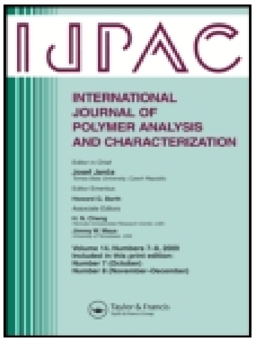


International Journal of Polymer Analysis and Characterization

ISSN: 1023-666X (Print) 1563-5341 (Online) Journal homepage: http://www.tandfonline.com/loi/gpac20

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**To cite this article:** C. E. Maepa, J. Jayaramudu, J. O. Okonkwo, S. S. Ray, E. R. Sadiku & J. Ramontja (2015) Extraction and Characterization of Natural Cellulose Fibers from Maize Tassel, International Journal of Polymer Analysis and Characterization, 20:2, 99-109, DOI: 10.1080/1023666X.2014.961118

**To link to this article:** http://dx.doi.org/10.1080/1023666X.2014.961118

Accepted author version posted online: 11 Sep 2014.
Published online: 11 Sep 2014.

*International Journal of Polymer Anal. Charact.*, 20: 99–109, 2015

ISSN: 1023-666X print/1563-5341 online
DOI: 10.1080/1023666X.2014.961118

# Extraction and Characterization of Natural Cellulose Fibers from Maize Tassel

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This article reports on the extraction and characterization of novel natural cellulose fibers obtained from the maize (tassel) plant. Cellulose was extracted from the agricultural residue (waste biomaterial) of maize tassel. The maize tassel fibers were obtained after treatment with NaOH and were carefully characterized while the chemical composition was determined. The chemical composition of the maize tassel fibers showed that the cellulose content increased from 41% to 56%, following alkali treatment. FT-IR spectroscopic analysis of maize tassel fibers confirmed that this chemical treatment also shows the way to partial elimination of hemicelluloses and lignin from the structure of the maize tassel fibers. X-ray diffraction results indicated that this process resulted in enhanced crystallinity of the maize tassel fibers. The thermal properties of the maize tassel fibers were studied by the TGA technique and were found to have improved significantly. The degradation temperature of the alkali-treated maize tassel fiber is higher than that of the untreated maize tassel fibers. This value convincingly showed the potential of maize tassel fibers for use in reinforced biocomposites and waste water treatment.

Keywords: Chemical composition; Infrared spectroscopy; Maize tassel fiber; Thermal properties; X-ray diffraction

## INTRODUCTION

Cellulose is a renewable raw material, with a linear polymer of $\beta$-D-glucose, which is the main component. As would be expected, it is an abundant biomass resource. It has been estimated that the yearly biomass production of cellulose is 1.5 trillion tons. It is an unlimited source of raw material, eco-friendly, and biocompatible for the production of green products.[1–3] Producing new sustainable and eco-friendly materials has gained the attention of researchers worldwide.

Submitted 15 August 2014; accepted 21 August 2014.
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It includes a search for alternatives to petroleum-based materials. Advances in biomaterials hold enormous assurance in the quest to solve sustainability problems and contribute to renewability, biodegradability, and a pathway to reducing hazardous materials.[4] This report focuses on the growth of specialized techniques for the manufacture of natural fillers as reinforcing agents designed for biocomposite applications. Chemical composition, structure, and physical and thermal properties[5–7] of fibers/fabrics can be used to estimate and approximate their capabilities,[8–11] hence maize tassel can be used as protective material (filler) in biocomposite applications.

In the past two decades, investigation focusing on the use of cellulosic waste as filler has grown steadily. Agricultural waste fibers, such as: cornstalks,[12] wheat, straw, and soy hulls,[13] coconut husk fibers,[14] cassava bagasse,[15] banana rachis,[16,17] and soybean pods[18] have been considered as resources in making crystalline cellulose fibers. Although a selection of natural fibers has been studied in detail, the use of maize tassel as a natural source waste biomaterial for the production of cellulose biocomposites has not been widely explored yet.

Maize is a well-known cereal throughout the world. It is a yearly monoecious grass, grown mainly for feed, food, and industrial raw materials. It is also an important resource of waste raw material. A material that would be considered an ideal candidate should be of low price, locally available in large quantities, and easily regenerated or discarded thereafter with minimal

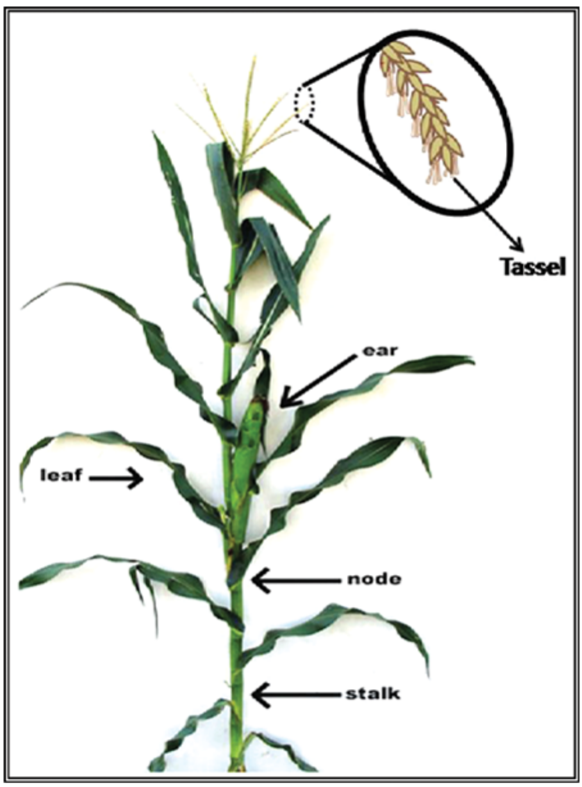


FIGURE 1 Photographs of maize plant with its tassel on top of the stem.

environmental impacts. Maize tassel, a waste biomaterial, is the ''male'' flower of the maize plant that forms at the top of the stem (a photograph of the maize tassel is shown in Figure 1).

The maize tassels have shades of yellow, green, and purple. Each maize plant grows these tassels on top of the plant after the major growth of the plant is complete. Maize tassel produces pollen that falls off and is blown off by the wind. Maize tassel grows at the apex of the maize stalk and ends up as agricultural waste product after being involved in maize fertilization. It is discarded by farmers in large quantities with the rest of the plant, once the crops have been harvested. The authors have not so far seen any information in the literature on the physical characteristics or chemical composition of tassel. As a fibrous part of a plant that is rich in carbohydrates, it is expected to contain a high quantity of polysaccharides with the cellulosic surface containing hydroxyl groups and residual aromatic compounds, which give it some scent during its vegetative stage. The hydroxyl, carbonyl, conjugated bonds, and amine functional groups on its surface can provide possible binding sites for metal cations and oxoanions.

The aim of this study was to extract cellulose fibers from maize tassel, via alkali treatment. The effects of alkali treatment on the characterization of the maize tassel fibers were studied. Characterizations, such as chemical analysis, FT-IR, SEM, TGA, and X-ray diffraction (XRD) patterns of maize tassel fibers, were carried out.

## EXPERIMENTAL SECTION

### Materials

The maize tassel used as raw material was obtained from the Tshwane University of Technology research farm in Pretoria, South Africa. Sodium hydroxide (99% purity, purchased from Merck Ltd., Mumbai, India) was used for the alkaline treatment. In the present study, benzene, sodium chlorite, acetic acid, sodium bisulfite, and ethanol (all from S.D. Fine Chemicals Ltd., Mumbai, India) were also used for chemical analyses.

### Extraction and Treatment of Maize Tassel Fibers

Maize tassels that form at the top of the stems were used for extraction of the male inflorescence of the maize plant. They were soaked in water for one day and some water-soluble materials dissolved. Next, the separated maize tassels were thoroughly washed with tap water, followed by distilled water, and they were subsequently dried in the sun for one week. Thereafter, the maize tassels were kept in a hot air oven for 24 h at a temperature between 105° and 110°C in order to remove any residual moisture. Some maize tassels were treated with 5% aqueous sodium hydroxide (NaOH) solution, at room temperature, maintaining a liquor ratio of 20:1 for 45 min in order to remove the hemicellulose and other greasy materials. Thereafter, the maize tassels were repeatedly washed with water and treated with dilute acetic acid in order to neutralize them. Finally, the tassels were washed with distilled water before drying in a hot air oven for 24 h.

### Chemical Analysis

Chemical analysis of the untreated and treated tassel fibers was carried out as per the standard procedure from our previous work.[19] In this analysis, the percentage values of α-cellulose, hemicellulose, and lignin were determined. In each case, the average of five tested samples was reported.

### Fourier Transform-Infrared (FT-IR) Spectroscopy

FT-IR spectra were used to examine the structure of maize tassel fibers that were obatained after alkali treatment. A Nicolet 560 spectrophotometer was used to obtain the spectra of each sample. The untreated and treated maize tassel fibers were grounded and mixed with KBr powders and the mixture was compressed into plates for FT-IR analysis. The FT-IR spectra of the samples were obtained in the wavelength range of 4000–400 $cm^{-1}$. A total of 32 scans were co-added in order to achieve an acceptable signal-to-noise ratio. In all cases, spectra resolution was maintained at 4 $cm^{-1}$.

### Crystal Structure Measurements

The crystallinities of untreated and treated maize tassel fibers were examined by using the wide-angle X-ray diffraction (WAXD) technique, with $CuK_\alpha$ radiation ($\lambda = 1.54$ Å), with a computerized data acquisition facility and analytical tools. The X-ray source was operated at a voltage of 40 kV and a filament current of 40 mA. All samples were scanned in the $2\theta$ range between 5 and 50°, at a rate of 1°/min in order to obtain an acceptable diffraction pattern.

### Morphology

The morphology of the maize tassel fibers was examined using a scanning electron microscope (SEM; Quanta 200, USA). Gold sputter-coated samples were examined using a Cambridge Stereoscan 250 with an accelerating voltage of 10 kV.

### Thermal Stability

Thermogravimetric analysis (TGA) was performed in order to compare the degradation characteristics of the maize tassel alkali-treated fibers with untreated fibers. The thermal stability of each sample was determined using a TGA Q500 series Thermogravimetric analyzer (TA Instruments, USA), at a heating rate of 10°C/min in a nitrogen environment.

## RESULTS AND DISCUSSION

### Effect of Alkali Treatment on the Structure and Morphology of the Fibers

In order to confirm the chemical structures of untreated and alkali-treated maize tassel fibers, FT-IR analysis was performed. The composition changes observed for untreated and alkali-treated maize tassel fibers are shown in Figure 2. The spectra show the presence of hydroxyl, carbonyl, ether groups, and absorbed water in the untreated maize tassel fiber. Important changes in the FT-IR spectra are associated with the peak intensities at around 1730, 1240, 1500, 1625, and 896 $cm^{-1}$. As

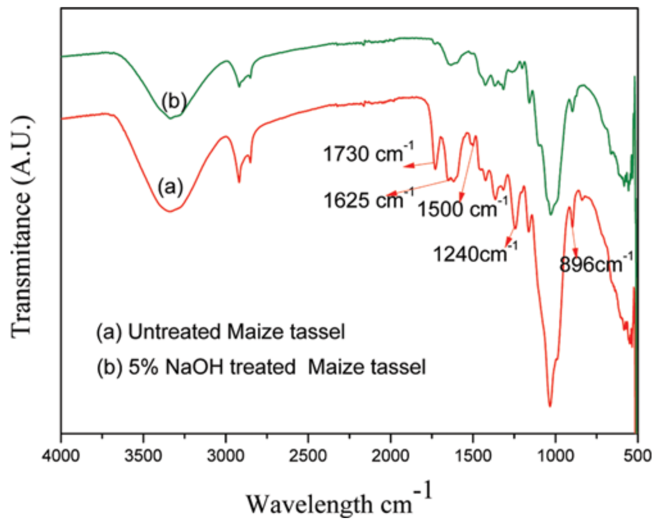


FIGURE 2 FT-IR spectra of maize tassel fiber: spectrum (a) untreated and (b) alkali-treated (NaOH) maize tassel fibers.

shown in Figure 2, the hemicellulose intensity of the peak occurred[13,20–24] at around 1730 and 1240 $cm^{-1}$ in the untreated tassel, and the spectrum is not visible in the alkali-treated tassel, which indicates that the hemicelluloses were removed completely by the alkali treatment. This is quite expected as hemicellulose is soluble in aqueous NaOH (alkali) solution.[6,23] The band C=C in the tassel fiber represents the aromatic vibration at 1500 $cm^{-1}$ from methoxyl groups of lignin.[13] This band was significantly reduced in the alkali-treated sample because of the removal of most of the hemicellulose and lignin from the tassel fiber through treatment. The results indicate that the intensity of the peak at around 1625 $cm^{-1}$ is attributed to the absorbed extra moisture on the surface of the fiber after alkali treatment.[19] The peak at around 896 $cm^{-1}$ (due to the $\beta$-glucosidic linkage) was attributed to the O-C-O stretching during the C–H deformation of cellulose.[20] Finally, the result indicates that there were no considerable changes noticed in the intensity of other peaks in the FT-IR spectra. The intensity of the peaks around 3339 and 2921 $cm^{-1}$ are associated with the $\alpha$-cellulose (O-H) stretching. Finally, the FT-IR results indicate that the hemicelluloses and lignin were removed from the maize tassel fibers during alkali treatment.

## Chemical Composition of Fibers

The chemical composition of lignocellulosic fibers is intrinsic to the exact requirements of each plant. Cellulose, hemicellulose, and lignin are the three major constituents of any lignocellulosic resource, and the quantities of these constituents in a fiber depend on the age, source of the fiber, and process of fiber extraction.[25] The composition of the fibers can be estimated by chemical analysis techniques, as reported in the literature.[26] The chemical composition of maize tassel fibers and the crystallinity index were determined for the untreated and alkali-treated tassels, and representative data are summarized in Table I. From the table, it is obvious that the chemical composition of the treated sample was, to a great extent, influenced by the alkali treatment. The

TABLE I
Chemical analysis and crystallinity index of the untreated and alkali-treated (NaOH) maize tassel fibers

| *Maize tassel* | *Cellulose (%)* | *Hemicelluloses (%)* | *Lignin (%)* | *Crystallinity index (%)* |
|---|---|---|---|---|
| Untreated fibers | 41 | 29 | 18 | 85.48 |
| Alkali-treated (NaOH) fibers | 56 | 10 | 15 | 88.41 |

lignin and hemicellulose contents decreased with alkali treatment, while the percentage cellulose content increased. Comparable data were also observed in the cases of corn stover, wheat straw, rice straw, sorghum stalks, and barley.[27–31] This is in conformity with the explanation advanced in the FT-IR analysis.

## Fiber Surface Morphology: SEM Analysis

The aim of this section is to explain the SEM results obtained for untreated and alkali-treated maize tassel fibers in relation to the morphological structure shown in Figure 3. As can be seen from Figures 3(a)–(c), the untreated maize tassel fibers have a relatively smooth surface, with linear ridges and furrows on the surface of the fiber. It is shown that the surface of the untreated fibers was composed mainly of lignin, pectin, ash, and hemicellulose that enclosed the cellulose fibers.[19] As can be seen in Figure 3(c), the epidermal cells are arranged in linear ridges and furrows, and the ridges are punctuated on the surface of the morphology with hair-like fibers.[32]

Figure 3(d)–(f) shows the morphological structure of the alkali-treated maize tassel fibers. As seen in these figures, most of the surface substances were removed following fiber treatment with alkali at room temperature, resulting in higher roughness and clean fiber surface, and the size (diameter) of the fibers might have been reduced due to the removal of most hemicellulose, which was hydrolyzed and became water soluble, and, in addition, the lignin was partially

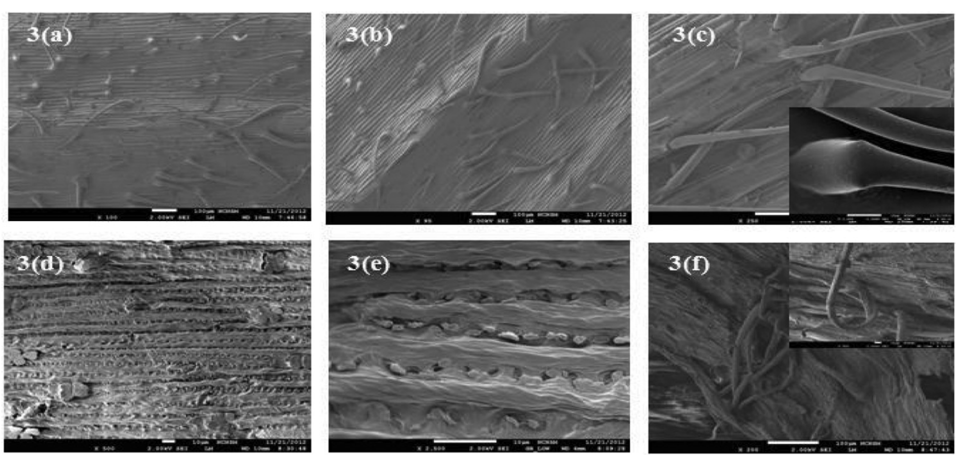


FIGURE 3 SEM micrographs of untreated and alkali-treated maize tassel fiber: (a)–(c) untreated, (d)–(f) alkali-treated (NaOH) fibers at different magnifications.

depolymerized by alkali treatment. This was further supported by the chemical analysis data, given in Table I. Comparable studies have also been reported for some other natural cellulose fibers.[6,20,24] In addition, the surface of maize tassel alkali-treated fibers shows that the epidermal cells were broken at the top of the surface of the hair-like fibers, resulting in marginal brightness in this region of the fiber, which can be attributed mainly to the elimination of waxes, the cementing materials (refer to Figure 3(f)). As a result, fiber size decreased and the rough surface area can easily enhance the chances of an increase in the extent of bonding in a polymeric matrix-fiber interface suitable for the manufacture of green composites.

## X-ray Diffraction Analysis

The XRD patterns of the maize tassel fibers showed that they are in a crystalline state. The X-ray diffractograms of untreated and alkali-treated maize tassel fibers can be seen in Figure 4. In the untreated tassel, crystalline cellulose components are oriented within the amorphous materials of lignin, hemicellulose, pectin, and waxes. For the duration of alkali treatment, the amorphous materials were dissolved, leaving pure crystalline cellulose. In natural cellulose fibers, the regions of intermediate order in the structure play an important role in the determination of the extent of crystallinity. As can be seen in Figure 4, the XRD patterns of tassel fibers show a confirmation of two well-defined peaks at $2\theta = 16.2^\circ$ and $22.5^\circ$. The $2\theta$ reflections correspond to the (110) and (002) crystallographic planes, respectively. The prominent reflection in accredited to crystalline component ($I_{002}$) arising from the α-cellulose. The crystallinity index of the tassel fiber was determined[34] by using the following equation:

$$(X_c) = \left[\frac{I_{(002)} - I_{(110)}}{I_{(002)}}\right] \times 100 \tag{1}$$

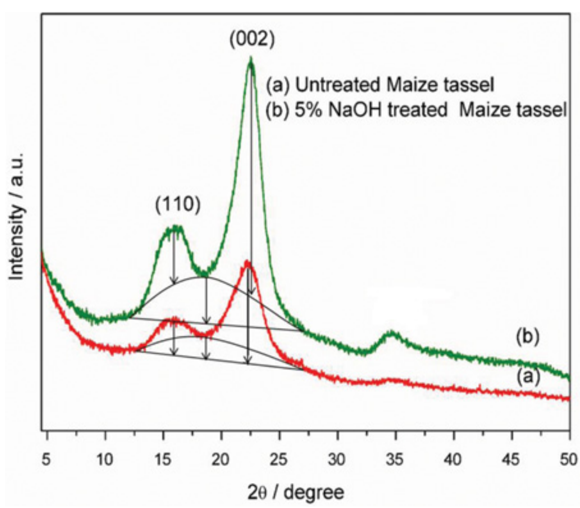


FIGURE 4 X-ray diffraction patterns of (a) untreated and (b) alkali-treated (NaOH) maize tassel fibers.

where $I_{(002)}$ $(2\theta = 22.5°)$ and $I_{(110)}(2\theta = 16.2°)$ represent the intensities of the crystalline and amorphous peaks in the crystallographic planes.

The broad peak at $2\theta$, close to 16.2°, corresponds to the (110) crystallographic plane. When the crystalline cellulose fraction is high in the fibers, the peak is more evident and as the fiber is enclosed in large amorphous material, such as lignin, hemicelluloses, pectin, and amorphous cellulose, the peak is smeared and becomes one broad peak.[33–36] As can be seen in Figure 4, the peak at 16.2° is better defined for the alkali-treated maize tassel fiber, thereby signifying that the alkali treatment eliminated significant amounts of the amorphous materials from the maize tassel fiber.

On the other hand, the major crystalline peak observed in the pattern occurred at around a $2\theta \sim 22.5°$, which corresponds to the (002) plane. The X-ray diffractograms show that the intensity of the (002) crystallographic plane was significantly increased following alkali treatment of the maize tassel fiber. As can be seen in Figure 4, the crystallinity index of the maize tassel fiber increased with alkali treatment. This is due to the removal of the protective material and the possible relaxation of stress in the cellulose chains as a result of the removal of its amorphous constituents and pectin from the fibers.[37–39] Consequently, the calculated crystallinity indexes for the untreated and alkali-treated fibers showed an increase from 85.48% to 88.41%, respectively; the results are summarized in Table I. The increase in crystallinity in the treated fibers is due to the loss of the amorphous hemicelluloses. This increase, a result of alkali treatment, might be the main causative factor for the improvement in the thermal properties of tassel fibers (refer to Figure 4). This is consistent with the results of FT-IR and chemical analyses.

## TGA Analysis

Thermal properties of this material are very important in order to determine its possible application in polymer-tassel reinforced biocomposites. This is because the processing temperature normally considered for many polymeric materials is below 240°C. Figure 5 shows the TGA thermograms of maize tassel fibers (untreated and alkali-treated). The thermogram is divided into three regions. Region I, the initial weight loss region (initial shoulder peak), occurred between 205° and 278°C, and this is due to the decomposing hemicelluloses. Region II, the most significant weight loss with its decomposition peak occurring between 278° and 346°C, relates to the thermal depolymerization of the remaining hemicelluloses, lignin, and cellulose. Region III, the third phase of weight loss, occurring between 346° and 700°C, is attributed to the additional rupture of the cellulose glucosidic chains and the nonliving compounds in the material.[21,40,41] As can be seen in Figure 5, it is evident that the degradation temperature significantly increased after alkali treatment. From these thermograms, it is very apparent that the thermal stability of the alkali-treated maize tassel fibers is higher than that of the untreated maize tassel fibers. The decrease in the amorphous hemicellulose and lignin contents of the fibers following alkali treatment may be the explanation for this behavior. Similar results were obtained in a previous study on fibers/fabrics.[6,13,22–24,42] As an end result, fibers became more hydrophobic and will improve the possibility of adhering to the polymer matrix when added to a polymer, as reinforcement. This will be the subject of a subsequent study on a polymer-tassel biocomposite

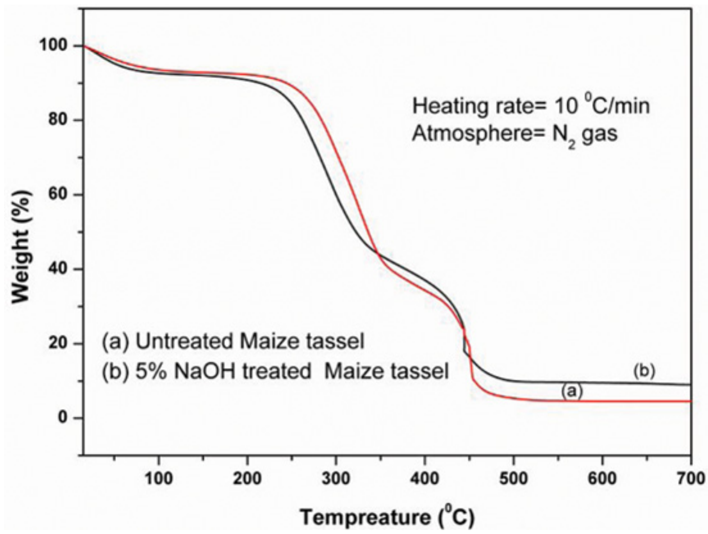


FIGURE 5 TGA thermograms of (a) untreated and (b) alkali-treated (NaOH) maize tassel fibers.

for possible water treatment (the fibers show potential use as an activated carbon for waste water treatment), automotive, and structural applications.

## CONCLUSIONS

A residue from agricultural plant, such as the by-product of maize (tassel fibers), is obviously an attractive substitute as a cellulose resource for a number of applications. This material is renewable and in great abundance in several regions of the world. It is usually burned off or disposed of for ambient degradation. Chemical analysis results showed that alkali-treated maize tassel fibers had higher percentage cellulose content, while lignin and hemicellulose contents were considerably lower in the untreated fibers. The results obtained from FT-IR analyses showed that the lignin and most of the hemicellulose were removed following NaOH treatment. SEM analysis of untreated and alkali-treated fibers indicated some changes in the morphology after NaOH treatment. The decrease in the content of hemicellulose resulted in void pattern and roughening of the surface, as noticed in the SEM micrographs of the alkali-treated fibers. XRD analyses revealed an increase in the crystallinity of the maize tassel fibers after alkali treatment. The thermal stability of maize tassel fiber improved on alkali treatment. This study advances the possibility of utilizing the by-product maize tassel fibers (waste), which is renewable, inexpensive, and biodegradable, as reinforcement in biocomposites.

## FUNDING

The authors would like to thank the DST/CSIR National Centre for Nanostructured Materials, and the Tshwane University of Technology for their financial support.

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