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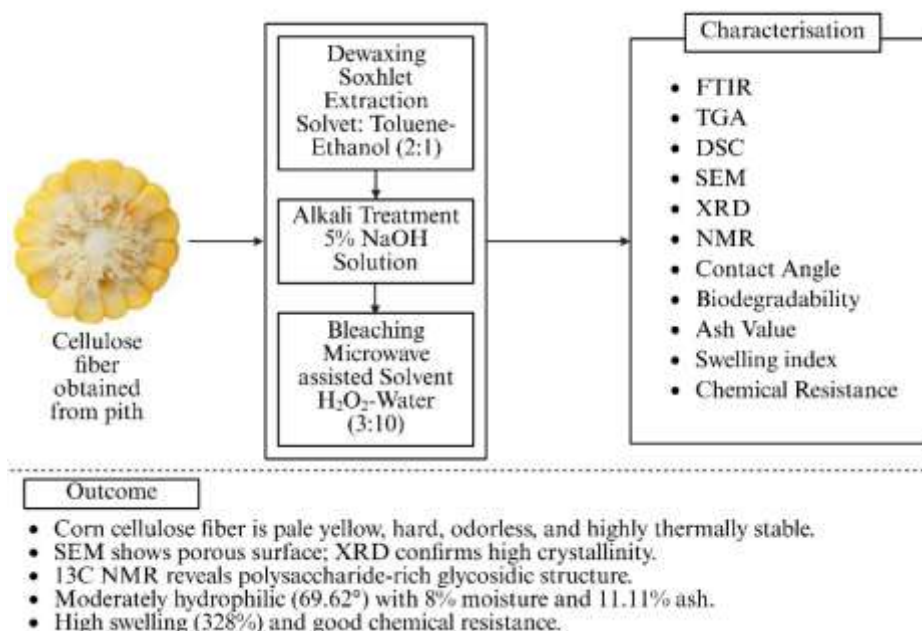
Microwave-Assisted Extraction and Characterisation of Corn (*Zea mays*) Cob Waste-Derived Cellulose for Biomedical Application: Sustainable Valorisation of Food Waste

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ABSTRACT: Corn (*Zea mays* L, family: Poaceae) cob, a widely available agricultural by-product, holds significant potential for sustainable material development. Cellulose is a major component of corn cob. This research aims to extract and characterise cellulose obtained from corn cob. The extraction of cellulose from maize cob was conducted utilising a microwave-based green technique and thereafter characterised using FTIR, TGA, DSC, SEM, XRD, solid-state NMR, contact angle measurement, ash value, swelling index, moisture content, chemical resistance, and biodegradability tests. Cellulose was successfully extracted from corn cob and comprehensively characterised using various parameters. Organoleptic evaluation showed the cellulose to be pale yellow, hard in texture, and odourless. FTIR spectroscopy demonstrated the presence of functional groups associated with cellulose and hemicellulose, while TGA revealed a total mass loss of 57.41%, indicating the presence of thermally labile components, with 30.35% of the residue remaining stable. DSC demonstrated a significant endothermic transition, with peak at 550.48 °C, suggesting high thermal stability. SEM images showed a rough, layered, and porous surface morphology, while XRD displayed sharp peaks at $2\theta \approx 16^\circ$, 22° , and 34° , confirming a highly crystalline cellulose I β structure. Solid-state ^{13}C NMR revealed distinct peaks between 60–110 ppm, showing that the sample is polysaccharide-rich crystalline material. The cellulose exhibited moderate hydrophilicity, with a mean contact angle of 69.62° . Additionally, the moisture content was found to be 8%, and the ash value was found to be 11.11%. The swelling index was found to be 328% in distilled water, 256% in phosphate buffer, and 204% in HCl, indicating high water absorption capacity. Chemical resistance was stronger in distilled water (4%) compared to phosphate buffer and acid solution (6%). Biodegradability studies revealed a 14.9% weight loss over 21 days, indicating moderate natural decomposition and highlighting its potential as a sustainable, biodegradable material. Corn cob cellulose demonstrates promising structural, thermal, and physicochemical properties, making it a sustainable, low-cost biomaterial suitable for diverse industrial and environmental applications.

GRAPHICAL ABSTRACT:



INTRODUCTION

Each year, an estimated 140 gigatons of biomass are disposed of as waste worldwide, resulting in significant environmental consequences, such as greenhouse gas emissions and degradation of soil and water quality. This also includes forest biomass, which is estimated to be approximately 4.6 Gt annually. Approximately 60% of this total is used for energy generation, whereas the remaining fraction comprises waste biomass [1]. Population growth and food and fuel debates put pressure on agricultural sectors to produce more biomass. The increasing need and accessibility of agricultural products have resulted in an increased disposal of biomass residues. The contemporary approaches to the disposal and utilisation of discarded biomasses have evolved from conventional practices, including fertiliser production, animal feeding, and incineration, to encompass the generation of biogas, biofuels, and various value-added bioproducts [2].

Cellulose is a plant material categorised as a polysaccharide and is made up of a long chain of more than 500 glucose molecules [3, 4]. Cellulose has evolved into an important material in the modern day. It has substantial applications in a wide variety of industries, such as the textile and garment industry, the paper industry, the cosmetics industry, the veterinary food industry, and the pharmaceutical industry. The two-century-old industry of cellulose production, which is extracted from plant material, was first discovered by French chemist Anselme Payen in 1838 [5]. Hemicellulose and lignin are the components of cellulose and are amorphous in nature [6-8]. Cellulose is typically extracted using physical, chemical, or enzymatic mechanisms [9]. A significant proportion of cellulose is present in agricultural residues, including palm bunch chaff, cocoa pods, plantain peels, banana leaves, maize cobs, wood, sugar beetroot waste and others. Upon mechanical reduction of these materials, the resulting fractions comprise cellulose, which, when broken down, yield crude cellulose [10]. Cellulose is insoluble in water and has the capacity to absorb water; additionally, it also has a high molecular weight and dense packing of its linear chains. Cellulose is generally crystalline in nature [11, 12]. The extraction of cellulose from biomass has been the subject of extensive research due to its essential role in both natural and industrial systems, as well as its substantial potential to advance sustainable development goals and apply principles of industrial ecology [13]. Recent research demonstrates that cellulose can be derived from a variety of plant materials, including cocoa pod husk, sugarcane bagasse, rice husk, soybean hulls, corn stover, coconut coir, jute bast, oil palm waste, banana leaves, and Ackee [14, 15].

The escalating environmental issues linked to synthetic and non-biodegradable substances have heightened the search for sustainable materials sourced from renewable agricultural resources. Corn is a major agricultural product, with the kernels mostly used by humans, while the stem, stalk and leaves serve as cattle fodder. The maize cob is often considered a byproduct

of agricultural practices. Corn cobs, an abundant agricultural by-product, could serve as a viable supply of cellulose for the development of sustainable biomaterials. The primary components of corn cob is cellulose, which could be effectively removed using a green technology. Various characterisation parameters of the cellulose encompass Fourier Transform Infrared Spectroscopy (FTIR), Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), Scanning Electron Microscopy (SEM), solid-state Nuclear Magnetic Resonance (NMR), X-ray Diffraction (XRD), contact angle measurement, moisture content assessment, ash value determination, swelling index evaluation, and chemical resistance testing. Utilising corn cobs for obtaining cellulose reduces pollution in the environment and improves agricultural waste management by making it into high-performing, biodegradable materials that may be used in a variety of industrial and environmental settings.

MATERIALS AND METHODS

Chemical reagents

The chemicals used in the research were toluene, hydrogen peroxide, hydrochloric acid, ethanol, and sodium hydroxide. Chemicals utilised in the research were procured from Sigma-Aldrich. The corn cob was obtained from agricultural farms located in Sonipat, Haryana (28.8971° N, 77.1920° E). Additionally, the plant was authenticated by Dr. Priyanka Ingle, Scientist D, BGIR Noida from the Ministry of Environment, Forest and Climate Change, Botanical Survey of India, Botanic Garden of Indian Republic, Sector 38A, Gautam Budh Nagar, Noida-201303, with authentication reference number as “BSI/BGIR/1/TECH./2025/222.”

Extraction of cellulose from corn cob

The corn cob, an extensively available agricultural by-product, possesses considerable potential as it can be utilised as a sustainable biomaterial. This research investigates the efficient extraction and thorough characterisation of maize cob cellulose. The extraction process comprised a series of steps including dewaxing, alkali treatment, and bleaching, which effectively eliminated lignin, hemicellulose, waxes, and other non-cellulosic constituents, resulting in efficient extraction of cellulose. The corn cobs were thoroughly dried, and the biomaterial was removed from the pith of the dried corn cobs. The obtained biomaterial was then subjected to dewaxing using a binary solvent system containing ethanol and toluene in a ratio of 2:1. The dewaxing process was conducted utilising a Soxhlet apparatus for a duration of 4 h at 60 to 70° C temperature range [16]. After the dewaxing, the material was subjected to alkaline treatment utilising a 5% (w/v) sodium hydroxide solution, at a temperature of 50 °C for a duration of 2 hours. The alkali treatment was conducted to remove hemicellulose and lignin [17]. Subsequently, the material was allowed to cool to normal temperature and underwent multiple washings with distilled water.

Furthermore, the material was subjected to bleaching for decolouration. The bleaching was performed using the bleaching solution of hydrogen peroxide with distilled water in the ratio of 3:10 (v/v). Microwave-assisted bleaching method was performed, with alternating periods of heating and cooling cycles of 5 minutes each, for a duration of 30 minutes [18]. After bleaching, the material was allowed to cool to normal temperature and underwent multiple washings with distilled water. Furthermore, the neutralised cellulose was thoroughly dried in the hot air oven. Figure 1 shows the extraction procedure of corn cob cellulose. The extracted cellulose biomaterial was then further processed to various characterisation parameters.

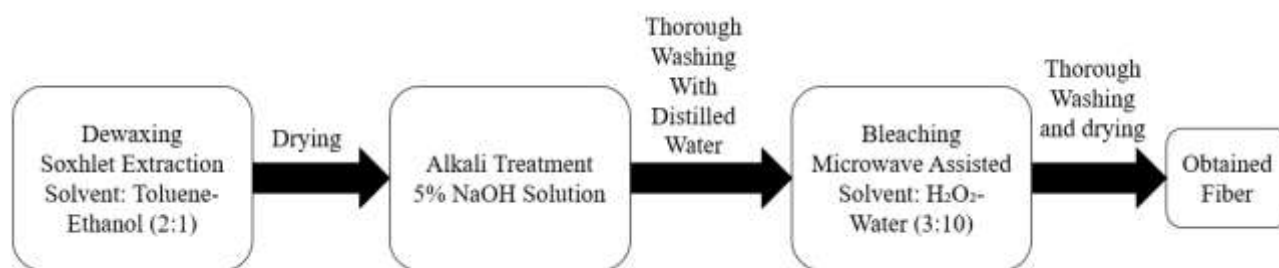


Figure 1: Diagrammatic representation of corn cob cellulose extraction procedure: (1) solvent dewaxing and drying, (2) alkali treatment with NaOH and thorough washing, (3) microwave-assisted H₂O₂ bleaching, final washing and drying, and (4) Cellulose obtained.

CHARACTERIZATION METHOD

After the extraction of cellulose from the corn cob, the obtained cellulose was characterised using various parameters such as FTIR, TGA, DSC, SEM, XRD, solid-state NMR, contact angle measurement, ash value, swelling index, moisture content, chemical resistance, and biodegradability tests.

Organoleptic Properties: The organoleptic evaluation of corn cob cellulose was conducted to assess its colour, physical appearance, and odour.

Fourier transform infrared spectroscopy (FTIR): FTIR spectroscopy was performed for the determination and identification of the main chemical functional groups present in the extracted cellulose using the Bruker FTIR Alpha II instrument. The cellulose sample was analysed in the range of 4000–400 cm^{-1} and the spectra were recorded.

Thermogravimetric analysis (TGA): The thermogravimetric analysis (TGA) of cellulose was conducted to determine the thermal stability, measure the fraction of volatile components, and evaluate the variations in mass as a function of temperature. The TGA was performed on Mettler Toledo TGA/DSC 3+ apparatus. The sample was heated in nitrogen surrounding at a controlled rate of 10°C per minute, and the temperature ranged from 30°C to 600°C. The TGA curves were generated and analysed.

Differential scanning calorimetry (DSC): The DSC of the cellulose was performed for the determination of enthalpy (heat flow) alteration in a sample because of a function of temperature. The DSC was performed on the Mettler Toledo TGA/DSC 3+ apparatus. The sample was heated in a nitrogen surrounding at a controlled rate of 10°C per minute, and the temperature ranged from 30°C to 600°C. The DSC curves were generated and analysed.

Morphological Characterisation: The SEM (scanning electron microscopy) was carried out for the visualization and microstructural analysis of the cellulose. The SEM was performed on JEOL JSM-7610F Plus SEM instrument. The sample was coated with gold and were analysed in high vacuum condition. The cellulose was visualised at a magnification of 2500x.

X-ray diffraction (XRD): The XRD of the sample was performed for the assessment of the cellulose crystallinity. The X-ray diffraction (XRD) analysis of the sample was performed using a Bruker D8 Advance X-ray Diffractometer. The sample was analysed utilising Cu, K α radiation ($\lambda = 1.5406 \text{ \AA}$) within the 2θ range of 5° to 40°.

Solid-state Carbon-13 Nuclear Magnetic Resonance (Solid-state ^{13}C NMR): Solid-state ^{13}C NMR was performed to determine the chemical structure and characteristics of cellulose. The solid-state ^{13}C NMR was conducted utilising a 400 MHz Bruker AVANCE NEO Solid State NMR Spectrometer. Cross-polarisation magic angle spinning (CP/MAS) NMR spectra were obtained. Peaks associated with the C1–C6 carbon atoms in cellulose were identified.

Contact Angle: The contact angle test was performed to assess the hydrophobic or hydrophilic characteristics of the cellulose. The Rame Goniometer model 100-00-230 was used to determine the contact angle. The findings were reported as the mean of triplicate measurements with standard deviations [19].

Moisture Content: The moisture content of the sample was determined by measuring the weight of water present within the cellulose. The moisture content was assessed by measuring the initial mass (W_1) of the sample, which was dried in a hot air oven at $105 \pm 2^\circ\text{C}$ for a duration of 24 hours [20]. The dried sample was then allowed to cool in a desiccator, and thereafter again the weight was measured (W_2). The moisture content was calculated using equation 1. The data was presented as the average of triplicate readings with standard deviations.

$$\text{Moisture content (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad \text{Equation 1 Where:}$$

W_1 – initial weight of the cellulose (g)

W_2 – final weight of the cellulose (g)

Ash Value: Ash value is the amount of non-combustible inorganic material that remains after burning a cellulose sample. This value indicates the sample's mineral content and purity. Ash content is the amount of inorganic residue left after combustion [21]. Ash value of the cellulose was determined by burning a pre-weighed sample in a crucible at a high temperature, then

weighing the remaining ash. The sample was precisely weighed (100 mg). Thereafter, the sample was incinerated within the crucible at around 500°C in a muffle furnace for several minutes. After ashing, the crucible was carefully cooled within a desiccator to prevent moisture absorption, and the ash of the cellulose was weighed again. The ash content was determined using equation 2, and the findings are presented as the mean of triplicate measurements with standard deviations.

$$\text{Ash content (\%)} = \frac{\text{Weight of Ash}}{\text{Weight of sample}} \times 100 \quad \text{Equation 2}$$

Swelling Index: The swelling index was assessed to determine the swelling capacity of the cellulose in three different liquid solvents, that is, 0.1 N HCl, distilled water, and a phosphate buffer at a pH of 7.4. A pre-weighed sample (W_1) was submerged in distilled water for 24 hours. After 24 hours, the excess liquid was removed and the sample was reweighed (W_2). The swelling index was calculated using equation 3 [22]. The results were expressed as the mean of triplicate measurements, with standard deviations.

$$\text{Swelling index (\%)} = \frac{W_s - W_d}{W_d} \times 100 \quad \text{Equation 3}$$

Where:

W_s – Swollen weight of cellulose (g)

W_d – Dried weight of the cellulose (g)

Chemical Resistance Test: The chemical resistance test of the cellulose was performed by exposing a sample to specific chemical solutions (distilled water, phosphate buffer and HCl solution) and then measuring changes in its weight. For the determination of chemical resistance, 50 mg of the cellulose sample was immersed in three different solutions, that is pH 7.4 phosphate buffer, distilled water, and 0.1 N HCl for a period of 24 hours to determine its stability across acidic, physiological, and neutral conditions.

Following a duration of 24 hours, the extra liquid was removed, and the samples were subsequently dried in a hot air oven. After complete drying, the weight of the samples was measured [23]. Thereafter, the chemical resistance was calculated using Equation 4. The data were expressed as the mean of triplicate measurements, with standard deviations.

$$\text{Weight remaining (\%)} = \frac{W_i - W_f}{W_f} \quad \text{Equation 4}$$

Where:

W_i : Initial weight

W_f : Final weight

Biodegradability: A soil burial biodegradability test is used to evaluate the decomposition of the sample when buried in soil by observing mass loss and changes in mechanical properties. The biodegradability of the cellulose sample was calculated by the soil burial method. The research employed 200 mL plastic container, containing 200 g of compost collected from the agricultural land of Dankaur, Greater Noida, Uttar Pradesh, India (28.3486° North and 77.5529° East). A quantity of 0.1 g of cellulose sample was placed within a container, at a depth of 5 cm. To maintain the moisture, 5 ml of water was added to the container every 24 hours [24].

A small drain was made at the base of the container for the removal of excess water. The biodegradability test was conducted at normal room temperature. After 21 days, the sample was removed from the container, followed by washing it with distilled water. Subsequently, the sample underwent drying and was then weighed. The determination of biodegradability was conducted by calculating the percentage of weight loss, using equation 5, through the analysis of the FTIR of the sample. The data were presented as the mean of triplicate measurements, accompanied by the standard deviation.

$$\text{Weight loss (\%)} = \frac{\text{Initial weight of the sample} - \text{Final weight of sample}}{\text{Initial weight of sample}} \times 100 \quad \text{Equation 5}$$

RESULT AND DISCUSSION

Organoleptic Properties: The cellulose obtained from corn cob has a pale-yellow colour, hard texture with no odour.

Fourier transform infrared spectroscopy (FTIR): The principal chemical functional groups of cellulose were identified and characterised through FTIR analysis (Figure 2 and Table 1). The FTIR analysis of the extracted biomaterial revealed various significant peaks that demonstrated the successful isolation of cellulose. The FTIR spectrum of the cellulose reveals the presence of various functional groups, confirming its complex chemical composition. A medium peak observed at 3346 cm^{-1} indicates N–H stretching associated with secondary amines, whereas a strong, broad peak at 3026 cm^{-1} shows N–H stretching of amine salts. The C–H stretching of alkanes is determined by 2944 cm^{-1} , and a weak band at 2672 cm^{-1} represents S–H stretching from thiol groups. A strong absorption at 2348 cm^{-1} indicates O=C=O stretching due to carbon dioxide. The peaks seen at 1942 cm^{-1} and 724 cm^{-1} show aromatic C–H bending and C=C bending in alkenes. The peaks identified at 1730 cm^{-1} and 1627 cm^{-1} relate to the C=O stretching vibrations of α,β -unsaturated esters and the C=C stretching vibrations of α,β -unsaturated ketones, respectively. Additionally, more peaks observed are 1517 cm^{-1} (N–O stretching, nitro compounds), 1248 cm^{-1} (C–N stretching, aromatic amines), and 1076 cm^{-1} (C–O stretching, primary alcohols), indicating various functional groups in the cellulose. The findings from the FTIR analysis showed peaks that indicate the presence of differing functional groups. The results were analysed in relation to prior studies, which indicated that the FTIR analysis of corn cob biomaterial yielded comparable findings. The study carried out by Maryli et al. reveals a significant alteration in peaks at 1730 cm^{-2} , $2922\text{--}2854\text{ cm}^{-3}$, and $1318\text{--}1372\text{ cm}^{-3}$, indicating the stretching of carbonyl bonds, C-H and CH_2 bonds, along with phenolic O-H groups [25]. Moreover, the spectra provided by Paola et al. exhibit bands that are indicative of several functional groups, including hydroxyl (-OH), methyl (-CH₃), methylene (-CH₂), and acetal/ketal groups that are associated with glycosidic C-O-C bonds. Furthermore, a carbonyl (C=O) band and C-H bond bands are detected at 1420 cm^{-1} and 822 cm^{-1} [26].

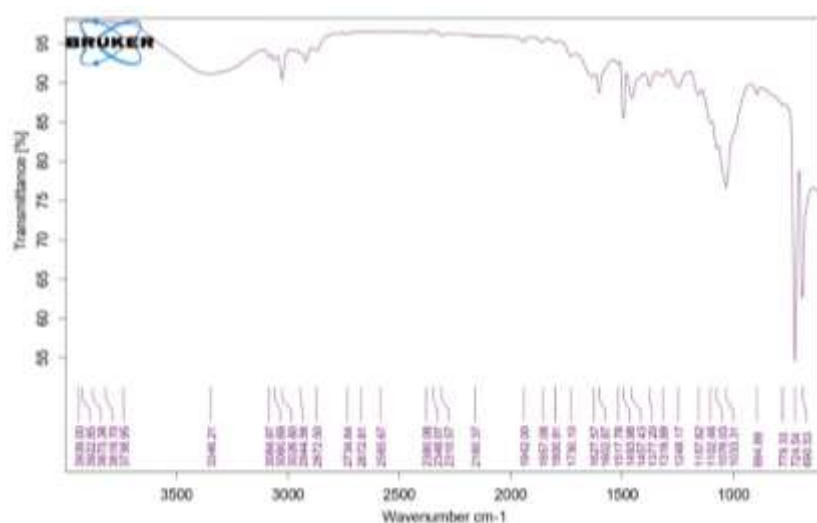


Figure 2: FTIR spectroscopy of corn cob cellulose.

Table 1: FTIR spectroscopy of corn cob cellulose.

Wavenumber (cm ⁻¹)	Appearance	Group	Compound Class
3346	Medium	N-H stretching	Secondary Amine
3026	Strong, Broad	N-H stretching	Amine Salt
2944	Medium	C-H stretching	Alkane
2672	Weak	S-H stretching	Thiol

2348	Strong	O=C=O stretching	Carbon Dioxide
1942	Weak	C-H bending	Aromatic Compound
1730	Strong	C=O stretching	A,B-Unsaturated Ester
1627	Strong	C=C stretching	A,B-Unsaturated Ketone
1517	Strong	N-O stretching	Nitro Compound
1493	Medium	O-H bending	Carboxylic Acid
1377	Medium	O-H bending	Pheno
1248	Strong	C-N stretching	Aromatic Amine
1076	Strong	C-O stretching	Primary Alcohol
724	Strong	C=C bending	Alkene

Thermogravimetric analysis (TGA): The TGA of corn cob cellulose was performed to evaluate the thermal stability (Figure 3). The TGA indicates a total mass loss of 57.41%, corresponding to a weight reduction of 1.7223 mg from the initial 3.0000 mg sample at above 300°C. This degradation indicates the presence of considerable thermally stable material. The residue of 30.35% (0.9105 mg) confirms the presence of a thermally stable substance, likely composed of inorganic or carbonaceous components. The TGA analysis revealed thermal behaviour through a two-step degradation pattern, that is an initial weight loss due to moisture and volatile components, followed by a substantial decomposition associated with organic matter. Additionally, 30% mass residual showed that the cellulose is thermally stable. The findings were compared with prior research, which indicated that the thermal degradation of corn-based biomaterial yielded comparable results. For example, Paola et al. conducted TGA analysis, and the results indicated two primary stages of mass loss in the corn-based biomaterial sample. The initial phase, occurring in the temperature range of 25 to 120 °C, was characterised by the removal of water, leading to a 7% reduction in mass. The second stage, occurring between 120 and 350 °C, involved the thermal decomposition of organic components, leading to a mass loss of 58% [26]. Additionally, research performed by Wang *et al.*, showed similar thermal stability characteristics, where lignin showed the highest resistance to pyrolysis, cellulose degraded sharply around 345°C, and hemicellulose decomposed earlier due to its branched structure [27].

Differential scanning calorimetry (DSC): The DSC of corn cob cellulose was performed to evaluate the thermal characteristics (Figure 3). The DSC result showed a significant endothermic phase, commencing at a temperature of 494.08 °C and reaching its peak at 550.48 °C, suggesting a substantial decomposition or phase transition. The enthalpy change associated with this shift was recorded as 8008.52 mJ, corresponding to a normalized heat flow of 2669.51 J/g, reflecting a significant energy requirement for structural disintegration or transformation within the sample matrix. The DSC confirmed the endothermic transition of the cellulose, confirming its structural stability. Therefore, it can be inferred that corn-based cellulose could be appropriate for industries like packaging, coatings, or advanced composites where thermal endurance is crucial because of its high decomposition temperature and substantial residue content, which indicate its potential for high-temperature applications or resistance to thermal deterioration. The findings were compared with prior research, which showed that the differential scanning calorimetry (DSC) of corn cellulose yielded comparable results. For example, Azubuike et al. conducted a DSC analysis of corn cob, revealing a broad endothermic peak attributed to moisture loss. The thermal decomposition process starts at around 200 °C, a temperature associated with the breakdown of hemicellulose and cellulose. The decomposition of lignin, on the other hand, occurs between 300 and 500 °C [28].

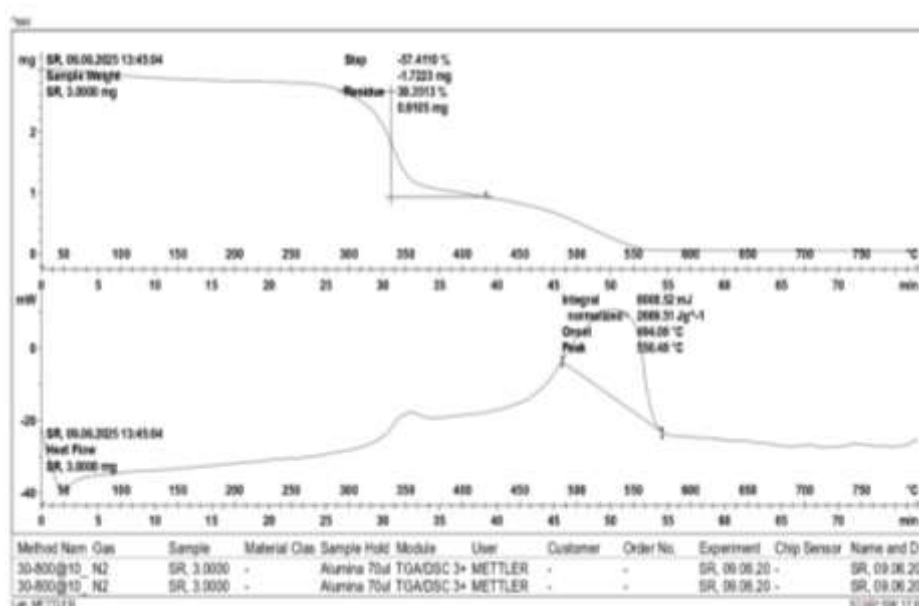


Figure 3: TGA and DSC analysis of corn cob cellulose.

Scanning electron microscopy (SEM): The morphology of the corn cob cellulose was examined through SEM analysis. The images obtained through scanning electron microscopy of the corn cob cellulose efficiently showed the intricate surface morphology (Figure 4). The SEM image at 2500 magnification illustrates a rough, layered, and wrinkled surface morphology, suggesting a highly porous and irregular structure. The SEM of the cellulose showed surface of the cellulose with layers, wrinkles, and pores. The high surface area of the cellulose can be useful for many applications. This type of surface morphology is advantageous for adsorption and functionalisation processes. These characteristics align with previous research findings. For example, the SEM micrographs presented by Sahare *et al.* illustrated that corn cob cellulose exhibited a porous, loose, and fibrillated structure, accompanied by an increased surface area [29]. Additionally, Honglin *et al.* also reported similar results [30].

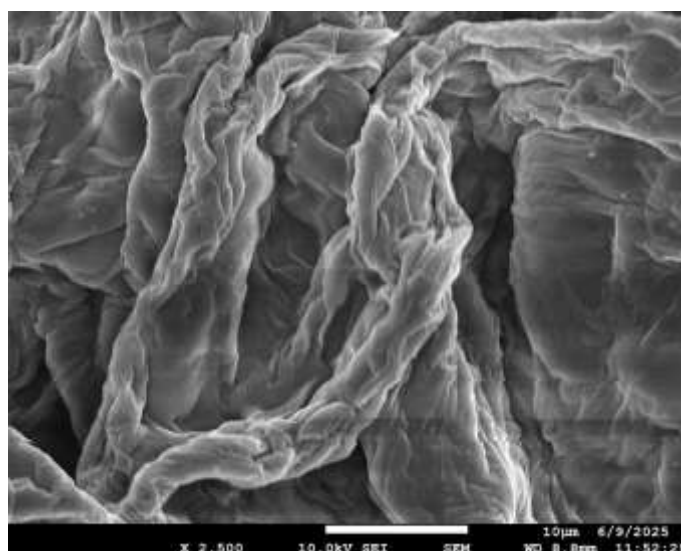


Figure 4: SEM image of corn cob cellulose.

X-ray diffraction (XRD): The XRD graph of the corn cob cellulose sample reveals distinct and sharp peaks, approximately at $2\theta \approx 22^\circ$, indicating that the cellulose exhibits a high degree of crystallinity (Figure 5). However, this intense peak is attributed to the (002) plane of crystalline polymers such as cellulose or related biopolymers, indicating a well-ordered internal structure.

The observation of supplementary diffraction peaks around $2\theta \approx 15^\circ$ and 30° indicates the presence of additional crystallographic planes within the cellulose. Furthermore, the graph displayed sharp peaks, particularly at $2\theta \approx 16^\circ$, 22° , and 34° , that are characteristic of cellulose I β , the dominant crystalline allomorph in plant cellulose. The observed peaks align with the (110), (200), and (004) crystallographic planes, respectively. The overall pattern, with sharp peaks superimposed on a relatively low background, confirms the high crystalline nature of the cellulose. The residual plot shown in the lower panel displays minimal deviation between experimental and calculated data, supporting the reliability and accuracy of the peak fitting and phase identification. The XRD analysis revealed distinct peaks at 16° , 22° , and 34° (2θ), thereby confirming the presence of a highly crystalline cellulose I β configuration. This analysis showed that the sample is made of polymeric material characterised by crystalline properties, obtained from a natural source. The characteristics observed align with findings from previous research. For example, Dewi et al. conducted X-ray diffraction (XRD) analysis on maize cob biomaterial, revealing that the XRD analysis of raw maize cob depicted cellulose and nanocrystalline cellulose (NCC) with crystallinity indices ranging from approximately 65.5% to 69.9%, while NCC exhibited values between 50.1% and 65.3%. Additionally, distinct diffraction peaks observed at $2\theta \approx 22.5^\circ$ provide confirmation of the Cellulose-I structure and indicate an increase in crystallinity [31].

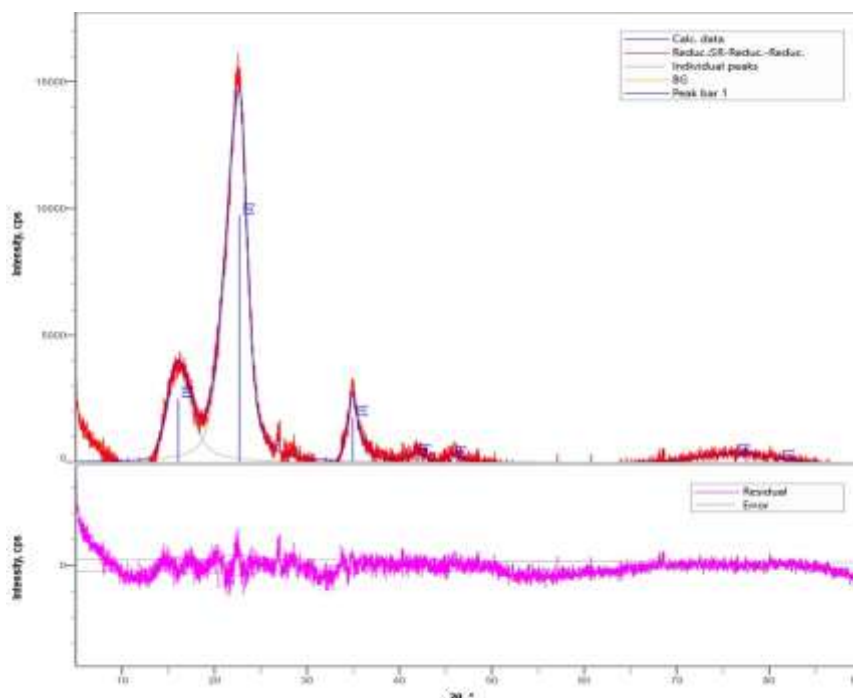


Figure 5: XRD of the Corn cob cellulose

Solid-state Carbon-13 Nuclear Magnetic Resonance (Solid-state ^{13}C NMR): The solid-state ^{13}C NMR spectrum showed various distinct peaks in the chemical shift range of 0 to 180 ppm (Figure 6). The peaks observed between 60–110 ppm indicate the presence of C–O, C–OH, or anomeric carbon compounds, which are common in polysaccharides. Sharp peaks at 70–80 ppm showed secondary alcohols or carbons bonded to oxygen. The peaks near 100–105 ppm indicate anomeric carbons in glycosidic linkages. On the basis of the analysis, the result shows that the sample is a crystalline or semi-crystalline polysaccharide-rich biopolymer. The solid-state ^{13}C NMR spectra showed that the cellulose has polysaccharides, confirmed by the presence of peaks in the 60–110 ppm region, especially at 100–105 ppm, which correspond to the anomeric carbons of cellulose. This suggests that the structural integrity of the cellulose is maintained following the extraction process. The observed characteristics align with findings from previous research. For example, Rui et al. conducted an analysis of the corn cob utilising solid-state NMR spectra, which indicated the presence of a cellulose signal peak and a lignin signal peak. The signal peak for cellulose was detected at 89.1 or 88.9 ppm, whereas the signal peak for lignin was identified at 83.4 or 84.2 ppm. The observed lignin signal peaks at 126.9 or 128.9 ppm and at 148.3 or 147.5 ppm have been related to the carbon atoms located in the benzene ring structure, in addition to the methoxy group present within the aromatic structure [32].

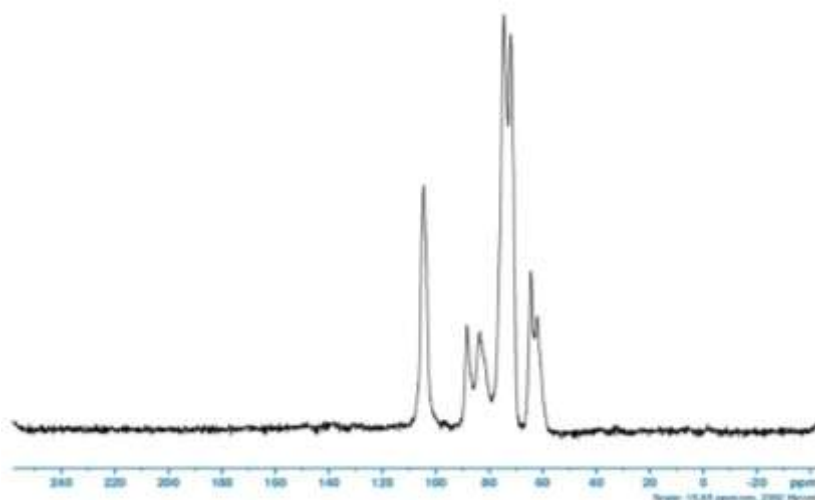


Figure 6: Solid state NMR spectrum of corn cob cellulose.

Contact Angle: The contact angle of the corn cob cellulose was found to be 67.38°, 71.67°, and 69.83°. The mean value of the contact angle of the cellulose is 69.62° (Table 2). As the contact angle is calculated to determine the hydrophobic and hydrophilic characteristics of a surface. A contact angle measuring below 90° denotes hydrophilicity in the sample, whereas an angle above 90° indicates hydrophobic characteristics. The measurement of the contact angle, approximately 69.6°, depicted the cellulose as hydrophilic in nature, indicating its capacity for water interaction while preserving structural integrity. Therefore, the corn cob cellulose was determined to possess hydrophilic characteristics [33]. This characteristic aligns with previous research findings. For example, Sara et al. performed research on the contact angles of biomaterials of various plants such as hemp, curaua, jute, fique, piassava, ramie, pineapple, and sisal. The contact angle of biomaterials of all the plants were observed to be weakly hydrophilic in nature, with contact angles measured between 40° and 50° [34].

Table 2: Characterisation parameters of corn cob cellulose

Parameters		Result
Contact Angel (°)		69.62±1.4°
Moisture content (%)		8±1.5
Ash value (%)		11.111 ± 1.24
Swelling study (%)	HCl (0.1 N)	204 ± 2.34
	pH 7.4 Phosphate buffer	256 ± 1.63
	H ₂ O	328 ± 1.73
Chemical resistance (%)	HCl (0.1 N)	6 ± 0.49
	pH 7.4 Phosphate buffer	6 ± 0.58
	H ₂ O	4 ± 0.34
Biodegradability (%)		14.9±2.16

Moisture Content: The moisture content of corn cob cellulose was evaluated utilising the oven-drying method, conducted at a temperature of 105 ± 2°C over a period of 24 hours (Table 2). The analysis of weight loss revealed that the moisture content of the corn cob cellulose is 8%. This indicates that the cellulose maintains stability and is appropriate for further processing, presenting minimal risk of microbial proliferation or deterioration. The analysis of moisture content, approximately 8%,

confirmed the stability post-extraction and the resistance to microbial degradation. This characteristic aligns with previous research findings. For example, Fahmi *et al.*, found the moisture content of the corn-based biomaterial to 6% [35].

Ash Value: The ash value of the sample was found to be 11.111%, indicating a significant presence of inorganic residues post-combustion (Table 2). This value indicates the existence of minerals within the sample, which are typically preserved following the thermal degradation of biomass. The low ash value indicates that corn cob cellulose can be regarded as a suitable biomaterial possessing the characteristics of a solid biomaterial [36].

Swelling Index: The swelling index of corn cob cellulose showed considerable variation across different media, with 328% in distilled water, 256% in phosphate buffer of pH 7.4, and 204% in HCl. Lindman *et al.* demonstrated that this swelling behaviour may be due to ionisation of cellulose [37]. The significant swelling observed in distilled water can be due to the free movement of water molecules into the cellulose framework, given the limited presence of ionic interactions [38]. In contrast, lowered swelling in acidic and buffered environments is because of stronger hydrogen bonding and reduced cellulose relaxation due to the presence of ions. The swelling index exceeding 300 % in distilled water illustrates a significant capacity for water absorption; however, slightly lowered swelling observed in phosphate buffer and acidic conditions indicates a sensitivity to ionic environments. These findings indicate that pH and solvent nature substantially influence the structural expansion and water-holding capacity of cellulose [39].

Chemical Resistant: The corn cob cellulose demonstrated greater chemical resistance in distilled water (4% erosion), compared to slightly higher erosion of 6% in both pH 7.4 phosphate buffer and 0.1 N HCl solution. The cellulose demonstrated significant chemical resistance, showing only minimal erosion across different solutions. This behaviour aligns with the inherent stability of cellulose, which retains structural integrity in neutral environments due to its closely packed hydrogen-bonding network and crystalline domains. In contrast, acidic and buffered conditions can disrupt these interactions, resulting in increased erosion. Cellulose is known for its robustness in water and moderate acid/buffer exposure, reinforcing the observed stability pattern in the biomaterial [40, 41].

Biodegradability: The biodegradability of corn cob cellulose was assessed through the observation of weight loss over a period of 21 days, which demonstrated a reduction of 14.9%, thereby indicating a moderate level of natural decomposition. The corn cob cellulose showed 14.9% weight loss in 21-day soil burial degradation study, indicating moderate biodegradability. This degradation may be because of the high crystallisation and dense structure of corn cob cellulose which impacts the rapid breakdown. Additionally, following a 21-day degradation procedure, the FTIR of the cellulose was performed, and the result showed significant chemical modifications (Figure 7 and Table 3). The FTIR analysis revealed the structural modifications following biodegradation, indicated by a reduction in the intensity of O–H stretching peaks at 3786 and 3319 cm^{-1} , as well as the C–O stretching peak at 1315 cm^{-1} . These findings indicate a partial degradation of cellulose and hemicellulose. In comparison to the pure cellulose, the biodegraded cellulose exhibited reduced characteristic peaks associated with alcohol, alkene, and ester functional groups, thereby depicting the occurrence of polymer chain cleavage as a result of microbial activity. The findings underscore the environmentally sustainable characteristics of corn cob cellulose and its potential characteristics of being biodegradable in nature [42, 43]. Previous research also showed similar results as for instance Brunšek *et al.* calculated the biodegradability study of various biomaterials using the soil burial method for durations of 2, 4, 7, 9, and 10 days. Hemp exhibited the highest mass loss at 44.51% and a reduction in tenacity of 66.17% following 10 days. The degradation of sisal was only 17.58%, while that of jute was 24.01%. Viscose exhibited a mass loss of 22.79%, attributed to its protective skin, despite its low crystallinity [44]. The biodegradation of recycled polypropylene composites loaded with oil palm trunk biomaterial was investigated by Khalil *et al.* where researcher performed 12-month soil burial tests. The findings revealed a gradual decrease in mechanical properties, with reductions in tensile, and impact strength ranging from 38% to 50%. Additionally, the recorded mass loss was 12.7%, primarily attributed to the processes of moisture absorption and microbial growth [45].

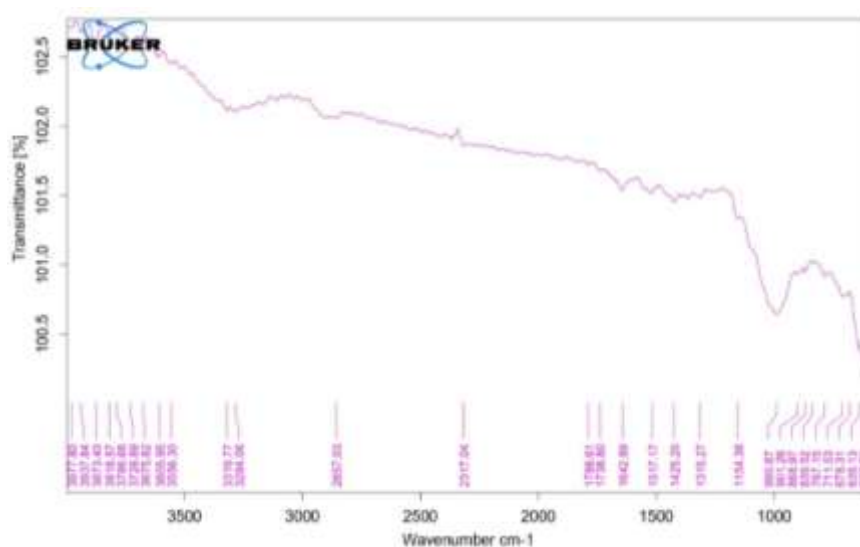


Figure 7: FTIR spectra of cellulose post biodegradability study.

Table 3: FTIR spectral analysis of cellulose post 21 days biodegradability study.

Wavenumber (cm ⁻¹)	Appearance	Group	Compound Class
3786	Medium, Sharp	O-H stretching	Alcohol
3319	Strong, Broad	O-H stretching	Alcohol
2857	Medium	C-H stretching	Alkane
2317	Strong	O=C=O stretching	Carbon Dioxide
1642	Medium	C=N stretching	Imine / Oxime
1517	Strong	N-O stretching	Nitro Compound
1425	Medium	O-H bending	Alcohol
1315	Strong	C-O stretching	Aromatic Ester
990	Strong	C=C bending	Alkene
839	Strong	C=C bending	Alkene
678	Strong	C=C bending	Alkene

CONCLUSION

The corn cob is a widely available by-product of agriculture that holds significant promise as a sustainable biomaterial. This study explores the effective extraction and comprehensive characterisation of corn cob cellulose. The extraction process involved dewaxing, alkali treatment, and bleaching, which efficiently removed lignin, hemicellulose, waxes, and other non-cellulosic components, leading to the obtaining of cellulose. The cellulose was characterised using various parameters. The FTIR analysis of the cellulose validated the existence of functional groups that are indicative of cellulose. Additionally, the XRD

analysis revealed distinct peaks at 2θ of 16°, 22°, and 34°, which shows the sample is crystalline. Thereafter, the TGA and DSC demonstrated good thermal stability, showing minimal mass loss up to approximately 300 °C. The SEM of the cellulose showed the surface is rough and has pores, indicating increased surface area. Furthermore, the chemical resistance test revealed that the cellulose has minimal degradation in distilled water, acidic environments, and buffer solution. Also, the cellulose showed a swelling index of 300% in distilled water and slightly decreased swelling in acidic and phosphate buffer solutions. Thereafter, the biodegradability study of the cellulose showed 14.9% degradation in 21 days, indicating moderate biodegradability. Also, the FTIR analysis confirmed the degradation. Overall, the result showed that the cellulose was effectively extracted from the agricultural byproduct of corn. Thereafter, the characterisation parameters of the cellulose showed that the cellulose possesses crystallinity, porosity, thermal stability, hydrophilicity, and biodegradability, thereby establishing it as an advanced, eco-friendly biomaterial for a range of sustainable pharmaceutical applications. This research provides a foundation for the improvement of the value of agricultural waste within the framework of circular bioeconomy practices. This research supports increasing agricultural waste value in the circular bioeconomy.

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