EXTRACTION AND CHARACTERIZATION OF α-CELLULOSE-RICH RESIDUE FROM MAIZE (*ZEA MAYS* L.) HUSK

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Agricultural waste leads to a number of environmental issues, including pollution and environmental degradation. In Nigeria, Zea mays husk is one of the most prevalent agricultural wastes, and it can be turned into a valuable resource of quality cellulose. The goal of this study was to establish a low-cost and sustainable chemical treatment method for isolating cellulose from Z. mays husk feedstock. A series of alkaline delignification, digesting, and bleaching techniques were used to extract and purify cellulose. TAPPI T203 OS-74, TAPPI T222 OS-83 and TAPPI T222 OM-02 methods were used to determine the cellulose, hemicellulose and lignin contents, respectively. The samples were also characterised by scanning electron microscopy, X-ray diffraction, Fourier transform infrared spectroscopy (FTIR), and CHNS/O analyses. The resulting product was found to contain 97.95% α-cellulose, 0.19% β-cellulose, and 1.86% γ-cellulose. The presence of 40.95% carbon, 2.98% hydrogen, 0.72% nitrogen, 0.07% sulphur, and 55.28% oxygen was found by the CHNS/O analysis of cellulose. The untreated husk microscopy displayed an uneven, flake-like, and nonuniform surface, whereas the delignified husk, digested husk, and pure cellulose (ZMH-C) micrographs revealed, respectively, a smooth non-uniform surface, an irregular porous surface, and a smooth wool-like surface. The FTIR spectra of the treated samples demonstrated an increase in the intensity of the polar property of the OH group, as well as the elimination of the hemiacetal group and β -1,4-glycosidic linkages. The ZMH-C diffractogram verified the existence of characteristic 20 peaks of cellulose at 220, 240, and 300, as well as a 4.7% crystallinity index. The comparatively low-temperature sequential alkaline delignification, digesting, and bleaching method adopted extracted low-lignin crystalline cellulose material from Z. mays husk. The flexibility, biodegradability, and availability of husk make it a viable source of high-quality cellulose with several possible applications. Z. mays cellulose has been thus demonstrated to be an appealing material for a wide variety of industries seeking environmentally acceptable and sustainable solutions.

Keyword: Zea mays husk, cellulose, hydrolysis, lignin, hemicelluloses, characterization

INTRODUCTION

The annual global output of agricultural waste, according to Ratna *et al.*,¹ is around 998 million metric tons. Agriculture is a vital industry that provides the world with food, fiber, and fuel. However, the production of agricultural products can also generate significant amounts of waste, which can have detrimental environmental consequences if not adequately managed.² Crop residue, animal dung, and food manufacturing wastes are all examples of agricultural waste.

Nigeria presently produces the highest quantity of maize annually among other African nations and is rated the 10th largest producer in the world, with an estimated production of over 33 million tons, followed by South Africa, Egypt, and Ethiopia.³ With such a big output, the country

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generates a substantial amount of maize husk waste. The outer layer of the maize grain, which is a rich source of lignocellulosic material, is normally removed during processing to reveal the edible section of the grain.⁴

In Nigeria, the production of maize husk waste generates a variety of environmental concerns. One of the primary worries is the possibility of water contamination. When maize husk waste is not adequately managed, it can get washed into neighboring bodies of water, where it decomposes and releases nutrients such as nitrogen and phosphorus. These nutrients can promote eutrophication, which can result in the growth of hazardous algal blooms and the depletion of oxygen in the water, both of which have severe consequences for aquatic life.⁵ Aside from water contamination, inappropriate disposal of maize husk waste can lead to air pollution and greenhouse gas emissions.⁶ When the waste is burned, it releases carbon dioxide, methane, and other pollutants into the atmosphere, contributing to climate change and negative health impacts for nearby communities.

There are opportunities to utilize maize husk waste in Nigeria. One possible application is as animal feed. Maize husk is high in fibre, which may be used by ruminant animals, such as cattle and sheep, but it can also serve as a substrate for the development of biofuels and other value-added products.^{7,8} Another approach is to develop local value chains for the utilization of the husk waste, such as by converting it into animal feed, cellulose or other value-added products.^{9,10} This can create economic opportunities for local communities, while also reducing the negative environmental impacts of the waste.^{7,11}

Cellulose is a complex carbohydrate that is found in the cell walls of all plants, including maize. It is made up of glucose units joined together by β -1,4-glycosidic bonds, which create a crystalline structure that resists enzymatic breakdown. Cellulose extracted from *Z. mays* is a highly renewable and sustainable resource. It is a natural polymer that is biodegradable and nontoxic, making it an eco-friendly alternative to synthetic materials.¹² Cellulose is extracted from the maize plant through a variety of processes, which remove lignin and increase the purity of cellulose.^{13,14}

Lignin restricts the ability of glycoside hydrolases,¹⁵ however, to effectively break down the feedstock, the lignin must be removed. Agricultural biomass has been subjected to chemical pretreatments with different acids, such as phosphoric acid, sulfuric acid and acetic acid, for this purpose.¹⁶ The use of acid pretreatment on agricultural feedstock successfully eliminates lignin, but also results in the loss of a significant amount of hemicelluloses. Moreover, certain acids, such as sulfuric acid, are not eco-friendly and require higher temperatures and longer durations (140 °C for 6 hours) to achieve the desired effect.¹⁷

Alternative, eco-friendly pretreatment technologies involving the use of fungi or bacteria to decompose lignin have been developed.¹⁸ According to Dubey et al.,¹⁹ their study involving the use of Coriolus versicolor in a solid-state fermentation resulted in the elimination of more than 40%w/w lignin from the solid-state biomass. Nunta et al.²⁰ reported that solid-state biomass underwent more than 60% lignin removal when treated with 6% w/v H2O2 within 4 hours, with 32% of hemicelluloses being solubilized in the same process. However, this pretreatment approach requires a longer duration, of several days, to effectively remove significant amounts of lignin and obtain pure cellulose.

One of the most established and relatively cost-effective methods for lignin removal or redistribution and mercerization of the biomass is alkaline pretreatment.²¹ A study by Ling *et al.*²² demonstrated that alkaline pretreatment resulted in increased biomass swelling, removal of lignin, and conversion of crystalline cellulose I to a less crystalline form known as cellulose II. This method is highly selective in separating lignin, operates under mild reaction conditions, allowing for the recovery and reuse of alkaline chemicals.²³

Due to its distinct qualities, Z. mays cellulose has a wide range of industrial applications. One of the most popular applications is in the manufacturing of paper and cardboard. The high cellulose content of Z. mays makes it an attractive raw material source for the paper industry.²⁴ The use of Z. mays cellulose in the paper industry helps to lessen dependency on trees, which is critical for forest protection.²⁵ Furthermore, Z. mays cellulose is utilised in textile production as a common element in the production of rayon and other non-synthetic fibres, reducing dependency on synthetic fibres, which are frequently derived from non-renewable petroleum resources.²⁶ Z. mavs cellulose has also been used as a food ingredient, most notably as a thickening in processed foods, such as ice cream, salad dressings, and sauces.²⁷ Cellulose is a low-calorie

component that adds bulk and texture to a food, without adding substantial calories. It is also employed as a nutritional fibre source in some meals, such as morning cereals.

Given the substantial environmental concerns associated with agricultural waste, particularly Z. mays waste, it is critical to consider it as a resource rather than a liability by developing a low-cost pretreatment method for the extraction of premium cellulose. This would consequently lead to minimising waste and promoting resource conservation, while protecting the environment and human health.

EXPERIMENTAL

Figure 1 is a flowchart of the husk processing techniques used in this work, leading to the formation of an α -cellulose rich material. This was followed by the physico-chemical characterization of the product.

Collection and identification of samples

The husk used in the study was obtained from a nearby plantation situated in Ado Kasa, latitude 9.0128 and longitude 7.6314, Karu LGA, Nasarawa State, Nigeria. The sample was identified and assigned a code: NIPRD/H/7317.

Sample preparation

The husk was oven dried at 100 °C for 4 hours and ground into powder using a USHA Mixer Electric Grinder (MG2053N, India). The husk was reduced to 50 mesh particle size, using American Society for Testing and Materials (ASTM) standard sieves. The powdered husk was then stored in moisture-free and air-tight plastic containers.

Delignification of the sample

Husk cellulose was delignified by the modified method of Akerele and Okhamafe.²⁸ Exactly 200 g of the powdered husk was treated with 500 mL of a 4%w/v NaOH solution at 80 °C for 12 hours, after which, the mixture was filtered with Whatman filter No.1. The obtained residue was washed with distilled water, then dried at 60 °C for 6 hours and labeled "delignified *Z. mays* husk" (De-ZMH).

Digestion of the sample

Digestion of De-ZMH was performed according to the modified method of Akerele and Okhamafe.²⁸ Exactly 180 g of the De-ZMH was further delignified as described above. The residue obtained was treated with 175 mL of 17.5% w/v NaOH solution at 80 °C for 1 hour. It was then filtered and washed several times in distilled water. The obtained residue was then dried at 60 °C for 6 hours and labeled "digested *Z. mays* husk" (Dig-ZMH).

Bleaching of the sample

The bleaching of Dig-ZMH was also carried out based on the modified method of Akerele and Okhamafe.²⁸ Exactly 150 g of Dig-ZMH was used. The residue obtained was further treated with 175 mL of 17.5% w/v NaOH solution at 80 °C for 1 hour to digest the powdered materials.

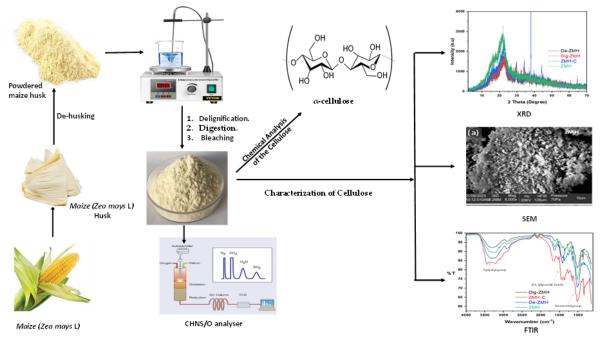


Figure 1: Graphical summary of α-cellulose extraction

The resulting slurry was filtered and the residue was thoroughly washed with distilled water and further treated with 3.2% NaClO solution for 20 min at 80 °C to bleach the residue.²⁹ The residue obtained was further washed with 5 L of distilled water to pH 7. The residue was filtered and the water was manually removed to obtain a small lump, which was dried at 60 °C for 6 hours and labeled "*Z. mays* husk cellulose" (ZMH-C).³⁰

Chemical analysis of the cellulose

The chemical constituents of the husk in the NaOH and NaClO stages of treatments were determined according to Technical Association of the Pulp and Paper Industry (TAPPI) standards. Cellulose, hemicelluloses and lignin were determined by TAPPI T203 OS-74, TAPPI T222 OS-83/TAPPI T203 OS-74, and TAPPI T222 OS-83 protocols.³¹

Determination of alpha (α)-cellulose content

A total of 25 mL of the ZMH-C filtrate and 10 mL of 0.5N K₂Cr₂O₇ solution were carefully transferred into a 250-mL flask. The flask was then swirled, while 50 mL of concentrated H₂SO₄ was added cautiously. The solution was left to stand for 15 min, then 50 mL of water was added and cooled at 25 °C, then 2 to 4 drops of ferroin indicator were added and titrated with 0.1*N* ferrous ammonium sulfate solution to a purple color. A blank titration substituting the ZMH-C filtrate with 12.5 mL of 17.5% NaOH and 12.5 mL of water was also performed.³²

The α -cellulose content of ZMH was calculated using Equation (1):

$$\alpha \text{-cellulose \%} = 100 - \frac{6.85(V2 - V1) \times N \times 20}{A \times W}$$
(1)

where V1 – titration of ZMH-C filtrate (mL), V2 – blank titration (mL), N – exact normality of the ferrous ammonium sulfate solution (0.1N), A – volume of the ZMH-C filtrate used in the oxidation = 20 mL, and W– oven-dry weight of ZMH specimen.

Determination of gamma (y)-cellulose content

Approximately 50 mL of the ZMH-C filtrate was pipetted into a 100-mL graduated cylinder having a ground glass stopper. Then, 50 mL of 3N H₂SO₄ was added and mixed thoroughly by inverting the cylinder submerged in a hot water bath and heated to 90 °C for 10 minutes to coagulate the β -cellulose. The precipitate was left to settle for several hours, then filtered, to obtain a clear solution. Then, 50 mL of the clear solution and 10 mL of 0.5N K₂Cr₂O₇ were pipetted into a 300-mL flask, and 90 mL of concentrated H₂SO₄ was cautiously added; the solution was maintained at 35 °C for 15 min. Then, a blank titration was carried out by substituting the solution with 12.5 mL of 17.5% NaOH, 12.5 mL of water and 25 mL of 3N H₂SO₄.³³ Percent y-cellulose was determined by using Equation (2):

$$\gamma - cellulose, \% = \frac{6.85[(V4-V3) \times N \times 20]}{25 \times W}$$
(2)

where V3 – titration volume of solution after precipitation of gamma (γ)-cellulose, V4 – blank titration (mL), and W – oven-dried weight of the sample (g).³⁴

Determination of beta (β) -cellulose content

The β -cellulose content of ZMH was calculated using Equation (3):

 β -cellulose % = 100 - (α -cellulose % + γ -cellulose %)(3)

Characterization of Z. mays cellulose sample Scanning electron microscopy (SEM)

The analysis was performed using a Jeol JSM-6400 scanning electron microscope to observe the surface morphology of husk at different stages of treatment. The samples were air-dried and coated with gold to avoid charging. The images were taken with an accelerating voltage of 15 kV.³⁵

Fourier transform infrared spectroscopy (FTIR)

Samples were analyzed using a Spectrum One FTIR spectrophotometer. FTIR spectral analysis was performed in the transmittance mode range of 4000-400 cm^{-1.35}

X-ray diffraction (XRD)

The crystallinity of the husk after different treatments was determined using an X-ray diffractometer (Bruker D-8 Discover) with CuK radiation ($\lambda = 0.1542$ nm). The scanning range and the scanning speed were 5-40° and 5 deg/s, respectively.³⁶ The deconvolution of the peaks from diffractograms was performed with the PeakFit 4.11 software. The surface method estimates the crystallinity index of the samples, by the following equation:

crystallinity index(%) =
$$\left(\frac{sc}{st}\right)100$$
 (4)

where Sc – area of the crystalline domain and St – area of the total domain.

RESULTS AND DISCUSSION

Chemical analysis and cellulose analysis of treated and untreated maize husk

The chemical composition of the samples at each stage of the treatment was determined and summarized in Table 1. The cellulose content of the untreated *Z. mays* husk was 73.30%, while the hemicellulose and lignin contents were 3.30% and 17%, respectively.

De-ZMH showed a high cellulose content (76%), relatively low hemicelluloses (2.41%), and a significant reduction in the lignin (6.2%) content, compared to the untreated sample. According to Geng *et al.*,³⁷ the delignification of a cellulosic sample reduces the amount of lignin in

the sample, leading to an increase in the relative proportion of cellulose and hemicelluloses. The digested sample (Dig-ZMH) had a slightly higher cellulose content (89.73%), indicating the further breakdown of the hemicellulose and lignin components.³⁸ Lastly, ZMH-C had the highest cellulose content of 97.95%, with the hemicellulose and lignin contents further reduced to 1.86% and 0.2%, respectively. The purpose of bleaching is to improve the brightness, purity, and visual appearance of the cellulose-rich material by removing impurities and colorants. However, bleaching typically leads to a reduction in lignin and hemicellulose contents, while the cellulose content remains relatively stable.³⁹ Generally, the results demonstrate that the different treatments applied to the husk successfully altered its chemical composition.40

The characteristic type of cellulose in the husk sample at different stages of the treatment is summarized in Table 2. The table provides information on the analysis of cellulose in different samples. The table shows that untreated sample (ZMH) has the highest α -cellulose content (73.30%), followed by β -cellulose (23.40%) and γ -cellulose (3.30%). This is expected since α cellulose is the most crystalline form of cellulose, which is resistant to chemical and enzymatic degradation. β -cellulose is less crystalline and more susceptible to degradation than α -cellulose, while γ -cellulose is the least crystalline form. The De-ZMH has a higher α -cellulose content (76.00%), lower β -cellulose (21.59%) and γ cellulose (2.41%) contents compared to the untreated sample.

This is consistent with the report of Schmetz et al.,⁴¹ according to which delignification tends to increase the relative proportion of cellulose, including α -cellulose, within the material, because delignification removes lignin, and makes it more accessible to chemical and enzymatic treatments. The β -cellulose content decreased significantly (8.19%), while γ -cellulose remained relatively constant (2.08%). This finding is confirmed by the fact that the crystalline structure of β -cellulose renders it more susceptible to disruption and alteration by chemical treatments, whereas the amorphous form of y-cellulose gives some resistance or protection against chemical reagents.⁴² The ZMH-C has the highest α cellulose content (97.95%) and the lowest β cellulose content (0.19%) among all the samples. This is because bleaching removes residual lignin, hemicelluloses, and other impurities, leaving behind highly purified cellulose.

Sample	Cellulose (%)	Hemicelluloses (%)	Lignin (%)	
ZMH	73.30	3.30	17	
De-ZMH	76.00	2.41	6.2	
Dig-ZMH	89.73	2.08	0.4	
ZMH-C 97.95		1.86	0.2	

Table 1 Chemical analysis of husk samples at each stage of treatment

 Table 2

 Cellulose analysis of husk samples at each stage of treatment

Sample	α-cellulose (%)	β-cellulose (%)	Υ-cellulose (%)
ZMH	73.30	23.40	3.30
De-ZMH	76.00	21.59	2.41
Dig-ZMH	89.73	8.19	2.08
ZMH-C	95.90	0.19	1.86

Table 3

Percentage CHNSO analysis of husk samples at each stage of treatment

Sample	C (%)	H (%)	N (%)	S (%)	O (%)
ZMH	40.79	2.39	0.31	0.14	56.37
De-ZMH	42.32	3.13	0.78	0.10	53.67
Dig-ZMH	43.16	2.91	0.56	0.07	53.30
ZMH-C	40.95	2.98	0.72	0.07	55.28

CHNS/O analysis of treated and untreated maize husk

The analysis provides information on the relative amounts of carbon (C), hydrogen (H), nitrogen (N), sulphur (S), and oxygen (O) present in the husk sample and the finding is reported in Table 3. It was observed that the carbon content of the sample gradually increases with the following treatments: delignification, digestion and bleaching. ZMH-C has the highest carbon content (43.16%), followed by Dig-ZMH (42.32%), De-ZMH (40.95%), and ZMH (40.79%).

Similarly, the nitrogen content also increased at each stage of the treatment, with ZMH having the lowest nitrogen content (0.31%) and ZMH-C having the highest nitrogen content (0.72%). The sulphur content of the husk is low (≤ 0.1) across all samples, with the highest sulphur content being observed in the untreated ZMH. The oxygen content is high in all the samples, ranging from 53.30% to 56.37%. This is in consonance with other studies by Iram et al.,43 Díez et al.,44 and More et al.,⁴⁵ which have shown that, during delignification, the lignin is broken down or dissolved, while digestion primarily affects the removal of hemicelluloses, and bleaching can oxidise and further degrade lignin, generally resulting in a reduction in the carbon content. This contrasting observation may be due to the specific effects of delignification, digestion, and bleaching on the carbon, hydrogen, nitrogen,

sulphur, and oxygen content of a sample, as well as the process parameters, chemicals used, and the starting composition of the raw material.

Characterization of treated and untreated maize husk

Figures 2 and 3 display the scanning electron micrographs of the treated and untreated maize husk samples recorded at magnifications of x8000, 9000, and 10,000. The SEM images reveal that ZMH (Fig. 2 (a-c)) has an irregular, flakelike, and non-uniform material, which can be attributed to the presence of complex inorganic and organic molecules, and relatively high proportions of β -cellulose and γ -cellulose.⁴⁶ However, the morphology of ZMH changed after undergoing chemical delignification; resulting in the formation of smooth, but non-uniform surface (Fig. 2(d-f)). This is strongly linked to the substantial elimination of the outer non-cellulosic molecules made up of hemicelluloses and lignin, as reported in Table 1, and other impurities, such as pectin and wax, present in ZMH.47

Digestion of the delignified husk (De-ZMH) facilitates the defibrillation and opening of the fiber bundles, as evidenced in Figure 3 (b),⁴⁸ resulting in the production of an irregular porous surface. Furthermore, bleaching the sample causes the fiber bundles to break down and leads to the formation of a wool-like surface (Fig. 3 (d-f)).

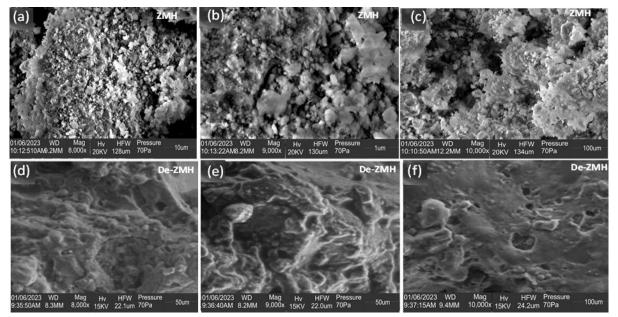


Figure 2: SEM micrographs of ZMH and De-ZMH samples

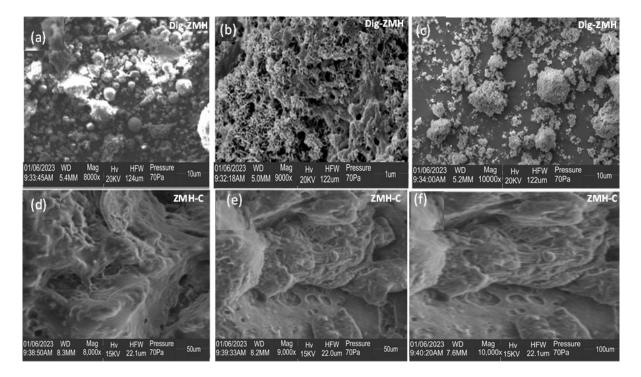


Figure 3: SEM micrographs of Dig-ZMH and ZMH-C samples

The fiber breakdown suggests that, practically, all of the components binding the corn husk fibril structure were eliminated during the rigorous chemical treatment.⁴⁹

Figure 4 depicts the samples' FTIR spectra. In general, the existence of hydroxyl group (-OH) stretching vibration (3200 to 3600 cm⁻¹) was seen in the samples throughout the treatment phases; however, the intensity increased after the alkaline digesting phase. This is expected because alkaline digestion of cellulose decreases the polarity of the hydroxyl groups by deprotonating them and forming alkoxide ions. The disruption of the cellulose structure during digestion also reduces the extent of hydrogen bonding, leading to a decrease in overall polarity.⁵⁰ The delignification and bleaching stages, on the other hand, induced an increase in intensity, strengthening the polar features of the OH group. This is because alkaline delignification and bleaching remove the nonpolar lignin from cellulose fibers, increasing the relative proportion of polar cellulose in the sample. This removal enhances the exposure and accessibility of the hydroxyl groups in cellulose, leading to an increase in their polarity and the overall polarity of the cellulose sample.⁵¹

Most notably, there was no breakdown or modification of the hemiacetal group (1000 to 1100 cm⁻¹) and β -1,4-glycosidic bonds (1200 cm⁻¹)

¹) in husk cellulose. This is a confirmation of the cellulose integrity during the treatment process.

Figure 5 shows the X-ray diffractograms of the sample at different stages of the treatment. Cellulose was identified by the 2 θ peaks at 220, 240, and 300, which represented the HKL of 148 ×10⁻³, 109 × 10⁻³, and 156 × 10⁻³ crystallographic planes, as depicted in Figure 5.^{52–57} The peaks became more distinct in ZMH-C after the removal of non-cellulosic components from ZMH through chemical treatment.

Figure 6 shows the crystallinity index of the sample at different stages of the treatment. The crystallinity index is a measure of the degree of crystallinity in a material, with higher values indicating a more ordered and crystalline structure.⁵⁸ ZMH-C exhibited the highest crystallinity index, with the sharpest and strongest peak at 2θ of 390. The results show that ZMH, De-ZMH, and Dig-ZMH have a comparatively low crystallinity value, indicating a greater number of amorphous areas and a lower level of This is because of the high orderliness. of β -cellulose and γ -cellulose proportions observed in ZMH, De-ZMH, and Dig-ZMH, which is in consonance with the report of Shaikh et al.,⁵⁹ which revealed that a high proportion of β -cellulose and γ -cellulose may result in a more amorphous and irregular surface morphology

characteristic. This has implications for the material's qualities, reactivity, and behaviour, since amorphous parts differ in structure and

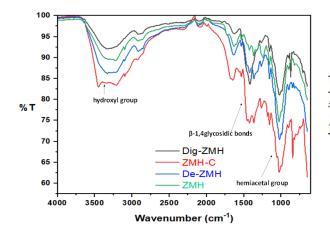


Figure 4: Functional analysis of treated and untreated maize husk samples

features from more ordered crystalline sections.^{59,60}

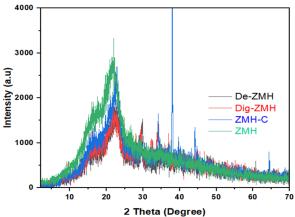


Figure 5: X-ray diffractograms of treated and untreated maize husk samples

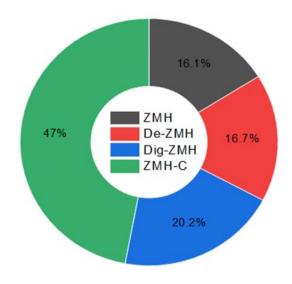


Figure 6: Crystallinity index of cellulose

The removal of lignin has a positive effect on the sample's degree of crystallinity; however, there was a decrease in the sample's crystallinity index by 3.5%. The crystallinity index of ZMH-C was higher (47%) compared to other samples, indicating its higher amount of α -cellulose, which may contribute to well-defined crystalline regions and the removal of impurities largely associated with the amorphous characteristic. Specifically, digestion and bleaching cause a substantial increase in the degree of crystallinity, which may have implications for the properties and potential uses of the material. The increase in crystallinity of the treated husk, compared to the untreated one, was attributed to the enhanced elimination of amorphous non-cellulosic constituents. The increase in crystallinity was also expected to improve the rigidity, leading to better mechanical properties and reinforcing capability of the material.⁶⁰

CONCLUSION

Chemical processes, notably delignification, digestion, and bleaching at relatively low temperature, were effectively used in this study to extract crystalline material from *Z. mays* husk. The cellulose was further investigated and found to have very low hemicellulose and lignin contents. It was discovered to have high cellulose content, as well as a high crystallinity index. Due

to its high crystalline content and lignin-free nature, the obtained cellulose is expected to have high mechanical strength, barrier qualities, and controlled release capabilities, which would make it a desirable material in a wide range of sectors, from composites and packaging to textiles and biomedical applications. Furthermore, its high crystallinity, purity, and specific characteristics due to its α -cellulose-rich content make it a valuable material in various industries, ranging from pharmaceuticals to cosmetics to specialty papers. The wide range of potential applications for this α -cellulose-rich residue makes the husk a valuable material to be valorized due to its versatility, biodegradability and abundance. Z. mays husk is thus recommended as an attractive material for various industries seeking sustainable and eco-friendly solutions.

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