



Comparative Study of the Effect of Green Cellulose Extraction Method on Corn Husk Biomass Cellulose

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How to cite this paper: Udebuani, A.C., Nnorom, O.O., Uzomah, A., Ufomadu, U.I. and Ebonine, D.M. (2026) Comparative Study of the Effect of Green Cellulose Extraction Method on Corn Husk Biomass Cellulose. *Open Access Library Journal*, 13: e15566.

<https://doi.org/10.4236/oalib.1115566>

Received: June 1, 2026

Accepted: June 28, 2026

Published: July 1, 2026

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Abstract

Corn husk, one of the most abundantly generated by-products of maize processing, is routinely discarded through open dumping or burning in many communities especially in Africa, contributing to environmental pollution and the loss of potentially valuable biomass. In this study cellulose was extracted corn husk using two green methods. The extraction involved de-waxing, pre-treatment and bleaching. Fourier Transform Infrared Spectroscopy (FTIR) was employed to confirm the chemical identity and content of the extracted cellulose. The result showed that both methods completely eliminated the hemicellulose and lignin content of corn husk. However, the cellulose content intensities of the first method were higher. These results confirm that the sequential extraction procedure successfully isolated cellulose from corn husk and underscore the potential of this agricultural waste as a sustainable, low-cost feedstock for cellulose-based materials.

Subject Areas

Material Experiment

Keywords

Corn Husk, FTIR, Cellulose, Green Extraction

1. Introduction

Cellulose is the most abundant natural polymer on Earth and serves as a key re-

newable feedstock for a wide range of sustainable materials, including biocomposites, nanocelluloses, membranes, packaging materials, and pharmaceutical excipients [1]. Structurally, cellulose is a linear polysaccharide composed of β -D-glucose units linked through β -1,4-glycosidic bonds, forming highly ordered crystalline and amorphous regions that contribute to its remarkable mechanical strength, biodegradability, and chemical versatility [2]. Due to increasing environmental concerns and the growing demand for sustainable materials, cellulose has gained substantial attention as a viable alternative to petroleum-derived materials in several industrial applications.

Conventionally, cellulose is obtained primarily from wood pulp and cotton. However, the increasing depletion of forest resources, rising processing costs, and environmental impacts associated with conventional extraction routes have intensified research into alternative lignocellulosic feedstocks, particularly agricultural residues and non-food biomass [3]. Utilizing agricultural waste as a cellulose source not only reduces environmental burden associated with biomass disposal but also supports circular-economy principles through waste valorization and resource efficiency.

Among the various agricultural residues investigated, corn husk, an abundant by-product generated during maize processing, has emerged as a promising cellulose source because of its high cellulose content, low cost, biodegradability, and widespread availability [4]. Large quantities of corn husk are generated annually and are often discarded through open dumping or burning, contributing to environmental pollution and underutilization of potentially valuable biomass resources. The conversion of corn husk into value-added cellulose materials therefore presents an environmentally sustainable approach for managing agricultural waste while creating functional biomaterials for industrial applications.

Corn husk is primarily composed of cellulose, hemicellulose, lignin, waxes, and other minor extractives arranged within a complex lignocellulosic matrix [5]. The presence of lignin and hemicellulose strongly influences the purity and performance of the extracted cellulose, thereby necessitating effective pretreatment and purification processes [6]. Several studies have demonstrated that alkaline treatment combined with bleaching processes can effectively disrupt the lignocellulosic structure, remove non-cellulosic components, and enhance cellulose purity and crystallinity [7] [8]. However, the efficiency of cellulose extraction is highly dependent on the extraction conditions employed, including alkali concentration, bleaching intensity, treatment duration, and reaction temperature.

Although cellulose extraction from corn husk has been reported in literature [2] [9], inconsistencies still exist regarding the optimization of extraction procedures and the extent to which extraction methods influence the purity of the isolated cellulose. This research analyses the effect of extraction methods on the purity of extracted cellulose from Corn husk a waste agricultural biomass using FTIR testing which is variously used to find and identify material contaminants. In addition, limited emphasis has been placed on using spectroscopic analysis to sys-

tematically verify the effectiveness of extraction treatments in removing lignin and hemicellulose residues from corn husk cellulose. Since the physicochemical properties and potential applications of cellulose are strongly affected by its purity, there remains a need for reproducible extraction strategies capable of producing high-quality cellulose from agricultural waste under relatively mild and environmentally conscious processing conditions.

2. Materials

The waste plant materials (Corn husk) were sourced from Eziobodo and Ihiagwa Communities in Owerri-west LGA of Imo State, Nigeria. Sodium hydroxide (NaOH) pellets, Benzene, Toluene, Acetic acid, Nitric acid (HNO₃), Sodium Borate (Na₂B₄O₇·10H₂O), Ethanol (CH₃COOH), Hydrogen peroxide (H₂O₂), distilled water, and Hydrochloric acid. All reagents are of analytical grade and were purchased from Pac Besh scientific Ltd. Owerri in Imo State, Nigeria.

2.1. Cellulose Extraction

Two methods were used for the extraction method. For the first method we followed a modified Procedure I method reported by Moran, J. I. *et al.* (2008) [10]. Corn husk was washed with distilled water, air dried to constant weight, pulverized and sieved to 300 µm particle size. 20 g of pulverized waste plant material was dewaxed using a 250 ml Soxhlet apparatus containing 125 ml of a boiling toluene/ethanol mixture (2:1 v/v) for approximately 6 h. Subsequently, 1 g of each dewaxed sample was soaked in 50 mL ethanol for 30 min, filtered, and air-dried. The dried sample was then treated with 100 ml of a 1:1 solution of 0.1 M NaOH and 50% ethanol at 45°C for 3 h, followed by air drying. Bleaching treatment was carried out sequentially using 50 ml of 3% hydrogen peroxide at pH 11 and 45°C, followed by 50 ml of 10% w/v sodium borate at 28°C, and finally with a 50 ml 1:10 (v/v) solution of 70% nitric acid and 80% acetic acid at 100°C for 15 min. The resulting solution was filtered, and the residue was washed successively with 95% ethanol, distilled water, and ethanol before the extracted cellulose was air-dried. Cellulose from this method is labeled C1.

For the second method, 20 g of pulverized waste plant material sample were dewaxed in a 250 ml Soxhlet apparatus with a solution of 125 ml Benzene and ethanol in a ratio of 2:1 for about 6 hours respectively. 1 g of each de-waxed sample was pretreated in a 100 ml beaker with 40 ml of 0.1 M HCl in a 70°C water bath for 2 hours. After which it was filtered, washed with distilled water, and air-dried. After drying, the sample was further soaked in 100 ml of 17.5% (w/v) NaOH for 1 h with continuous stirring. It was washed with distilled water, filtered and air dried. The dried samples were then bleached respectively with a solution of 60 ml of 20% hydrogen peroxide and 1% sodium hydroxide in 2:1 ratio for 45 minutes using a reflux condenser at a constant temperature of 50°C. The bleached samples were then washed with distilled water until neutral pH and air dried. Cellulose from this method is labeled C2.

2.2. FTIR Analysis

The functional groups of extracted cellulose and raw corn husk were examined using FTIR spectrometer (Agilent Cary 630). Scan range was between 650 - 4000 cm^{-1} using Happ-Genzel apodization technique, 8 resolutions with 32 scans using the transmittance mode.

3. Results and Discussion

Corn husk is a lignocelluloses material consisting of cellulose, hemicelluloses and lignin. FTIR is one of the most widely used procedures for quality control in material testing which is what informed our choice. The FTIR spectrum of each of the samples showed two distinct zones—the lower zone (400 - 1800 cm^{-1}) and the higher zone (2000 - 3500 cm^{-1}). The broad peak at the wave number between 3294.97 cm^{-1} to 3500 cm^{-1} of the three samples (**Figures 1-3**) corresponds to the

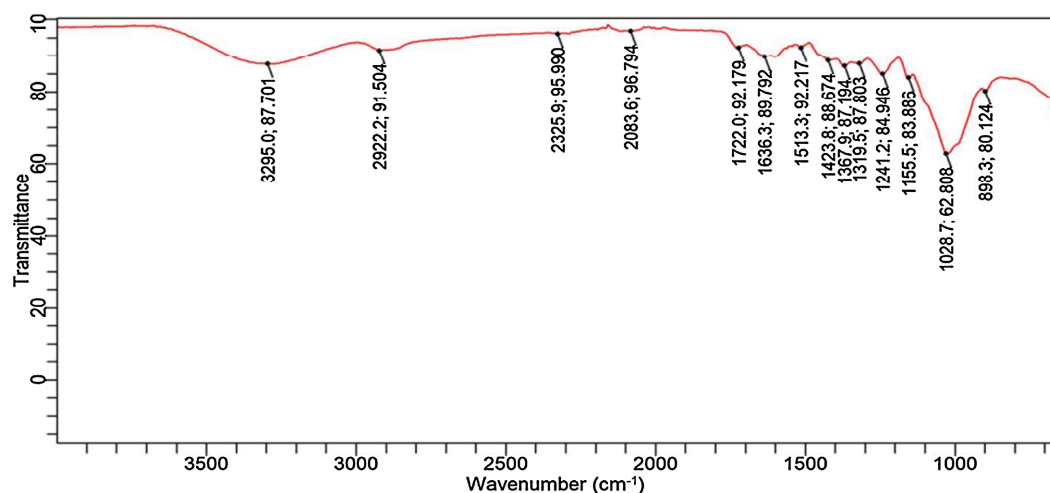


Figure 1. Raw corn husk powder (C).

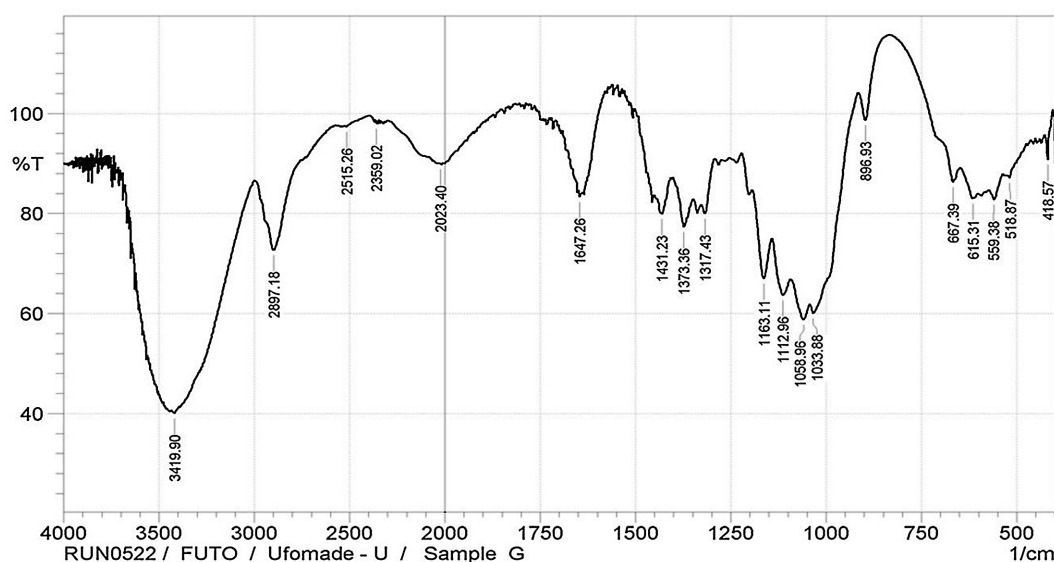


Figure 2. Corn husk cellulose (C1).

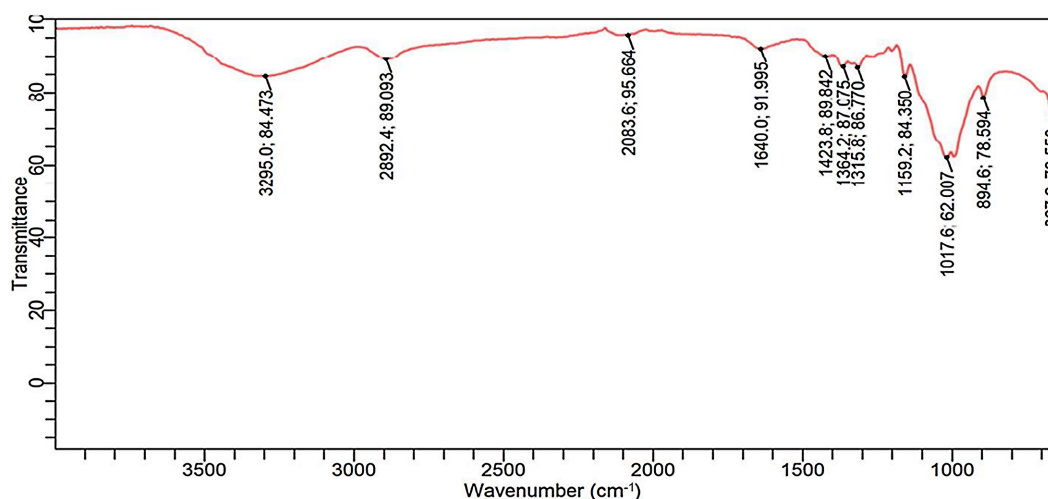


Figure 3. Corn husk cellulose (C2).

-OH stretching vibration, a characteristic absorption band of lignocelluloses materials [11]-[13]. The variation in the intensity of this broad peak signifies the OH content level, which reflects the degree of removal of non-cellulosic components such as lignin and hemicellulose during the extraction and bleaching process [14]. The peak at 2922, 2897 and 2892.41 cm^{-1} in C, C1 and C2 respectively is associated with C-H asymmetrical stretching vibrations, consistent with values reported for cellulose extracted from various lignocellulosic sources [11] [15] [16]. Peaks within 2000 - 2500 can be attributed to Carbon dioxide (CO_2) not fully removed from the background air during the analysis [17] [18]. We observed the absence of peaks 1722, 1513, and 1241 cm^{-1} found in the raw corn husk fig 1 representing C=O acetyl group in hemicellulose, and C-O-C of aryl-alkyl-ether in lignin respectively [19] [20] from the extracted cellulose C1 and C2. This is an indication that both extraction methods used removed hemicellulose and lignin from the raw corn husk powder. The bands at 1647 and 1640.03 cm^{-1} of C1 and C2 respectively is the O-H bending of absorbed water, mainly associated with cellulose [13] [21]. The vibration at 1431 and 1423.84 cm^{-1} is CH_2 scissoring bending in cellulose [11], while the band at 1373 and 1364.21 cm^{-1} is attributed to C-H bending in cellulose [11] [22]. The peak at 1317 and 1315 cm^{-1} is caused by CH_2 rocking vibration [23]. The band at 1163 and 1159 cm^{-1} represents the C-O-C stretching, corresponding to the β -(1,4)-glycosidic linkages between the glucose units in cellulose [11]. The broad peak at 1017 - 1133 cm^{-1} represents the C-O-H bending vibration, further supporting the cellulosic nature of the extracted material [16].

The band observed at 894.56 cm^{-1} is linked to cellulose crystallinity, representing the β -glycosidic linkage characteristic of the $I\beta$ cellulose structure [14]. It has been reported the intensities of 1420 - 1430 cm^{-1} is associated with the crystallinity level of cellulose ($I\beta$ cellulose structure), while the intensities of 893 - 897 cm^{-1} are associated with the β -(1,4)-glycosidic structure and $I\alpha$ cellulose [23]-[25]. Our results show that the intensities of the various peaks are higher in C1 than in C2. This difference is attributed to effect of the different extraction methods used.

Peak intensities have also been linked to material content with higher intensities indicating higher content [26]. It is also likely that the high NaOH concentration used in the extraction of C2 affected the cellulose content. High concentration of NaOH has been reported to affect cellulose adversely [27] [28].

4. Conclusion

Successful Cellulose extraction from corn husk using two different methods has been reported. The report of our analysis result was based solely on FTIR purity assessment carried out on the extracted cellulose from the different methods. Both extraction methods were green and removed hemicelluloses and lignin which are the major constituents of lignocelluloses materials. The intensities of the cellulose functional groups were however not the same. C1 cellulose had more cellulose content than C2 cellulose based on the FTIR intensities of their constituent groups. The use of high concentration of NaOH is a major factor affecting the cellulose content of extracted from corn husk and other plant biomass. Whereas our first extraction method yielded cellulose with higher contents it requires more reagents and time hence will be costlier than the second method. We therefore recommend the use of our second extraction method with reduced sodium hydroxide concentration. FTIR analysis provided solid confirmatory evidence of cellulose identity, however use of other characterization procedures is recommended for further studies.

Acknowledgements

The authors gratefully acknowledge the TETFund/DR & D-CE/ NRF 2020 Laboratory at Federal University of Technology, Owerri where the research was performed.

Funding

This research is part of TETFund Institution Based Research (FUT/OVC/TETF/GEN.8/VOL.4) 2025 funded by TETFund.

Conflicts of Interest

The authors declare no conflicts of interest.

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