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# Characterization and chemical modification of cellulose from some selected local varieties of *Oryza sativa* chaff in Northern Nigeria

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ABSTRACT

**Background**: This study was targeted at extracting and production of microcrystalline cellulose from some local varieties of rice chaff being one of the most versatile excipient use in pharmaceutical tableting.

Methods: Microcrystalline cellulose was derived from locally sourced rice chaffs in Taraba, Kano, Kaduna, and Kogi-Nigeria by standard extraction procedures from literature with modifications. Ethanol (95 %) was utilized in solvent extraction for 6 h to remove wax and resins leaving the fibrous material. Alkaline hydrolysis with 17.5 % NaOH was employed to remove hemicelluloses leaving α-cellulose with lignin followed by exposure to 200 mL of glacial acetic acid to neutralize excess of the alkali. This was then delignified and bleached by treatment with sodium hypochlorite solution. The α-cellulose was modified to microcrystalline cellulose by mineral acid (HCL) at 105°c for 15 mins leaving microcrystalline cellulose after washing with water. The final isolated product was characterized using Fourier Transform Infrared Spectroscopy (FTIR).

**Results**: The microcrystalline cellulose content of the four rice chaffs was 18, 15, 21, 11 % respectively. This compares reasonably with the findings of other previous studies, vis. 19 % for *Oryza sativa* sippi. From the extraction sets of studies, *Oryza sativa* sippi gave the highest content of microcrystalline cellulose. Fourier Transform Infra-red (FT-IR) spectra confirm the presence of characteristic functional groups and indicate the purity of the sample. Melting was observed at temperature 249°C, 231°C, 244°C, 203°C and 255°C with samples A, B, C, D and the reference respectively.

**Conclusions**: This study demonstrates that the geographical origin of the materials significantly influences their potential yields and may contribute to variations in specific physicochemical properties.

## 1. Introduction

Rice chaff (RC) is a major byproduct of rice mill, accounting for about 20 % of the paddy weight. RC has attracted attention as value added material towards waste utilization and cost reduction in both domestic and industrial processing.[1] It is widely available in some developed countries like China, India and Vietnam contributing up to 20 % of global rice production as byproduct of rice milling.[2] Traditionally, rice husk is seen as agricultural waste and its disposal is a major environmental concern [3] but as a result of its distinct composition of large amount of cellulose, lignin, hemicellulose and silica, it can be processed to valuable products.[4] The high silica content in chaff makes it suitable for use in adsorption and filtration applications.[5] Cellulose is a composite material found in a wide range of species such as wood, grass, bacteria and green algae. Cellulose arguably, one of the most abundant organic compound in the world today which mostly produced by plants.[6] However, the α-cellulose content which is essential for certain applications such as nitrocellulose production varies widely among these sources.[7] Other materials apart from cellulose includes

(besides α-cellulose) hemicelluloses, lignin, pectin, wax and a variety of resins.[8]

Cellulose is mostly degraded by enzyme group known as cellulase which is produced by several species fungi species.[9] Researchers have been working on isolation, characterization, estimation and application of cellulase from fungal and bacterial sources.[10] Despite the remarkable number of fungi capable of producing cellulase enzymes, only a few have been thoroughly investigated, primarily due to their extracellular mode of production.[11]

## Materials and Methods

### Materials

The Four (4) varieties of rice chaff were obtained from some local rice mills across the Northern states of Nigeria; *Oryza sativa* Nerica, from Takum, Taraba State (7.2667$^{O}$ N, 9.9833$^{0}$ E), *Oryza sativa* ITA from Kura, Kano state (11.7714$^{0}$ N, 84303$^{0}$ E), *Oryza sativa* sippi from Kaduna State (10.468$^{0}$ N, 7.421$^{0}$ E) and *Oryza sativa* Wit4 from Ejiba Kogi State (8.3033$^{0}$ N, 5.6392$^{0}$ E), and all varieties belonging to the plant family 'Poaceae'.

Chemicals used in this study were sourced from various companies and locations. The materials used include sodium hydroxide (Avondales Laboratory England), Hydrochloric acid and acetic acid (Sigma-Aldrich, Germany), Toulene, Hypochlorite solution Ethanol (BDH Chemicals Ltd Poole, England) microcrystalline cellulose Avicel PH-102 (AcrosOrganics, USA). Identification was done at the Herbaria, Department of Botany, Ahmadu Bello University, Zaria and Institute of Agricultural Research and Development, Samaru Zaria with tagged number of ZAR-212348(1-2) and IARD-1890(1-2).

### Method

#### Pre-treatment of rice chaff

A 300 g portion of each of the *Oryza sativa* chaffs (Nerica, ITA, Sippi, WIT4 150) was cut into smaller pieces and cleaned with 500 mL distilled water to remove dust and dirt adhering to them, followed by drying in an oven at 85$^{O}$C. The dried small samples were further grounded to fine powder using electrical mill to increase the surface area.[11]

#### *Dewaxing*

Extractable materials like: fats and oil, waxes and resins were removed by subjecting each of the powder sample to ethanol extraction (95 %) in Soxhlet extractor at 60$^{O}$C temperature for 6 hours (h).The extract residue (cellulose) was dried at room temperature for 16 h and weighed afterwards.[12]

#### *Delignification and Alkaline Hydrolysis*

Following the ethanol extraction procedure, the solid residue from each sample was transferred to a 250-ml flat-bottom flask. Subsequently, 1000 mL of 7.5 % sodium hydroxide (NaOH) solution was added, and the mixture was stirred continuously for 1 hour. Excess water was removed via fractional distillation, after which the residue was filtered and washed thoroughly with 95% ethanol. This was followed by a drying step. The dried sample was then treated with glacial acetic acid and 15 mL of hypochlorite solution at 75$^{O}$C for 2 h. The reaction mixture was allowed to cool and equilibrate for 24hr, after which the lignin-containing liquid was carefully decanted. The decanted liquid was transferred into a 250 mL flask, and 150 mL cold ice water was added. The residue was washed with 500 mL of ethanol and 1000 mL of distilled water. The sample was then dried in an oven at 65 $^{O}$C for 24 h, and the yield obtained was recorded for all 4 varieties.

#### Modification

The α-cellulose was modified to microcrystalline cellulose by mineral acid (HCl) at 105$^{O}$C for 15 min. The resulting microcrystalline cellulose was obtained after thorough washing with distilled water.

#### Percentage yield

The resulting cellulose from each variety was weighed, and the percentage yield was determined based on the initial sample (Wo) and the final cellulose (W1). The percentage yield Y was then calculated using the formula.

$$Y = \frac{WI}{WO} X\ 100\% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . Equation1$$

#### Fourier transform infrared (FTIR) spectroscopy

FTIR analysis was carried out using Perkin Elmer FTIR spectrophotometer 1650 using a scanning ranges of 4000.0cm$^{-1}$ to 400 cm$^{-1}$. A 100 mg portion of each sample was dissolved in a suitable solvent, placed in a sample holder, and subsequently analysed using infrared spectroscopy.

#### Melting point determination

A 0.1 g portion of the extracted sample was dried and placed into a capillary tube with an internal diameter of 0.1-0.2 mm. With the aid of a thermometer attached to the capillary tube, the sample was heated on a metal block with a gradual

increase in temperature. The thermometer was carefully monitored, and the temperature at which melting occurred was recorded.

**Results**

***Percentage yield***

The respective yields of the samples were determined, and the data were summarized in Table 1. Microcrystalline cellulose was successfully extracted from four samples (A, B, C, and D) of rice chaff.

**Table 1. Percentage yield of microcrystalline-cellulose produced from different varieties of rice chaff**

| Species | Percentage yield (%)<br>Microcrystalline cellulose |
|---|---|
| Sample A | 18 |
| Sample B | 15 |
| Sample C | 21 |
| Sample D | 11 |

**FTIR**

Figures 1 to 5 showed the spectra recorded for the four (4) sample, along with Avicel® PH-102, which was used as the standard.

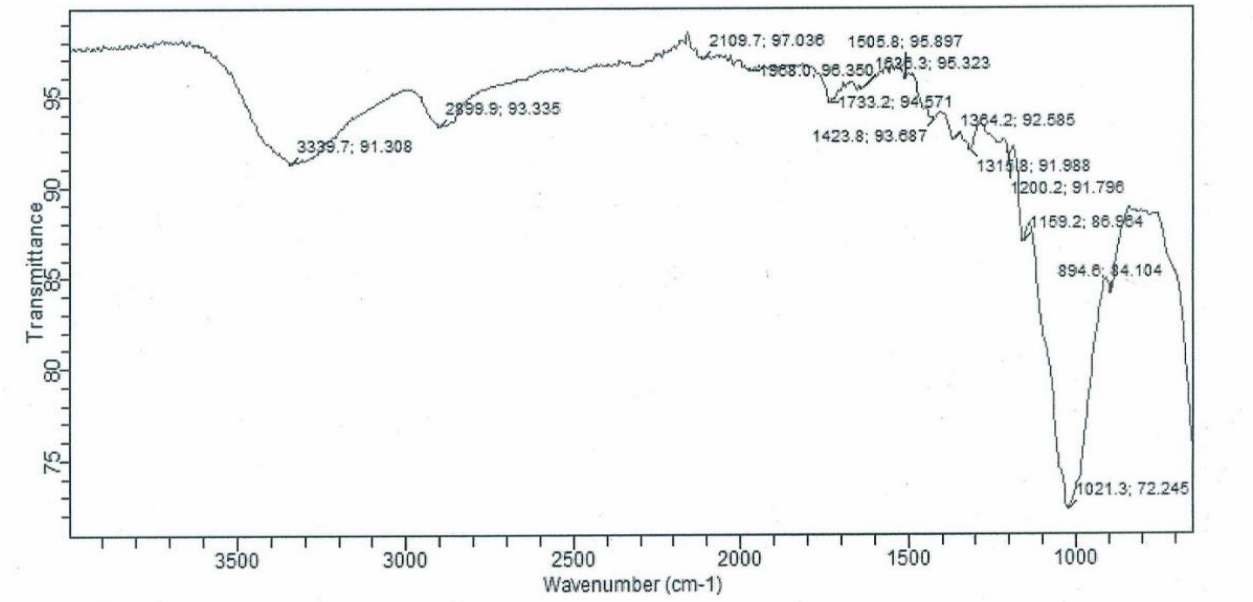


**Figure 1: FTIR Spectrum for Sample A**

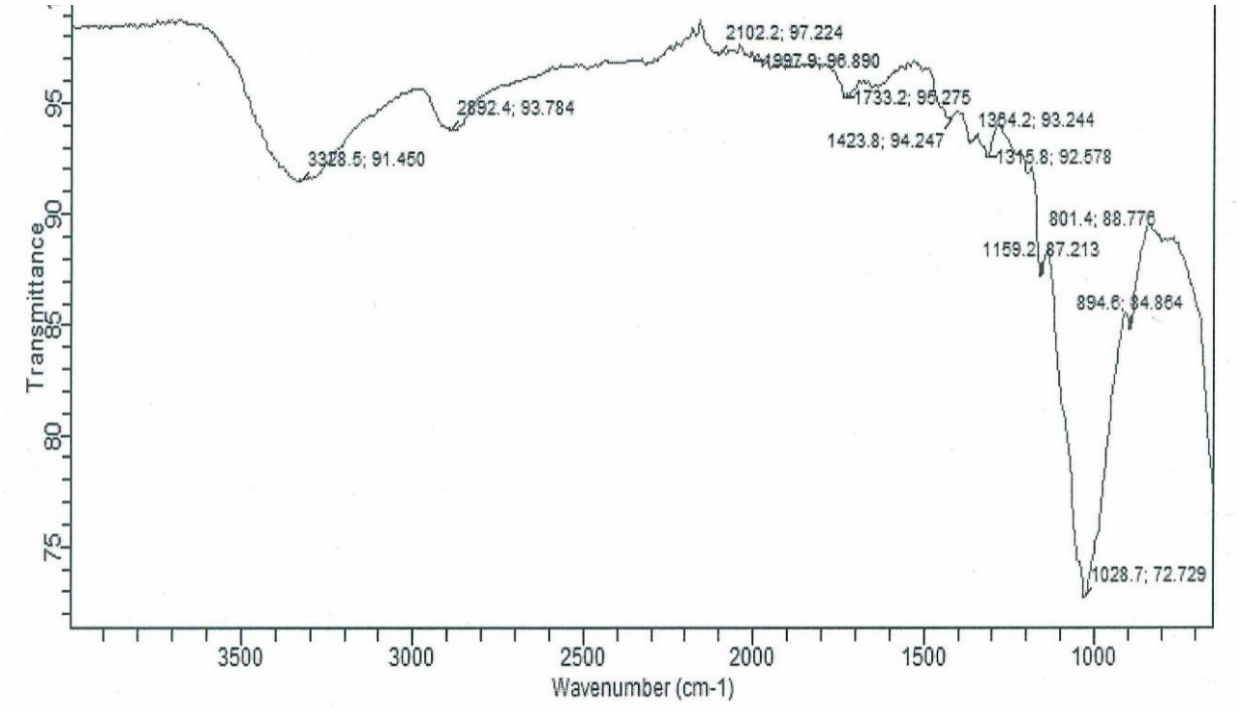


**Figure 2: FTIR Spectra for Sample B**

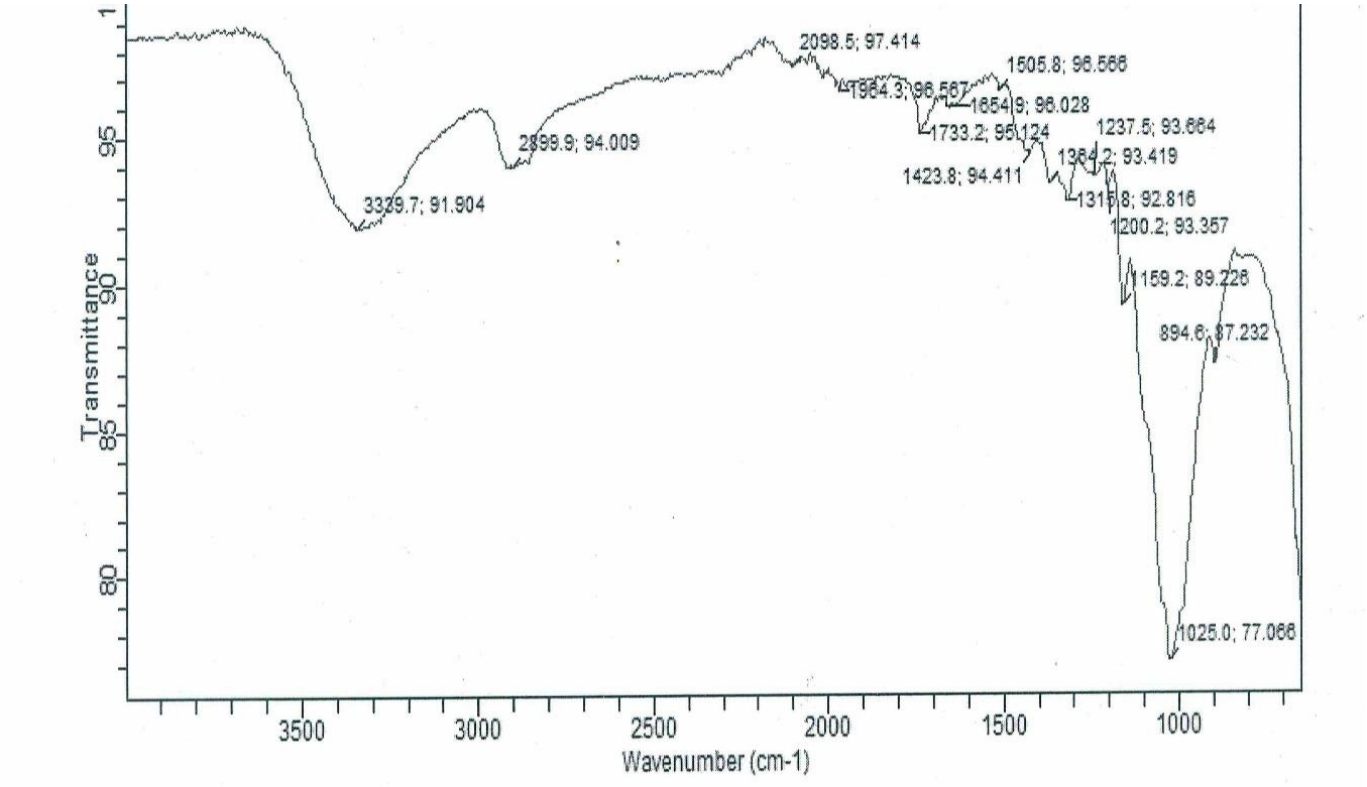


**Figure 3: FTIR Spectrum for Sample C**

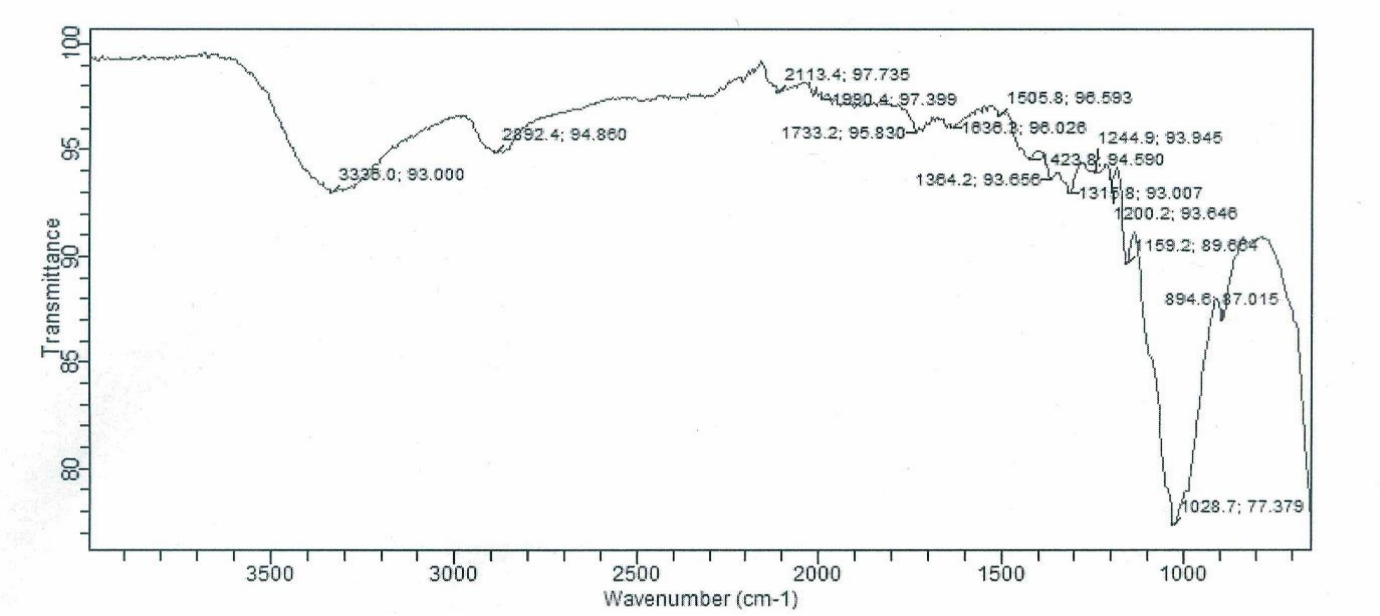


**Figure 4: FTIR Spectrum for Sample D**

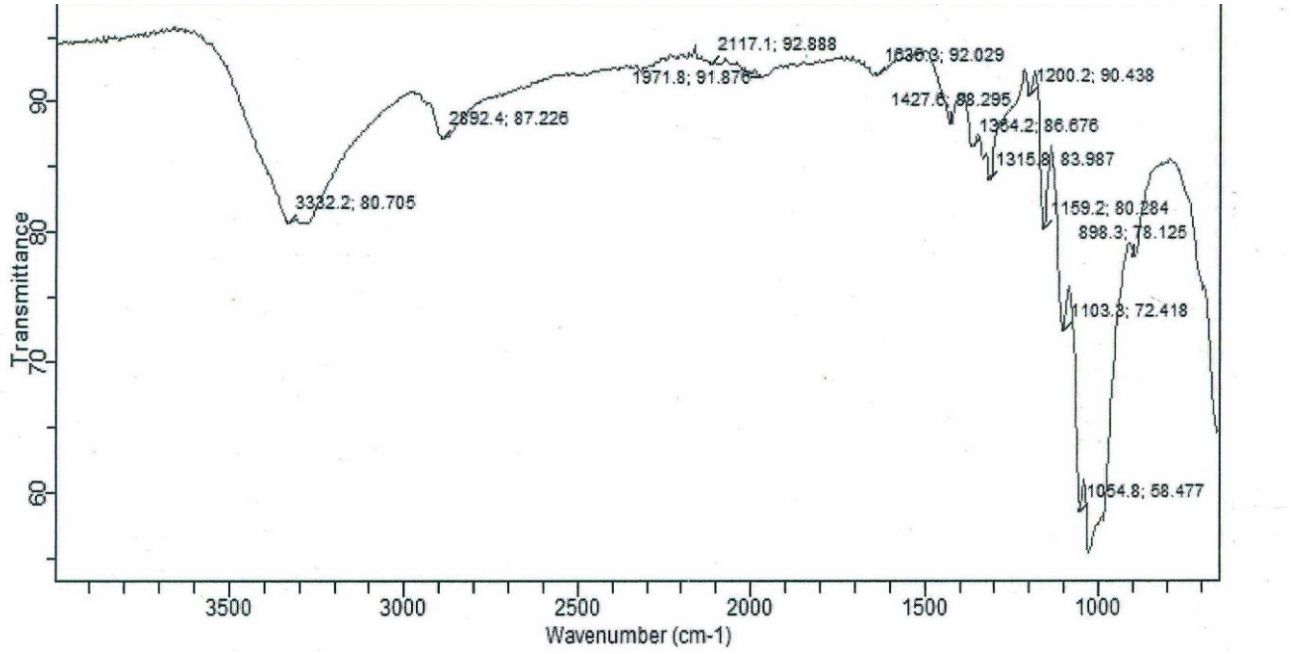


**Figure 5: FTIR Spectrum for Sample Reference**

**Melting Point determination**

The melting points of the four (4) samples and the AvicelPH-102 are shown in Table 2.

**Table 2.** Melting profile of the CDMCCs and Avicel®PH-102

| Sample | Starting point | End point |
|---|---|---|
| A | 240 | 258 |
| B | 216 | 244 |
| C | 232 | 256 |
| D | 203 | 202 |
| Avicel®PH-102 | 246 | 264 |

**Discussion**

From Table 1, Sample C was found out to have the highest percentage yield using the alcohol extraction method. Rice chaff is made up of 30-35 % cellulose[20] while the percentage yield of microcrystalline cellulose from alpha cellulose ranging from 7 to 85 % depending on the material used and the extraction method.[21] Samples showed varying percentage yield values due to differences in geographical sources, paddy types, inherent properties such as growing conditions, hybridization methods, time of harvest and the presence of impurities.[22, 23] Work-done by Ohwoavworhua *et al*.[24] reported a percentage yield of MCC from rice chaff to be 15.2 %, although the variety used was not stated.

FTIR in this study typically focuses on identifying the functional groups, assessing the purity and structure of microcrystalline cellulose. The characteristics of both the standard AvicelPH-102 and the derived cellulose (CDMCC) remain the same upon FTIR comparison with only slightly differences in wave numbers. The major peaks are identical to functional group in microcrystalline cellulose as observed, this shows a good reproducibility of the extraction method used in the study.[25] The absence of generation of new peaks in the four spectra against the standards shows no presence of other chemical agent.[26] The polymers showed absorbance at hydroxyl(-OH), amide group (CN) and aliphatic (-CH) stretching bands in the region between 3352 and 2989 $cm^{-1}$ respectively and this further confirmed the FTIR analysis of microcrystalline cellulose (MCC) fibre from Corn cob treatment in research conducted in 2018.[27] The absence of peaks in the range of 1509-1609$cm^{-1}$ for spectra of MCC obtained from rice chaff which correspond to the C=C aromatic skeletal vibrations indicate the complete removal of lignin.[28]

Pure compounds have a characteristic and sharp melting point or at a narrow specific range ($1 - 2^{O}C$).

Impurities lower and broaden the melting point range. Melting point determination continues to be an essential tool in various fields including pharmaceuticals, food and materials science.[29] From the result shown in Table 2, melting was observed at different points and temperatures across the four samples and the standard. Both samples A and C shows a higher melting point in comparison to the Avicel®PH-102 while the other two show a melt of the samples at much lower temperature. Those with relatively higher melting point in pharmaceutical sciences are highly value because they tend to have greater thermal stability, making them more suitable for process that involve sterilization and formulation of controlled released drugs, where slow dissolution rates are desirable. [30]From the study, it shows that samples A and C will be better for pharmaceutical formulation. From previous studies, materials made up of the same species can have different melting points because of differences in their structure and bonding. The same atoms can arrange themselves in different ways.[31]

**Conclusion**

In this study, cellulose was successfully extracted, characterized and chemically modified from selected local varieties in Northern Nigeria. The characterization results to a large extent confirmed the purity level after appropriate treatments as well as the structural integrity achieved

through chemical processes. The findings demonstrate that rice chaff, an abundant agricultural waste in Northern Nigeria, is a promising and sustainable source of cellulose for various industrial applications including pharmaceuticals. This work not only contributes to waste valorization but also the potential for local raw materials to support bio-based industries

**Recommendation**

1. Although this research focused on laboratory scale production process, further work is recommended to optimize extraction techniques for industrial scalability.
2. More advanced functionalization pathways can also be envisaged, which could enhance the mteterials properties for specialized applications.

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