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The Extraction and Bleaching of Plantain Peduncle waste Fiber through Alkaline Mercerization Process

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Abstract*: The increasing demand for fiber in fields such as engineering, building and construction, textile, medical, automobile etc. has resulted in rise in the production of synthetic fibers from petroleum products which are non-degradable and hazardous to humans and the environment. To minimize this hazard from synthetic fibers, has led to the uncontrolled cutting of forest for wood fibers thus, exposing the environment to infrared rays from the sun. The use of plant waste material such as plantain peduncle for fiber production, is one way of solving this problem. This research looks at the extraction and bleaching of plantain peduncle waste fiber using mercerization techniques and its characterization. The experimental results showed that the unbleached plantain peduncle fiber after alkaline mercerization had rough surfaces with some impurities which can be attributed to the NaOH solution dissolving waxy, gummy materials in the peduncle cells and the removal of lignin and hemicellulose. The bleached peduncle fiber was brighter in colour compared to the unbleached fiber due to its high crystallinity which indicates further removal of impurities, lignin and hemicellulose from the fiber as shown in the SEM images. The FTIR analysis of the unbleached and bleached fibers showed broad peaks at 3295 cm^{-1} and 3308 cm^{-1} respectively indicating the presence of O-H bonds in the fibers. The peaks at 2922 cm^{-1} indicates C–H group in both fibers. The absence of absorbance peak at 1736 cm^{-1} for the bleached fiber showed that lignin and some hemicellulose were removed from the unbleached fiber during bleaching process. The FTIR analysis indicates the functional groups present in the fiber and how bleaching affects the structure and chemical changes in the fiber. The XRD analysis showed peaks at 2θ of 22.14 º corresponding to the crystallographic phase of cellulose ($\boldsymbol{I_{200}}$) and 2θ of 16.18º indicating the amorphous phase ($\boldsymbol{I_{101}}$) for unbleached fiber. Similar peaks at 2θ of 22.33º and 16.14º was found for the bleached fiber. There was an increase in the crystalline index from 34% for unbleached fiber to and 50% for the bleached fiber, which showed that the bleaching process removed lignin, hemicellulose and the amorphous parts indicating an increased in the cellulose content of the bleached fiber. The tensile strength analysis of the fiber using universal tensile testing machine showed that the unbleached fiber had tensile strength of 118 MPa and bleached fiber (124 MPa) respectively. The increase in tensile strength of the bleached fiber is due to the improve crystallinity of the fiber after bleaching.*

Keywords—Alkaline mercerization; Plantain peduncle; Fiber; Bleaching; XRD; SEM; FTIR

1. INTRODUCTION

Global industrialization has increased the demand for fibers in various fields such as engineering, automotive, textile, material reinforcement and medical fields. This has led to the massive production of synthetic fibers from petroleum products with properties such as high strength, light weight, low water absorption, and high stiffness. These synthetic fibers are non-degradable in nature and can be hazardous to human and the environment [1]. This has led to the increased exploitation and destruction of forest in search of wood fibers, thus, exposing the environment to direct infrared rays from the sun which is of global concern. This has made researches to look for alternative source for fiber synthesis such as biological-based fibers from plant and plant waste that will encourage good and profitable use of natural resources. These plant-based fibers will act as a substitute for synthetic fibers produced from petrochemicals and fossils raw materials. One major way of achieving this is by extracting and synthesizing fiber from plantain residues such as plantain peduncle and pseudostem.

The Niger Delta states in Nigeria are known for their huge plantain farms and the false horn type of plantain is the most cultivated in these states because of the ability of the false horn type plantain to grow in a poor soil condition [2]. Plantain is a starchy tropical fruit resembling banana but bigger belonging to the Musa species. Research have shown that unripe plantain fruits contain crude protein, crude fiber, moisture, ash, carbohydrates, calcium, sodium, magnesium, potassium, iron, phosphorus and zinc [3]. After the removal of the plantain fruits from the bunch, the peduncle is discarded as waste to the environment causing pollution. Research works on plantain are mostly focused on its propagation, nutrients and proximate analysis and no work on plantain peduncle fiber has been carried out based on our knowledge. Series of research have been carried out on banana peduncle such as the effect of banana peduncle fiber on hybride composites [4]. Research has also found out that the stem of the banana peduncle contains cellulose lignin and ash [4].

Plantain peduncle is the stem of the plantain plant, which supports the flora that produces the plantain fruit. It can also be a good source of extracting fibers for engineering applications, textiles, food and biomedical uses. Plantain peduncles are readily available waste in Bayelsa state in the

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Niger Delta areas of Nigeria and are littered around markets in the state causing environmental pollution.

This research work looks at the extraction and bleaching of fiber from Plantain peduncle waste and the characterization of the fiber using Scanning electron microscopy (SEM) for fiber morphology, Xray diffractometry (XRD) for the determination of the crystalline phase of cellulose in the fiber and Fourier transform infrared spectrophotometry (FTIR) for identifying the functional groups and bonds in the fiber.

2. MATERIALS AND EQUIPMENT

The materials and equipment used for this experiment are; plantain peduncle, sodium hydroxide (NaOH), hydrogen peroxide, distilled water, magnetic stirrer, weighing balance, 250 ml beakers, measuring cylinders, oven, glass stirrer. XRD, SEM, FTIR and Universal tensile tester.

2.1 Extraction of Fiber from plantain peduncle

Extraction of fiber from plantain peduncle follow similar mercerization technique adopted by Nadirul and co-researchers [5] with modification. Plantain peduncle was washed thoroughly to remove dirt's and sands and cut open to dry at room temperature for an hour. The cut peduncle was placed in a beaker containing 20% NaOH aqueous solution (w/v) and stirred continuously for 24 h at room temperature. The mercerized fibers were then washed with distilled water severally to remove NaOH on the fiber. The fibers were further washed with 1% HCl solution to further neutralize the remaining NaOH solution on the fiber before rinsing severally with distilled water to a neutral pH. The fibers were dried in a vacuum oven at 60°C for 24 hours and further dried at 100°C for 1 hour.

2.2 Bleaching of Fiber synthesized from plantain peduncle

The alkaline bleaching of the fiber was carried out following similar method adopted by Amrita and Anjali [6] with modification. In this process, 5% volume hydrogen peroxide solution (100 ml) with pH 11 was prepared in 4 % sodium hydroxide base solution. 200 grams of synthesized fiber was mixed with the hydrogen peroxide solution and the mixture was heated for 30 minutes at 70ºC. After heating, the fibers were washed with distilled water to neutral pH, before drying at 60ºC in an oven for 24 hours. The bleached and unbleached fibers were characterized using SEM, XRD and FTIR.

3. RESULT AND DISCUSSION

Fig. 1 (a) shows the cut peduncle from plantain bunch and (b) the waste created when the peduncles are not properly disposed causing environmental pollution.

Fig. 1 Freshly cut and waste plantain peduncles

Fig. 2 shows the steps in the mercerization of the plantain peduncle. Fig. 2 (a) shows the plantain peduncle cut and washed with distilled water to remove dirt's; Fig. 2 (b) is the prepared 20% NaOH solution for the alkaline mercerization. Fig. 2 (c) shows the plantain peduncle cut into bits and placed inside the NaOH solution where it is stirred mechanically for 24 hours to loosen the fiber from the peduncle at room temperature.

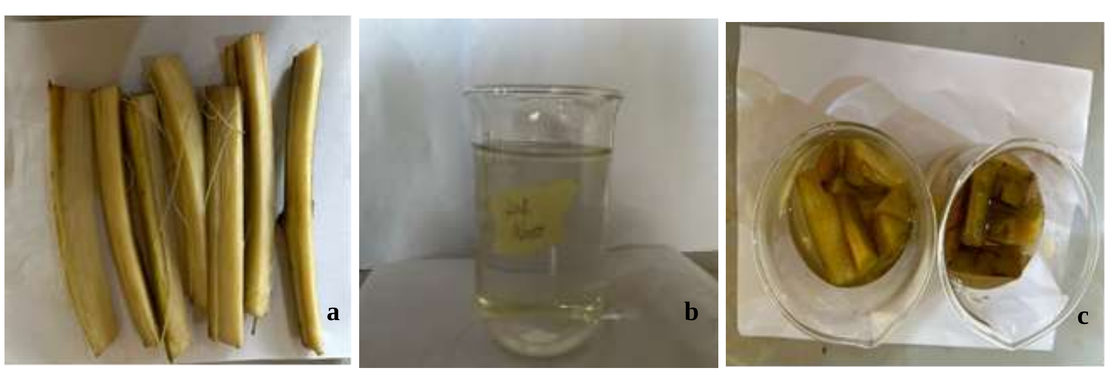

Fig. 2 Start of mercerization process

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Fig. 3 (a) shows the dissociated fiber from the plant cells due to the presence of NaOH after continuous stirring for a period of time. The stirred fiber solution turns dark in colour due to the NaOH dissolving and removing waxy, gummy and lignin from the peduncles. Fig. 3 (b) shows fiber extracted from the peduncle as continuous stirring takes place. Fig. 4 shows the fiber being washed with distilled water severally to remove NaOH after alkaline mercerization process. And from the pictures in Fig. 4, it can be seen that the dark colour of the fiber becomes brighter. This is due to the removal of the dissolved wax, gums, lignin and hemicellulose from the extracted fiber during washing.

Fig. 3 Fibers extracted using 20% Sodium hydroxide solution

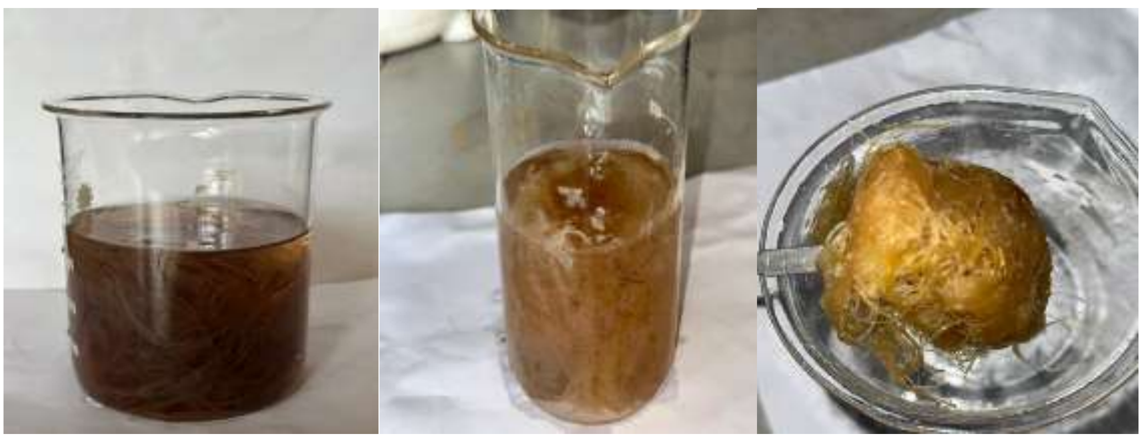

Fig. 4 Extracted fiber washed with distilled water

Fig. 5 shows the process of further washing the fiber with 1% HCl solution to remove remaining alkaline (NaOH) left in the fiber. Fig. 5 (a) shows the prepared 1% HCl solution, Fig. 5 (b) and (c) is the rinsing process of the fiber inside the 1% HCl solution to remove excess NaOH.

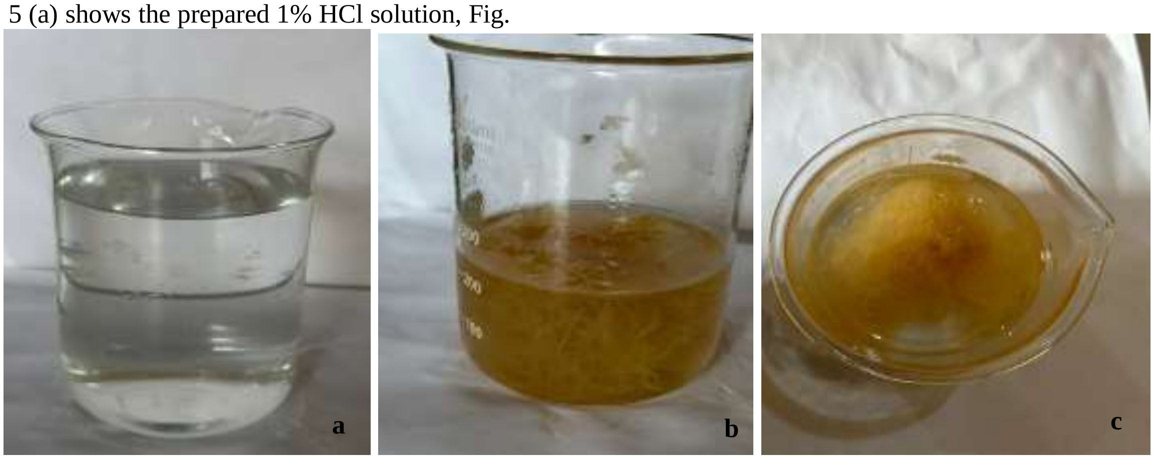

Fig. 5 Extracted fiber washed in 1% HCl solution

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Fig. 6 (a) shows the mercerized fiber being washed with distilled water to remove excess HCl to a neutral pH before drying in an oven at $60^{o}C$ for 24 hours before further drying at $100^{o}C$ for 1 hour as shown in Fig. 6 (b).

Fig. 6 Mercerized Fiber (unbleached) washed (a) in distilled water and dried (b)

Fig. 7 shows the bleaching processes of the extracted plantain peduncle fiber in hydrogen peroxide solution. From the figure it can be seen that the colour of the fiber becomes brighter due to the removal of impurities, lignin and hemicellulose from the fiber cells. Fig. 8 shows the washed fiber with distilled water after bleaching.

Fig. 7 Bleaching of the extracted plantain peduncle fiber in hydrogen peroxide

Fig. 8 Washed bleached fiber

Fig. 9 shows the picture of the unbleached (a) and bleached plantain peduncle fiber (b). From the figure it can be seen vividly the difference in colour of the fibers which is an indication of the removal of impurities, lignin and hemicellulose and other materials from the fiber as shown in Fig. 9 (b) compared to Fig. 9 (a). This result is further corroborated with the XRD diffractogram in Fig. 10 and Fig. 11 for the bleached and unbleached fibers respectively.

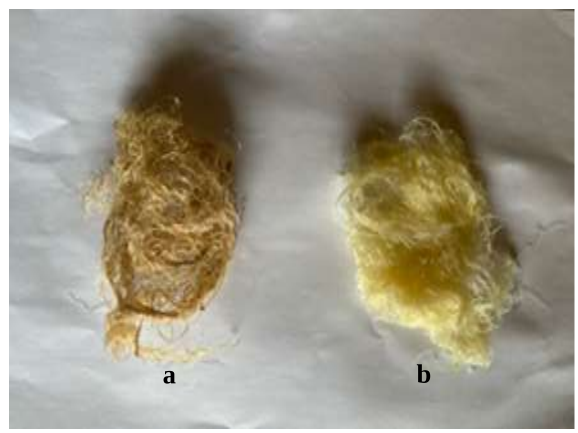

Fig. 9 Dried Unbleached (a) and bleached fiber (b)

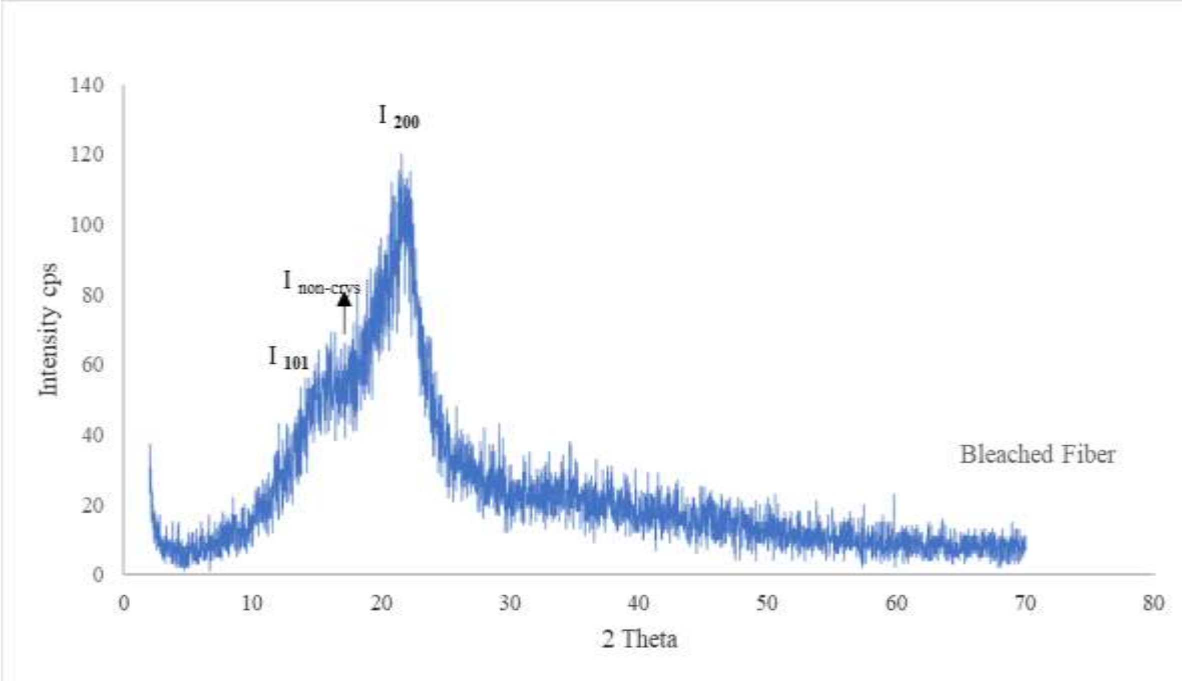

Fig. 10 XRD diffractogram for bleached plantain peduncle fiber

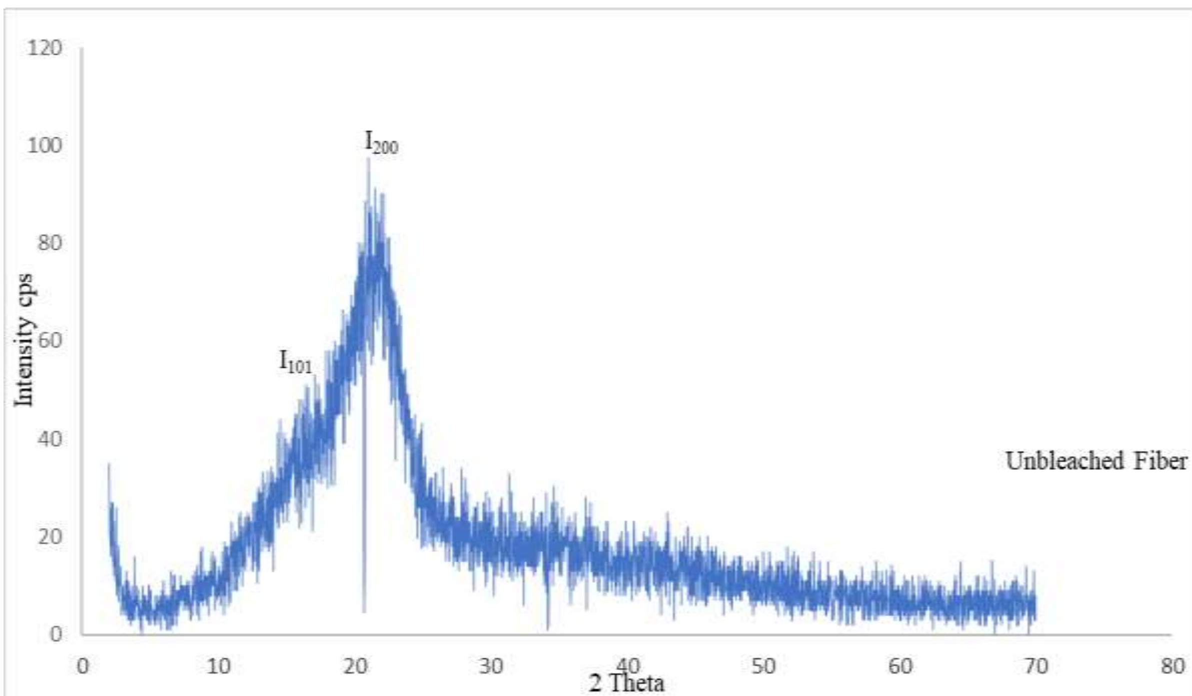

Fig. 11 XRD diffractogram for unbleached plantain peduncle fiber

Fig. 10 and 11 shows the XRD diffractogram of the bleached and unbleached fiber respectively. Fig. 10 contains peaks at 2θ of 22.14 º corresponding to the crystallographic phase of cellulose (I_{200}) and 2θ of 16.18º corresponding to the amorphous phase (I_{101}) resulting from superposition of peaks in cellulose. It also had increased intensity compared to Fig. 11. Similar phases are found in Fig. 11 for the unbleached fiber. The peaks at 2θ of 22.33º in Fig. 11 corresponds to the crystalline phase (I_{200}) in cellulose and peak at 16.14º showed the presence of amorphous phase in cellulose. The non-crystalline part of the fiber is observed clearly for the bleached fiber at 2θ of 18.18º as shown in Fig. 10.

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The crystalline index of cellulose in the unbleached and bleached fibers were calculated by applying the Segal empirical formula [7];

$$C_{I\%} = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \qquad 1$$

where I_{200} is the peak intensity of cellulose I, and I_{am}, is amorphous fraction peak intensity.

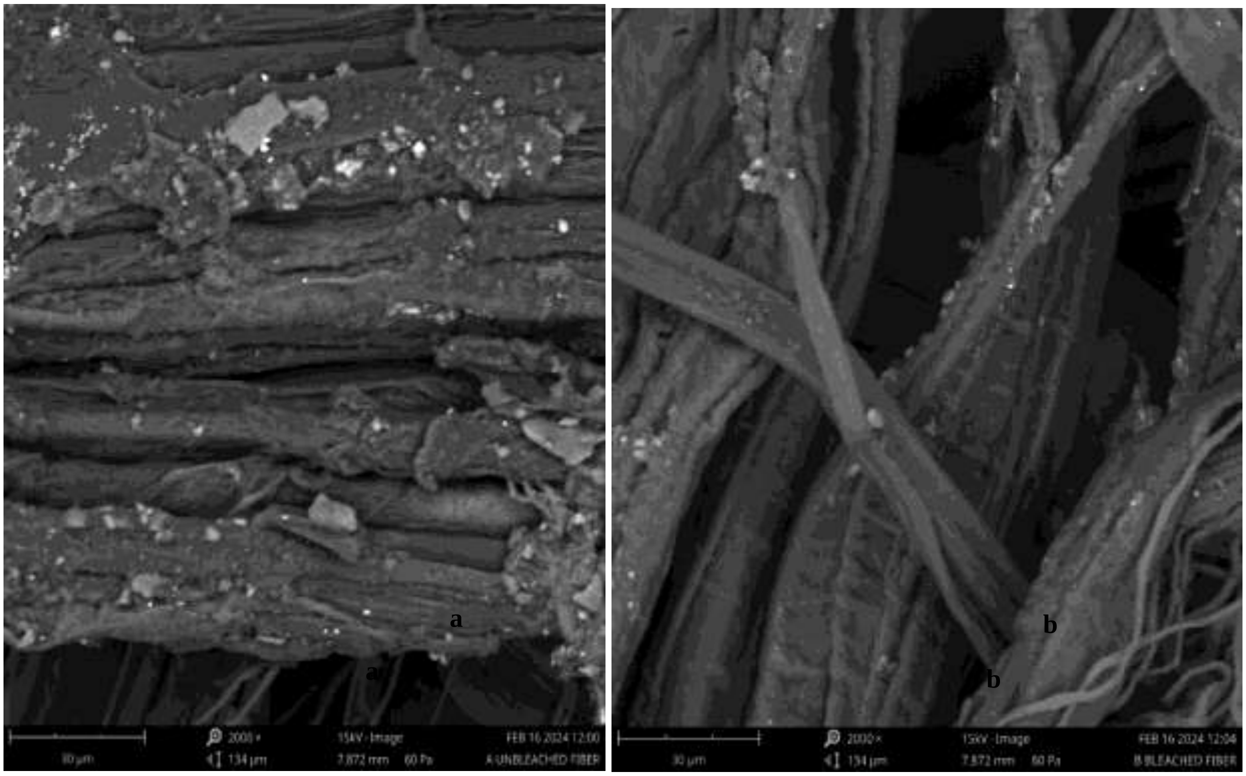

Fig. 12 SEM images of unbleached (a) and bleached fiber (b)

Fig. 12 shows the SEM image for the unbleached fiber (a) and bleached fiber (b). The figure showed changes in the surface morphology of the fibers and a dense fibrillar structure is seen which is due to the alkaline mercerization of the fibers resulting in the dissolution and removal of waxy and gummy materials from the peduncle fiber. Fig. 12 (a) showed that the unbleached fiber after mercerization had dense rough surface with some surface impurities while the bleached fiber (Fig. 12 b) had smooth and cleaner surfaces which is attributed to the removal of impurities on the fiber surface, and further removal of waxy, lignin and hemicellulose. Plantain and banana fibers are known to contain cellulose (71.08%), hemicellulose (12.61%) and 7.67% of lignin [8]. Research have shown that bleaching fiber removes lignin and hemicellulose from fibers [7].

Based on the XRD raw data, corresponding d-spacing for the phase I_{200} and $I_{101}$and their respective intensities, the unbleached fiber had crystalline index 34% and 50% for the bleached fiber. The increased value of crystalline index in the bleached fiber showed that during bleaching of the fiber, some lignin, hemicellulose and amorphous parts were further removed from the fiber and research have shown that this removal leads to improved tensile strength for fiber [7]. The XRD result is corroborated with the SEM image for bleached and unbleached fiber shown in Fig. 12.

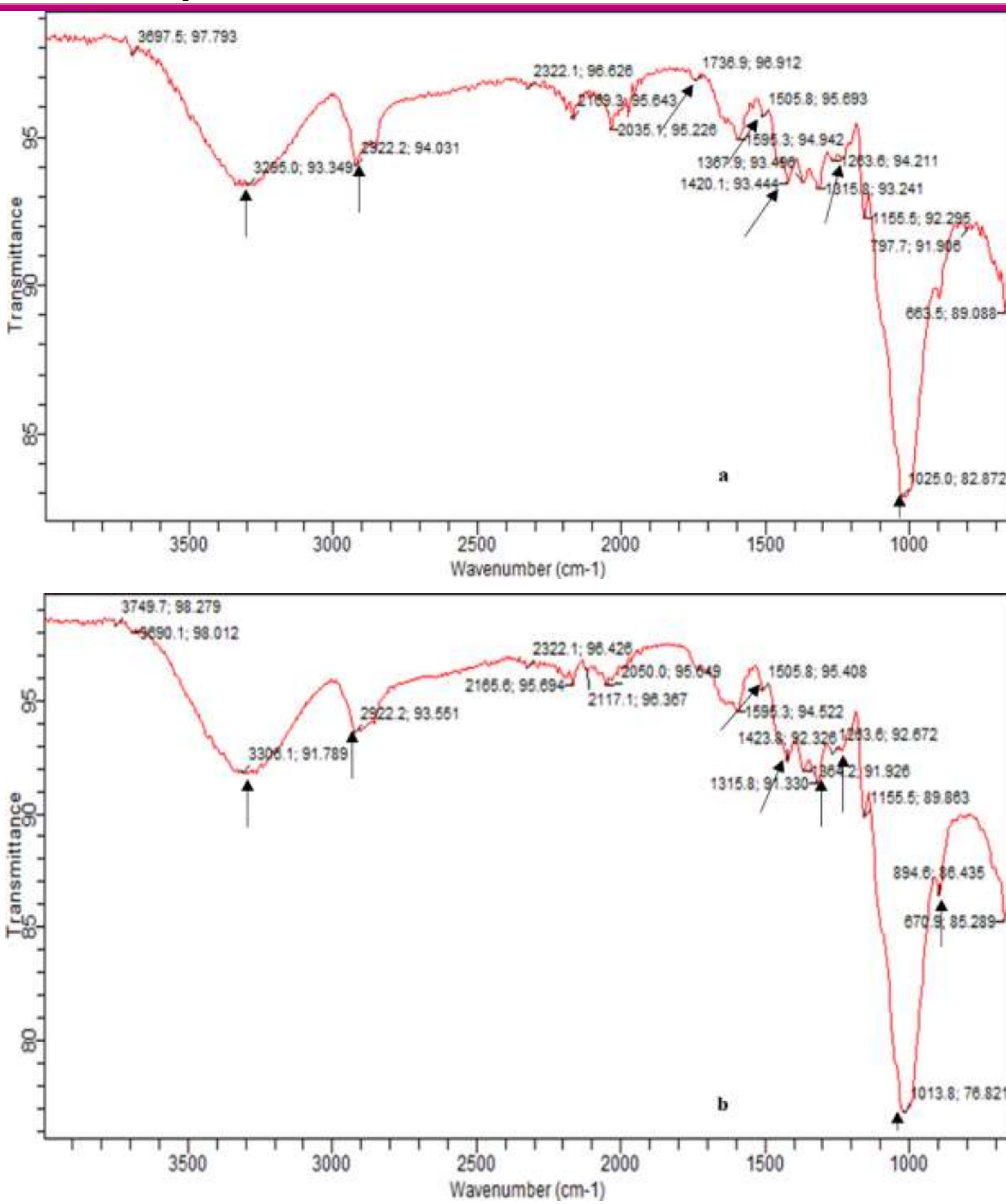

Fig. 13 FTIR analysis of unbleached (a) and bleached fiber (b)

Fig. 13 shows the FTIR spectra for unbleached (a) and bleached (b) synthesized plantain peduncle fiber. The FTIR spectra showed the functional groups present in the unbleached and bleached plantain peduncle fibers and how bleaching affects the structure and chemical changes in the fiber. In Fig. 13 (a), the FTIR spectra showed broad peak at absorbance peak of 3295 cm^{-1} for the unbleached fiber and a shift in absorbance peak to 3308 cm^{-1} for the bleached fiber as shown in Fig. 13 (b) indicating hydroxyl functional group (O-H) presence in both fibers. The peaks at 2922 cm^{-1} in Fig. 13 (a) and (b) respectively showed the stretching of C–H group in both fibers. The absorbance peak at 1736 cm^{-1} in Fig. 13 (a) for unbleached fiber is absent in Fig. 13 (b) for bleached fiber. And this peak shows the presence of C=O stretching in the unbleached fiber indicating the presence of acetyl group in hemicellulose and the presence of esters and carboxylic groups in the fiber [7].

The absence of this peak in the FTIR spectra for the bleached fiber in Fig. 13 (b) showed that lignin and some hemicellulose were removed from the unbleached fiber during bleaching process. The peak at 1505, 1263 cm^{-1} in Fig. 13 (a) and (b) respectively and peak 1420 cm^{-1} for unbleached fiber in Fig. 13 (a) shifted to 1423 cm^{-1} in Fig. 13 (b) all corresponds to skeletal vibration of aromatics associated with C-O bonds in lignin [9]. The shift in peak 1420 cm^{-1} for unbleached fiber to 1423 cm^{-1} with decreased peaks for bleached fiber indicates that some lignin was removed during bleaching process. The absorbance peak at 1025 cm^{-1} for unbleached fiber in Fig. 13 (a) and 1013 cm^{-1} in (b) respectively results from the stretching of O–H and C–O groups and the small peaks at 894 cm^{-1} in Fig. 13 (a) and (b) respectively indicates the presence of sugar units found in cellulose and hemicelluloses in the fibers [9-10].

The tensile strength of the fiber showed that the unbleached fiber had tensile strength of 118 MPa and bleached fiber (124 MPa) respectively. The increase in tensile strength of the bleached fiber is due to the improve crystallinity of the fiber after bleaching. The tensile strength of the fibers was determined using universal tensile testing machine based on ASTM D638 guidelines.

4. CONCLUSION

Plantain peduncle has been shown to be a good source of fiber which can be applied in engineering and other fields. The unbleached fiber after alkaline mercerization had rough surfaces with some impurities which can be attributed to the NaOH solution dissolving waxy, gummy materials in the peduncle cells and the removal of lignin and hemicellulose. Bleaching the peduncle fiber made the fiber brighter with improve crystallinity which is as a result of further removal of impurities, lignin and hemicellulose from the fiber as shown in the SEM, XRD and FTIR images. This in turn improved the tensile strength of the fibers from 118 MPa for unbleached to 124 MPa for bleached fiber. The increase in tensile strength of the bleached fiber is due to the improve crystallinity of the fiber after bleaching.

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