
C:ethanol: nitric	is in range	4	18.7	1	1	3

The multi stage pulping method of the groundnut husk using hydrolysis treatment was carried out using Central Composite Design (CCD) and the responses of percentage yield and purity of the microcrystalline cellulose were presented in Table 4.

Table 4: The Actual Design of the Experiment for the multi stage pulping and the results obtained as the Response of Percentage Yield and Percentage Purity of the Microcrystalline Cellulose

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Std	Run	Factor 1 A: Temperature (^{0}C)	Factor 2 B: Time (hr)	Factor 3 C: ethanol:nitric acid ratio	Response 1 Percentage Yield of Cellulose (%)	Response 2 Percentage Purity of Cellulose (%)
8	1	95.00	2.50	15.00	62.89	85.01
10	2	98.41	1.75	9.50	57.95	77.84
4	3	95.00	2.50	4.00	53.79	70.84
1	4	85.00	1.00	4.00	62.47	75.27
20	5	90.00	1.75	9.50	51.16	73.11
11	6	90.00	0.48	9.50	51.00	89.71
12	7	90.00	3.01	9.50	56.37	85.25
9	8	81.59	1.75	9.50	62.05	72.62
17	9	90.00	1.75	9.50	50.26	80.61
16	10	90.00	1.75	9.50	53.63	72.01
3	11	85.00	2.50	4.00	53.73	83.47
14	12	90.00	1.75	18.75	52.95	78.53
7	13	85.00	2.50	15.00	53.58	82.99
15	14	90.00	1.75	9.50	51.42	71.99
13	15	90.00	1.75	0.25	47.05	66.42
19	16	90.00	1.75	9.50	54.79	70.78
5	17	85.00	1.00	15.00	61.00	80.94
18	18	90.00	1.75	9.50	55.68	72.07
6	19	95.00	1.00	15.00	57.95	86.94
2	20	95.00	1.00	4.00	43.68	71.00

Percentage Yield of the Microcrystalline Cellulose

The percentage yield of the microcrystalline cellulose obtained after hydrolysis process was obtained using equation 1. The analysis of variance (ANOVA) shows that the p-value is 0.0007 indicating a significant model. This means that the independent variables; time, concentration and temperature has a considerable effect on the response in the term of the percentage yield. The R-square is 0.8988 also indicating that a statistical model can explain 89.88 % of the variability of the response. While the percentage coefficient of variation (CV) is 4.07 shows an adequate precision and reliability of the experiment. Table 5 present the summary of ANOVA.

Table 5: Statistical ANOVA Analysis of Response for the Percentage Yield of Cellulose

ANOVA for Response Surface Quadratic model

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	440.32	9	48.92	9.87	0.0007	Significant
A-temp	27.46	1	27.46	5.54	0.0404	
B-time	4.59	1	4.59	0.93	0.3585	
C-ethanol: nitric	73.45	1	73.45	14.81	0.0032	
AB	121.76	1	121.76	24.55	0.0006	
AC	78.06	1	78.06	15.74	0.0027	
BC	1.85	1	1.85	0.37	0.5547	
A^2	116.84	1	116.84	23.56	0.0007	
B^2	5.44	1	5.44	1.10	0.3194	
C^2	6.82	1	6.82	1.38	0.2679	
Residual	49.59	10	4.96			
Lack of Fit	25.60	5	5.12	1.07	0.4723	not significant
Pure Error	23.99	5	4.80			
Cor Total	489.91	19				

In this case A, C, AB, AC, A^2 are significant model terms.

Std.Dev--------- 2.23	R-Square ---------- 0.8988
Mean --------- 54.67	Adj-Square ---------- 0.8077
C.V. % --------- 4.07	Pred R-Square ---------- 0.5163
PRESS --------- 236.95	Adeq Precision -------- 12.605

The Model Equation

The second order polynomial equation expresses the predicted percentage yield of the cellulose. Equation 8 shows the predicted response of the percentage yield of the cellulose given as 52.78 %.

Percentage Yield of MCC (R1) = 52.78

$$-1.42A + 0.58B + 2.32C + 3.90AB + 3.12AC - 0.48BC + 2.85A^2 + 0.61B^2 - 0.69C^2 \quad (8)$$

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Percentage Purity of Cellulose

The percentage purity of the cellulose obtained after hydrolysis process were obtained by using proximate analysis method. The p-value is 0.0026 indicating a significant model, therefore, the independent variables; time, concentration and temperature has effect on the response in the term of the percentage purity. The R- square is 0.8644 which means that the statistical model can explain 86.44 % of the variability of the response. The percentage coefficient of variation (CV) is 4.35 indicating adequate precision and reliability of the experiment. Table 6 present the summary of ANOVA

Table 6: Statistical ANOVA Analysis of Response for the Percentage Purity of Cellulose

ANOVA for Response Surface Quadratic model

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
Model	722.07	9	80.23	7.08	0.0026	significant
A-temp	7.476E-004	1	7.476E-004	6.599E-005	0.9937	
B-time	0.032	1	0.032	2.809E-003	0.9588	
C-ethanol: nitric	226.90	1	226.90	20.03	0.0012	
AB	19.03	1	19.03	1.68	0.2240	
AC	77.63	1	77.63	6.85	0.0257	
BC	7.84	1	7.84	0.69	0.4249	
A^2	9.66	1	9.66	0.85	0.3775	
B^2	382.19	1	382.19	33.74	0.0002	
C^2	0.35	1	0.35	0.031	0.8644	
Residual	113.29	10	11.33			
Lack of Fit	48.67	5	9.73	0.75	0.6183	not significant
Pure Error	64.62	5	12.92			
Cor Total	835.36	19				

Std.Dev--------- 3.37 R-Square ---------- 0.8644
Mean --------- 77.37 Adj-Square ---------- 0.7423
C.V. % --------- 4.35 Pred R-Square ---------- 0.4417
PRESS --------- 466.37 Adeq Precision -------- 9.570
In this case C, AC, B^2 are significant model terms.

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The Model Equation

The model equation shows the predicted response of the percentage purity of the cellulose to be 73.40 % in Equation 9.

Percentage Purity of MCC (R2) = 73.40

$-7.399E - 003A + 0.048B + 4.08C - 1.54AB + 3.12AC - 0.99BC + 0.82A^2 + 5.15B^2 - 0.16C^2$..... (9)

PLOTS ON PERCENTAGE YIELD AND PERCENTAGE PURITY OF THE CELLULOSE

The Perturbation Plot

The Perturbation plot in Figure 1 shows that at Point A (temperature) and points B (time) increases while C (concentration) decreases. However, at deviation from reference point 0.00 (coded units) both Point A, B and C coincided to give optimal percentage yield of 52.60% of the cellulose which is slightly below the predicted response for the percentage yield at 52.78%. The error different between the experimented and predicted response is 0.18 %. Similarly, Point A (temperature) and points B (time) increases while C (concentration) decreases. The optimal percentage purity of the cellulose is 73.20% as shown in Figure 1 while the predicted response was 73.40 % having the error different between the experimented and predicted response as 0.20 %.

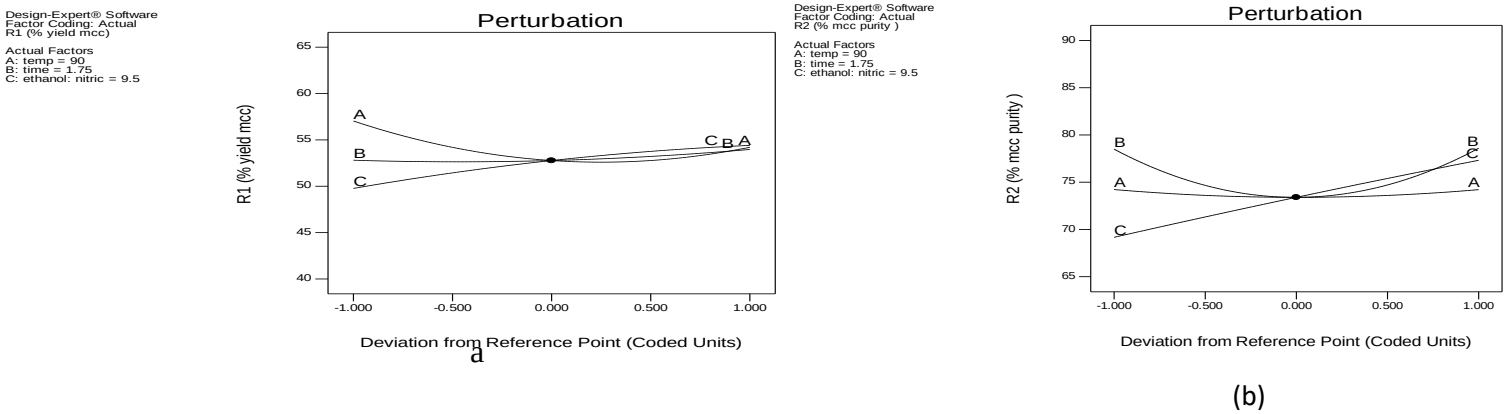

Figure 1: Perturbation Plot showing the effect of time, temperature and concentration on the percentage yield (a) and purity (b) of cell.

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The 3-Dimensional Design

The 3-Dimensional plot in Figure 2 shows the response surface of the percentage yield and purity of cellulose. The percentage of cellulose is the function of temperature and time. The optimal percentage yield of the cellulose is greatly influenced by temperature. Therefore, increase in temperature greatly increase the percentage yield of the cellulose at an increase time. However, in Figure (2b), the percentage purity of cellulose is affected by the concentration, as concentration is increased purity of cellulose increases with respect to temperature.

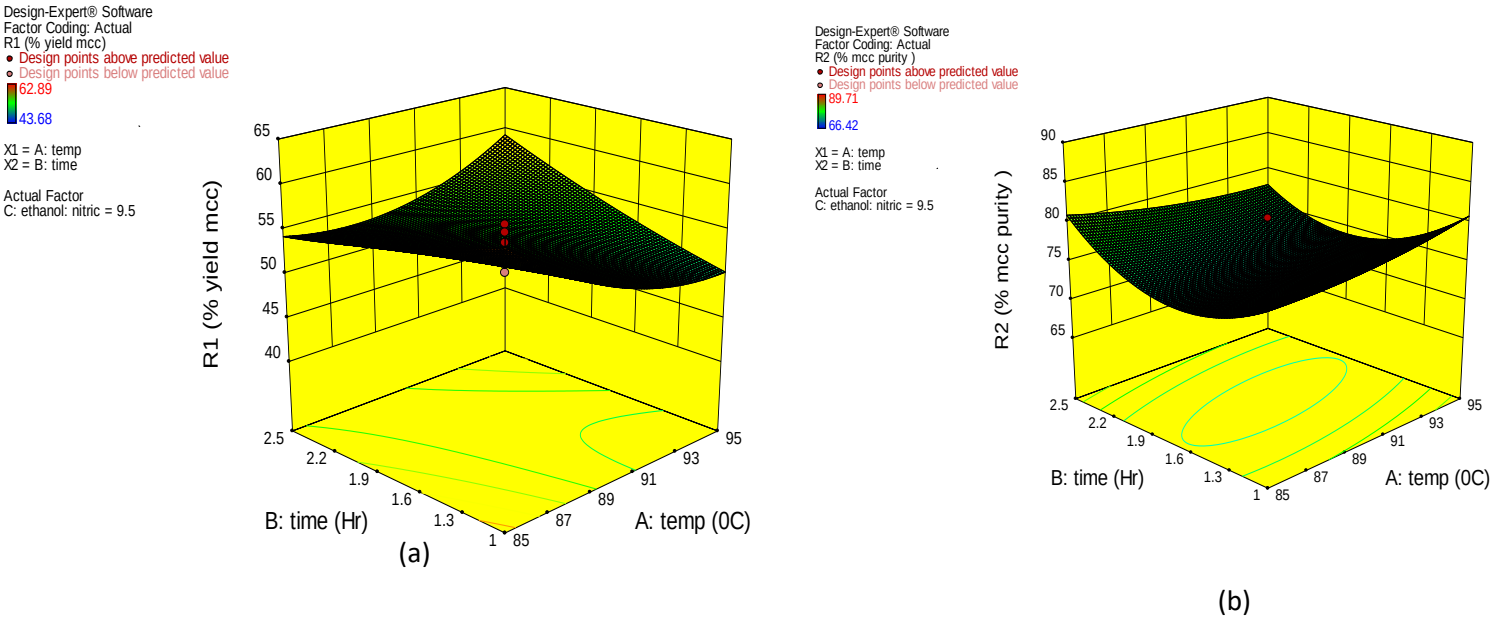

Figure 2: The 3-Dimensional Plot of percentage yield (a) and purity (b) of the cellulose

SOLUTION OF THE OPTIMISATION AND THE VALIDATION OF THE Microcrystalline cellulose

Design expert was used to compute the responses obtained, analyzed and the solution were present as the optimized solutions as shown in Table 7. The optimized results all shows that the desirability is 1.0 (100%). Although number 1 of the solutions gives the most favourable results having a higher percentages in the yield and purity and were considered for validation after compared with the other solutions. Therefore, the process parameters at temperature 98.36^{0}C, time at 2.45 hr and

ethanol to nitric acid ratio at 18.22 were used to validate sample number 1 and the outcome are recorded in Table 8.

Table 7: Solutions of the Optimization of the Microcrystalline Cellulose.

Number	Temperature (^{0}C)	Time (h)	Ethanol: Nitric	Response 1 Cellulose Yield (%)	Response 2 Cellulose Purity (%)	Desirability	
1	98.36	2.45	18.22	75.06	90.67	1	Selected
2	98.39	2.39	18.43	74.72	90.59	1	
3	98.38	2.50	17.34	74.81	89.82	1	
4	98.19	2.47	17.72	74.24	89.91	1	
5	98.15	2.46	17.89	74.15	90.01	1	
6	98.03	2.38	18.66	73.64	90.38	1	
7	98.28	2.44	17.67	74.18	89.71	1	
8	98.03	2.41	18.31	73.63	90.09	1	
9	97.74	2.45	18.52	73.23	90.28	1	

The validated sample (V_0) that was obtained under optimal conditions, at the temperature of 98.36^{0}C, time at 2.45h and ethanol to nitric ratio at 18.22 were recorded in Table 8. The optimal percentage yield and percentage purity of the sample (V_0) obtained were 73.79 % and 88.08 % cellulose.

Table 8: The validated Solution of the optimized Cellulose

Sample ID	Temperature (^{0}C)	Time (h)	Ethanol: Nitric	Response 1 Cellulose Yield (%)	Response 2 Cellulose Purity (%)	Desirability	
V_0	98.36	2.45	18.22	73.79	88.08	1	Selected

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THE PHYSICAL AND CHEMICAL CHARACTERISTICS OF THE CELLULOSE

The optimal percentage yield and percentage purity of the cellulose obtained from the validated sample (V_0) was 73.79% and 88.08% respectively. The bulk density, tapped density and true density were 0.216, 0.24 and 0.84 gcm^{-3} respectively. These values fell within the acceptable range as compare to standard as shown in Table 4.4 (*Chukwuemeka et al.*, 2012). The Carr's compressibility index was 10% and Hausner ratio was 1.11 indicating an excellent flow properties. The powder porosity was 71.32% also indicating that the powder can easily be compressible during tableting as compared to (Lalduhsanga *et al.,* 2014.) who stated that the powder porosity in his work for Avicek PH 101 was 66.9 % and Rawnal (Dendrocalamus longispathus) a bamboo fiber (R-MCC) was 68.22 %. This is slightly below the GH–MCC.powder porosity. The Angle of Repose was 32^0 which indicated a good flow characteristics and compared well with the standard control (Chukwuemeka *et al.,* 2012). Table 9 presents the summary of the physical and chemical properties of the cellulose.

Table 9: Summary of the Optimal Process Parameters for the Production of Microcrystalline from Groundnut husk cellulose

PARAMETERS	GROUNDNUT HUSK CELLULOSE
Percentage Yield (%)	73.79
Percentage Purity (%)	88.08
pH	6.0
Bulk density (gcm^{-3})	0.216
Tapped density (gcm^{-3})	0.24
True density (gcm^{-3})	0.84
Carr's Compressibility index (%)	10
Hausner ratio	1.11
Powder porosity (%)	71.32
Angle of repose (°)	32
Particle Size (µm)	300

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THE XRAY DIFFRACTOMETER OF THE OPTIMAL GROUNDNUT HUSK MICROCRYSTALLINE CELLULOSE

The X-ray diffraction pattern of the groundnut husk microcrystalline cellulose sample (V_0) is shown in Figure 3. Samples (V_0) exhibit a broad high peak of 2θ at 23.10^0, which indicates the presence of crystalline material (Subramanian *et al.*, 2005) and small less prominent peaks of 2θ at 15.62^0 and 34.64^0 respectively. The research works of (Li *et a.*, 2014) reported a similar XRD results in his publication where his prominent peak at 2θ was 22.50^0 and also showed smaller peaks. This explain that the glycosidic bond from amorphous regions degraded and cleave in the hydrolysis process as reported by (Li *et a.*, 2014), while the well-organized region of the crystalline changed a little and increasing the crystallinity. Therefore the sample (V_0) is crystalline in nature

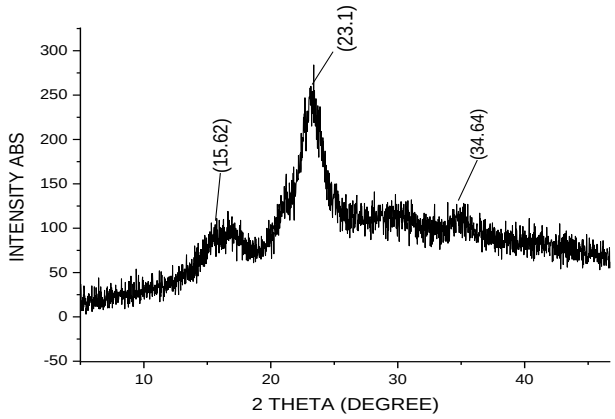

Figure 3: XRD Pattern of the validated Sample V_0 of the GH- MCC

THE SPECTRAL INTERPRETATION OF THE OPTIMAL GROUNDNUT HUSK MICROCRYSTALLINE CELLULOSE

The FT-IR spectrum of the optimal groundnut husk microcrystalline cellulose (V_0) has a peak of 3402.54cm^{-1}, indicating an intermolecular O—H stretching vibration band of the alcohol group for GH-MCC and showing a higher bond than the Avicel PH 101 but compares well as shown in Figure 4 and the regions of spectra bands recorded in Table 4.20. At this region two peaks are visible, the peak at 2924.18 cm^{-1} and 2870.17 cm^{-1} (V_0) is assigned to C-H stretching vibration of the alkanes group (Lalduhsanga *et al.*, 2013).The intensity of Avicel PH 101 was 2887cm^{-1} which corresponded to the peak reference of 2970-2850cm^{-1} respectively and compared well.

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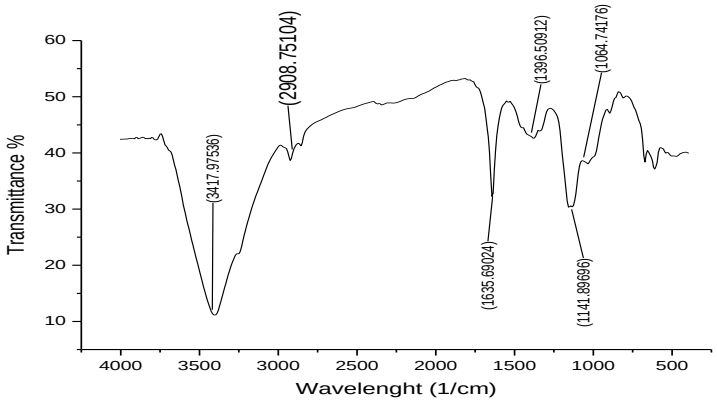

Figure 4: FTIR spectra of the optimal sample of groundnut husk microcrystalline cellulose produced (V_0)

There are two regions that appeared in the optimal sample which are of 2345.52cm^{-1}and 2268.36cm^{-1}, they 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 Avicel PH 101 respectively.

At the peak region of 1643.41 cm^{-1} the spectra is assigned to H_2O functional group while Avicel PH 101 is at 1638cm^{-1} respectively which corresponds to the peak of reference 1662-1626cm^{-1}. However, the GH-MCC intensity is stronger than Avicel PH 101.

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Table 10: List of Fourier Transform Infrared spectroscopy absorptions by frequency regions of Avicel 101, optimal Groundnut Husk MCC

Peak of Reference ($Cm^{-}1$)	3600-3200	2970–2850	2349	1750-1715	1662-1626	1550-1500	1600-1400	1390-1310	1275-1200	1300-1100
Functional group	O-H (alcohol)	C-H (alkanes)	O=C=O (carbondioxide)	C=O (β-unsaturated ester,liginin)	H_2O (water)	N-O (nitro compound)	C-H_2 (alkane)	O-H (alcohol)	C-O (alkyl ether)	C-O-C
Type of Vibration	Stretch, H-bonded	Stretch	Stretch	Stretch		Stretch	Stretch	Bending	Stretch	Stretch
Intensity	Strong	Strong	Strong	Strong	Medium	Strong	Weak	Medium	Strong	Medium
Sample ID										
V_B	3402.54	2924.18 2870.17	2345.52 2268.36		1643.41			1381.08		1141.9
AVICEL PH 101	3275	2887	-	-	1638		1425	1363	--	1154

Key: V_B = Validated (optimal) MCC groundnut husk, AVICEL PH 101= standard MCC

At 1381.08cm^{-1} the functional group is alcohol with an O-H bending, this slightly above Avicel PH 101 which is at 1363cm^{-1} but compares well. These peaks values correspond to the peak reference of 1390-1310cm^{-1} (Rani *et al.*, 2016).

However, the absorption frequency at 1750 - 1715 cm^{-1} and 1275 - 1200 cm^{-1} are absence in (V_0) and in Avicel PH 101. These bonds 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 two bands gradually disappeared during the chemical treatment using acid hydrolysis method. The absence of these two bands in the GH-MCC indicates the removal of lignin and hemicelluloses.

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 (V_0) and 1154cm^{-1} for Avicel PH 101. However, Avicel PH 101 has a stronger intensity compared to the GH-MCC. This peaks value corresponds to the peak reference of 1300-1100cm^{-1}(Rani *et al.*, 2016).

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