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FTIR EVALUATION OF ALKALINE HYDROGEN PEROXIDE BLEACHED CELLULOSE FROM JACKFRUIT PEEL AND LEAF

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Abstract

The need for plant waste valorization has become a subject area that most researchers have delved into. One of such aspects is cellulose extraction from jackfruit tree waste which is seen as a commercially underutilized plant. In this research cellulose was extracted from Jackfruit Peel (JFP) and Jackfruit Leaf (JFL) using hydrogen peroxide, nitric acid, acetic acid and alkaline hydrogen peroxide (AHP) bleaching solutions respectively to purify the extracted cellulose. Comprehensive characterization of the cellulose was performed using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). The peel yielded 17.4% of cellulose while the leaf gave 33.21% cellulose yield after AHP bleaching which achieved higher purification efficiency than the other bleaching solutions. The level of purity of cellulose after AHP bleaching is comparable to that of the commercial cellulose based on the FTIR results. However, with EDS carbon weight concentration of up to 84%, JFP cellulose proved to be purer than JFL cellulose. FTIR spectroscopy also confirmed their cellulose content in relation to commercial cellulose while morphological characterization with SEM revealed separated single cellulose fibres of JFL and the compacted cellulose fibre sheets of JFP. JFP cellulose also has a higher crystalline index (41.2%) than JFL cellulose based on the XRD result. Both plant parts can be sustainable sources of cellulose for industrial use.

Keywords: Jackfruit peel, jackfruit leaf, cellulose, Alkaline hydrogen peroxide, FTIR, waste valorization

1. INTRODUCTION

Cellulose, a biopolymer widely used in various industries, has garnered considerable attention due to its eco-friendly and renewable nature. It is a linear polysaccharide consisting of β-D-glucose units linked together by β-1,4-glycosidic bonds, forming a chain of high molecular weight [1]. As a significant component of plant cell walls, it is one of the most abundant biopolymers on Earth and due to its renewable nature and biodegradability has gained significant attention in various industries such as paper and pulp, textiles, food packaging, pharmaceuticals, and biocomposites [2]. Cellulose is majorly obtained industrially from wood pulp and cotton (cotton – 90% and wood- 40–50%). But its yield and purity is significantly affected by extraction method employed. Hence, researchers are exploring different methods of extraction [3][4][5]. Several studies have already shown that cellulose can be obtained from various plant materials and waste plant materials using diverse extraction methods [6][7][8][1]. Extraction of cellulose from waste plant materials is one way to encourage and support circular economy.

The jackfruit (Artocarpus heterophyllus), also known as jack tree is a tropical fruit widely cultivated around the tropical regions of the world. The food and pharmacological properties of the tree have been explored with significant results reported in literature [9][10]. Yet it is largely seen as a commercially underutilized plant due to the significant amount of agricultural waste it generates annually majorly due to poor knowledge and inadequate facilities to properly process most parts of the plant in some areas where

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it is grown [11] [12]. In major jackfruit producing countries like India, Indonesia, Nepal waste from Jackfruit is up to 65 – 75% while jackfruit peel which is the outer covering of the fruit contributes about 50% of the waste from the fruit [13]. In other to valorize its high waste, attention of researchers globally, has shifted toward exploring the cellulose content of the plant. There is however limited information on cellulose extractions from Jackfruit peel and leaf [10][12]. Similarly, the advantage of jackfruit cellulose over other plant extracted cellulose cannot be fully evaluated due to scarcity of research in that area. We however know that cellulose nano-crystals (CNC) and cellulose nanofibers (CNF) have been extracted from Jackfruit [12].

Nigeria in particular, most research on jackfruit is in the area of phytochemical analysis and health benefits of the plant [14][15]. Cultivation of jackfruit in Nigeria is not encouraged and its consumption is only regarded as food for the poor [16][15]. Ezim, O. E. et al, 2020, pointed out that it is one of the underutilized plant species with over 90% of its seeds wasted annually [17]. There are no published documented data on annual Jackfruit waste generation or disposal in Nigeria as well as cellulose yield percentage for JFP and JFL from Nigerian grown jackfruit tree, hence the novelty of our research. This research is also an attempt to increase the information on cellulose extracted from the fruit peel and leaf of the jackfruit tree globally. The leaves and fruit peels, which are usually discarded as waste, contain a substantial amount of cellulose as shown in some reports (Table 1) which is based on research from outside the shores of Nigeria. Researchers have reported several works on extraction of cellulose from jackfruit peel with very little work on cellulose extraction from jackfruit leaf. Most of the reported research concentrated on the use of sodium hypochlorite ($NaClO$) or sodium chlorite ($NaClO_2$) which is not environmentally friendly for bleaching [18][19][20]. This research paper however focuses on the extraction of cellulose from jackfruit peels and leaves and the use of alkali hydrogen peroxide (AHP) solution to bleach the extracted cellulose. Alkaline and hydrogen peroxide solution for bleaching is one of the cleanest and safest bleaching solutions. It is odourless and environmentally friendly [4]. We also compared the purity and yield of the extracted cellulose from the two different plant parts while using commercial cellulose as a standard by conducting FTIR analysis. Some other investigations like SEM, XRD and EDS were equally conducted to explore the surface morphology, crystalline nature and elemental content of the extracted cellulose from JFP and JFL.

Table 1. composition of jackfruit leaf and peel

S/N	Jackfruit component	Peel (%)	Leaf (%)
1	cellulose	27.75 [21] [22] [12]	21.45 & 13.72 [23]
2	pectin	7.25 [21] 9-15% [12][20]	
3	Protein	6.27 [21] 9% [12]	1.19 [15]
4	starch	4 [21]	

EXPERIMENTAL MATERIALS

Jackfruit leaves and mature fruits were collected from a local jackfruit tree orchard located at the Federal University of Technology, Owerri (FUTO). The plant was identified at the School of Agriculture and Agricultural Science of the same University. Hydrogen peroxide (H_2O_2), Sodium hydroxide ($NaOH$), Ethanol (CH_3COOH), Acetic acid, and distilled water were purchased from HI-TECH Diagnostics Ltd Owerri, Nigeria. Other chemicals used in the study include; Toluene, Sodium borate, Nitric acid, Acetic acid, Buffer solution from Kenten Nigeria Ltd, Owerri. All chemicals were of analytical grade and needed no further purification.

METHOD

1. JACKFRUIT TREE SAMPLE COLLECTION AND PREPARATION

The leaves were washed, air-dried, ground and stored in a plastic container. While the fruit bark was peeled with a kitchen knife, washed and dried till constant weight before grinding. Both samples were kept in an airtight container.

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2. CELLULOSE EXTRACTION

We used the procedure I method reported by Moran, J. I. *et al* (2008) [24] for cellulose extraction with slight modifications. 40g of dried and ground jackfruit leaf (JFL) and Jackfruit peel (JFP) was placed respectively in a 500ml mixture of toluene/ethanol (2:1 v/v) and boiled for 6hr in a soxhlet before filtering and washing the plant material with ethanol for 30min. The plant material was dried and treated with 200ml of 0.1 M sodium hydroxide (NaOH) and 200ml of 50% of ethanol at 45 ^oC for 3 h respectively under continuous stirring. After which it was treated with 200ml of 3% H_2O_2 at pH 11.5 (buffer solution) at 45 ^OC for 3 h still under continuous stirring. This was followed by treatment with 100ml of 10% w/v NaOH, 1% w/v $Na_2B_4O_7.10\ H_2O$ at 28^{o}C for 15 h, respectively under continuous agitation. Lastly, it was treated with 100ml solution of 70% HNO_3 and 80% Acetic acid (1/10 v/v) at 120 ^OC for 15 min before washing with 95% ethanol, washing with water and washing again with 95% ethanol before drying at 60 ^OC in an oven until constant weight.

3. BLEACHING WITH AHP

Further bleaching of the dried extracted cellulose was done using alkaline hydrogen peroxide (AHP) method reported by Qasim, U. *et al*, 2020[25] with modifications. A 60ml solution at the mix ratio of 1:2 (v/v) of 1% sodium hydroxide and 20% Hydrogen peroxide was prepared and used to bleach 0.32g (JFL) and 1.65g (JFP) respectively of the extracted cellulose in a reflux condenser unit at 50^{O}C for 45min. After bleaching the residue was washed until neutral with distilled water and oven dried 60 ^OC until constant weight. Extracted and bleached cellulose were kept in sample bottles pending further analysis.

The cellulose percentage yield was calculated with equation 1

$$Cellulose\% = \frac{Y_2}{Y_1} \times 100 \qquad (1)$$

Where Y_2 is the weight of extracted cellulose and Y_1 is the weight of the original sample.

4. CHARACTERIZATION OF EXTRACTED CELLULOSE

The cellulose samples were characterized using various analytical techniques:

i. FTIR Spectroscopy using Pelkin Elmer 3000 MX spectrometer. All spectra were recorded from 4000 to 400 cm^{-1} with 32 Scans per spectrum with a resolution of 4 cm^{-1}. This will be used to analyse the two extracted cellulose in comparison with commercial cellulose.

ii. Scanning Electrode Microscope (SEM) and Energy dispersive X-ray spectroscopy (EDS) of samples were studied using the Thermo Scientific Axia ChemiSEM system with accelerating voltage of 15kv integrated fully with EDS capabilities.

iii. XRD analysis was carried out with Rigaku Miniflex X-ray diffractometer developed by the Rigaku International Corporation Tokyo, Japan set to produce diffractions at scanning rate of 2^o/min within the range of 2^o to 80^o (2θ) at room temperature with a Cu Kα radiation set at 40kV and 20mA and wavelength λ = 0.1542nm. The crystalline index (Cl) for the two extracted cellulose was calculated using equation 2[18]

$$CI(\%) = \frac{A_{crystalline}}{A_{Amorphous} + A_{crystalline}} \times 100 \qquad (2)$$

Where $A_{crystalline}$ is the crystalline area and $A_{Amorphous}$ is the amorphous area of the diffractogram [18].

RESULTS AND DISCUSSION

Jackfruit leaves and peels, often considered as agricultural waste, have shown great promise as a source of cellulose. The extraction process produced cellulose with properties comparable to traditional sources.

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1.CELLULOSE YIELD

The cellulose extraction process yielded 41% of cellulose from the jackfruit leaves before AHP bleaching. After AHP bleaching 81% of the extracted cellulose was retained this is 33.21g of cellulose in 100g of jackfruit leaf. This is higher than the result achieved by Dewi Tristantini, et al 2018 [23] and their reported cellulose content of jackfruit leaf. In the case of the jackfruit peel, 58% of cellulose from jackfruit peel was extracted however after AHP bleaching only 30% of the extracted cellulose was retained that is 17.4g of cellulose in 100g of jackfruit peel which is 66.7% considering that the cellulose content of jackfruit peel is about 27g. This yield % from the peel is slightly lower than the report by Allwyn, S. A, *et al,* (2018) [22] and AbdulRahman, W. *et al*., (2019) [26] but higher than the results achieved by Padhi, S. *et al.,* (2023) [21] and Brahma, R et. al, 2022[20]. Such differences in yield could be attributed to the difference in extraction methods and purity level of the extracted cellulose. It can also be due to environmental factors on the plant arising from the different geographical regions of the plants [20]. The fact that the JFL yielded more cellulose than the JFP is an indication of higher cellulose composition JFL.

2. PHYSICAL APPEARANCE

It is evident that the high value of cellulose yield by AbdulRahman, W. *et al*., (2019) [26]is due to the poor bleaching which left their extracted cellulose having same colour as our cellulose before AHP bleaching (fig. 1a). The brown coloration of the cellulose before or after any bleaching process have been confirmed to be an indication of the presence of noncellulosic components especially lignin and hemicelluloses [25] [23]. This informed our decision to only investigate the purity level of the cellulose after AHP bleaching using FTIR. Several other reports with closely related physical appearance to ours used either sodium chlorite or sodium hypochlorite for their bleaching [18] [20]. Sodium chlorite is not eco-friendly due to the negative impact of chlorine to the environment [2] [27] [25] hence our choice of AHP which is eco-friendly.

Fig.1 (a). Jackfruit peel cellulose after extraction. (b). Jackfruit peel cellulose after AHP bleaching

(c). Jackfruit leaf cellulose after extraction. (d). Jackfruit leaf Cellulose AHP bleaching

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The colour difference between jackfruit peel in fig 1 a and b and jackfruit leaf in fig 1 c and d is an indication that the 3%H_2O_2 used for the initial bleaching and further purification with nitric and acetic acids though environmentally friendly is not efficient to properly purify the extracted cellulose. Hence the use of modified AHP method with a reflux condenser improved the extracted cellulose both in appearance and purity as shown in fig 1 b and d.

3. FTIR ANALYSIS OF JFL AND JFP

FTIR was used for confirmatory test and results of the analysis of the two extracted cellulose shown in fig 2 and 3 revealed spectrum consistent with cellulose, indicating successful cellulose extraction from both plant samples. Well detailed FTIR results were presented in other to clearly point out the differences between the two extracted cellulose when compared with each other and with commercial cellulose (CC) which served as standard spectrum presented in fig.4. The broad peaks at the wave lengths of 3295cm−1 (fig 2 & 4) and 3287 cm−1 (fig 3) indicate the –OH group. The variation in the intensities of these broad peaks signifies the OH content level [28], which is more in the commercial cellulose and least in the jackfruit leaf. Extraction method and synthesis plant material could be responsible for this variation in addition to slight presence of some non-cellulosic components found in the extracted cellulose. The peaks at 2892, 2918 and 2896 cm-1 in JFP, JFL and CC respectively (between 2500 – 3200cm-1), are associated with C – H stretching vibrations. Carbon dioxide (CO_2) not fully removed from the background air during the analysis were identified within 2000 – 2500 cm-1[29]. While 1640 (Fig 2&4) and 1654 cm-1(fig.3) is the O – H bending of absorbed water mainly associated with cellulose together with the band at 898 cm-1attributed to the asymmetric out of plane ring stretching in cellulose [29] [25] [4]. The vibrations at 1423cm−1 (fig. 2 &3) and 1427 cm−1 (fig 4) is CH_2 scissoring bending in cellulose [18] and those at 1367cm−1 to 1364cm−1 is attributed to C-H bending in cellulose. According to Anuj Kumar, et al 2014 [30], the peaks at 1319cm−1 to 1315cm−1 are caused by CH_2 rocking vibration. Shahin Hossain, et al. 2022[19], however attributed such peaks to O-H bending. Some authors also identified these peaks including 1833 and 1897 cm-1 (fig 3) as lignocelluloses overlap bands especially for cellulose and hemicelluloses corresponding to CH_3 symmetrical angular vibrations [29]. It is observed that the cellulose extracted from the leaf has a weak peak at 1722cm-1 (fig. 3) which is due to vibrations of acetyl and uronic ester groups in hemicelluloses [28] [7] [4]. It can also be argued that the use of acetic acid in the extraction process may have contributed to acetylation of the cellulose however this will not be the case as the same peak is not found in the JFP cellulose extract in fig 2. Peaks within 1500 – 1600 cm-1 have been identified as the range for characteristics lignin bands of aromatic skeletal vibrations [24][31]. Bands 1595 and 1267 cm-1in JFP are aromatic C=C skeletal stretch vibrations and out of plane C-O (aryl group) stretching in lignin respectively [30]. Such non-cellulosic components can be entirely removed through further bleaching cycles or change in bleaching solution concentration. The effect of bleaching cycles and different concentrations of bleaching solution on cellulose purification has already been reported [32] [20]. The band at 1159.2cm−1 in both JFP and JFL (fig 2 and 3) and 1162 cm-1 in CC (fig 4) are due to C-C ring stretching band. Whereas, 1103 and 1107cm-1 in JFL and CC respectively indicates the C-O-C stretching, the broad peaks at 1028.3cm−1 to 1021cm−1 represent the C – O-H bending vibration [29][33][30]. The amorphous region of cellulose is found within the bands 893-897cm-1 while the crystalline phase is within 1420-1430cm-1 [22] [30][34]. They peaks (893-897cm-1 and 1105cm-1) have been equally associated with the β-(1,4)-glycosidic linkages between the glucose units in cellulose (Iβ cellulose structure) and Iα cellulose while the intensities (of 1420 -1430cm-1) have been linked to cellulose crystallinity level [20][19]. We observe that the band ranges present in the two jackfruit extracts are similar to the bands in CC (Table 2), confirming that the extracts from JPF and JFL are cellulose. With this confirmation made, attention was shifted to analyzing only the JFP and JFL cellulose via SEM, XRD and EDS to understand the morphology, crystalline nature and elemental content.

Table 2. Major Cellulose functional Groups of CC compared to JPF and JFL Cellulose

Functional groups	CC (cm^{-1})	JPF (cm^{-1})	JFL (cm^{-1})
– OH	3295	3295	3287
C – H	2896	2892	2918
O – H	1640	1640	1654
CH_2	1427	1423	1423
C – H	1364	1367	1364
C – OH	1028	1021	1025
O – H	898	898	898

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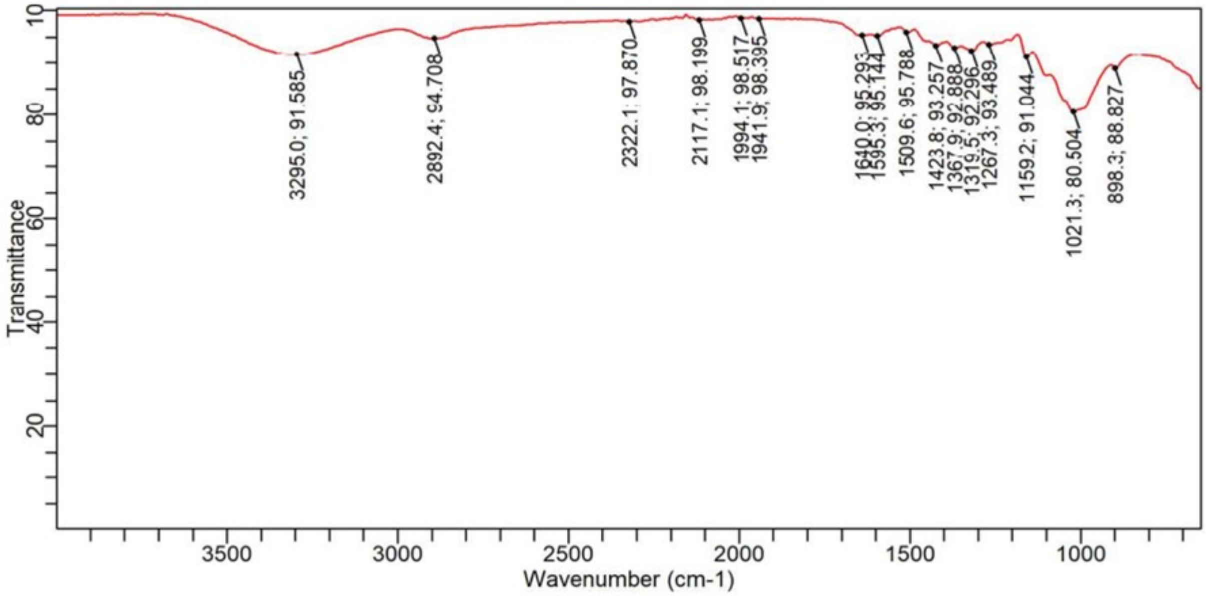

Fig. 2. FTIR of Jackfruit Peel cellulose

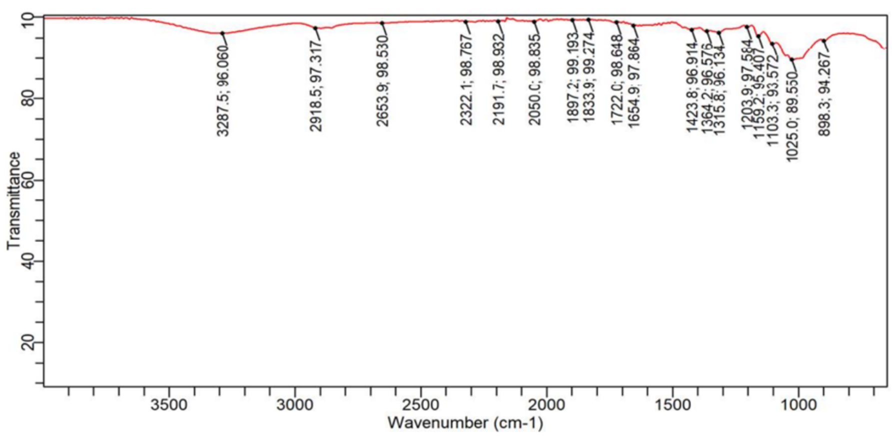

Fig. 3. FTIR of Jackfruit Leaf Cellulose

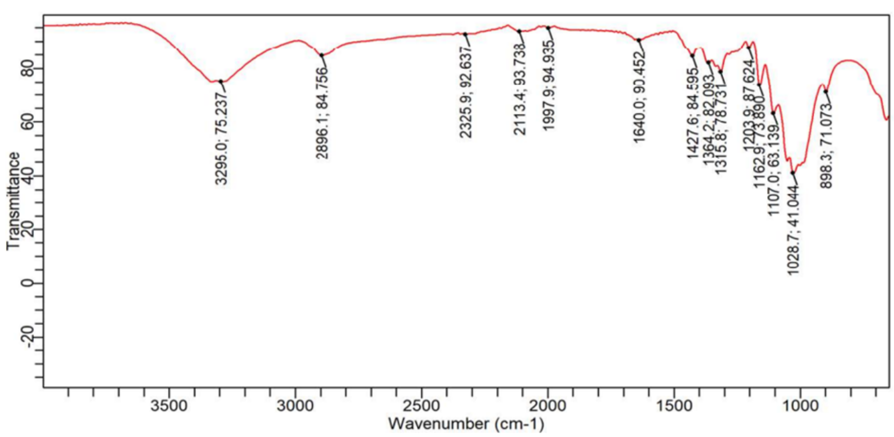

Fig. 4. FTIR of Commercial Cellulose

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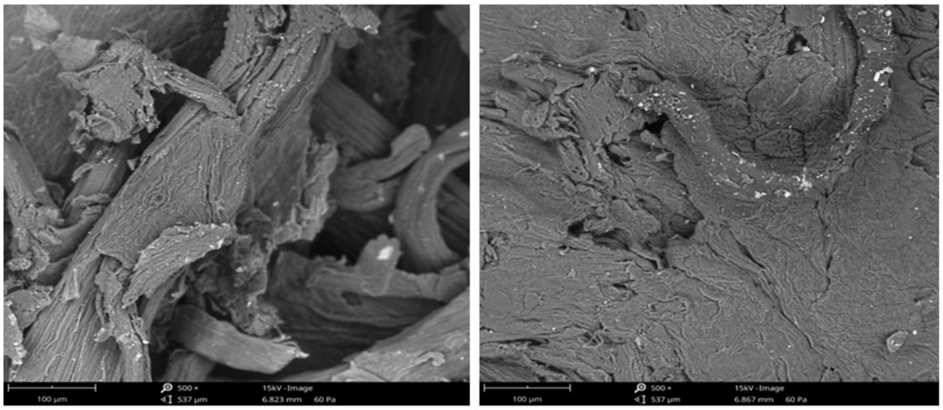
4. SEM ANALYSIS OF JFL AND JFP

Scanning electron microscopy (SEM) was performed to observe the surface morphological structure of the extracted cellulose. JFL cellulose SEM image displayed separate fibrous structures (fig 5a) while that of JFP are compacted fibres forming a sheet-like structure (Fig 5 b).



a b

Fig.5. SEM images (500X magnification) of (a) Jackfruit leaf cellulose and (b) Jackfruit peel cellulose

Our result on the JFP morphology is related to Trilokesh, C. et al 2019[18], report that JFP contains curled self assembled and soft-flat shaped cellulose with rough pits. The SEM results have also shown that extracted cellulose morphology depends on the source and not necessarily the extraction procedure. JFL cellulose can be used as fiber reinforcement for composite polymer products. While JFP cellulose can be hydrolysed and used for ethanol production. Cellulose from jackfruit has also been used for the production of bioplastics [12]. The water and oil holding capacities of jackfruit peel cellulose have been shown to give jackfruit an advantage over some other plant extracted cellulose in serving as bio-absorbent for pollution control [35]. It is however necessary to carry out some required test depending on other desired areas of application.

5. EDS ANALYSIS FOR JFL AND JFP

The presence of sodium, chlorine and nitrogen in both JFP and JFL as shown in fig. 6 (a and b) and fig 7 & 8 could be attributed to residues of the chemical treatment on the materials. However, there presence could simply be leftover traces of actual jackfruit peel and leaf micronutrients including calcium, phosphorous, magnesium and potassium [10]. We observed that while the weight concentration difference of magnesium and phosphorous agrees with earlier report [10] there was a slight difference between that of calcium and sodium. Similarly, with 84.93% carbon weight concentration our cellulose from JFP is purer than the cellulose [44%] extracted by Brahma et al [35]. In addition, JFP cellulose has slightly higher purity than JFL cellulose based on carbon weight concentration percent in fig 6 (a and b).

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	85.62	81.37
7	N	Nitrogen	12.12	13.44
11	Na	Sodium	0.58	1.05
14	Si	Silicon	0.40	0.90
13	Al	Aluminum	0.37	0.78
15	P	Phosphorus	0.17	0.41
12	Mg	Magnesium	0.20	0.39
16	S	Sulfur	0.15	0.38
20	Ca	Calcium	0.11	0.36
17	Cl	Chlorine	0.12	0.35
19	K	Potassium	0.06	0.20
26	Fe	Iron	0.00	0.00

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	88.48	84.93
7	N	Nitrogen	9.76	10.92
11	Na	Sodium	0.39	0.72
13	Al	Aluminum	0.30	0.65
20	Ca	Calcium	0.20	0.64
14	Si	Silicon	0.28	0.63
12	Mg	Magnesium	0.20	0.39
17	Cl	Chlorine	0.10	0.28
15	P	Phosphorus	0.11	0.27
19	K	Potassium	0.06	0.20
22	Ti	Titanium	0.05	0.20
16	S	Sulfur	0.07	0.18
26	Fe	Iron	0.00	0.00

Fig.6 Elemental analysis table (a) JFL (b) JFP

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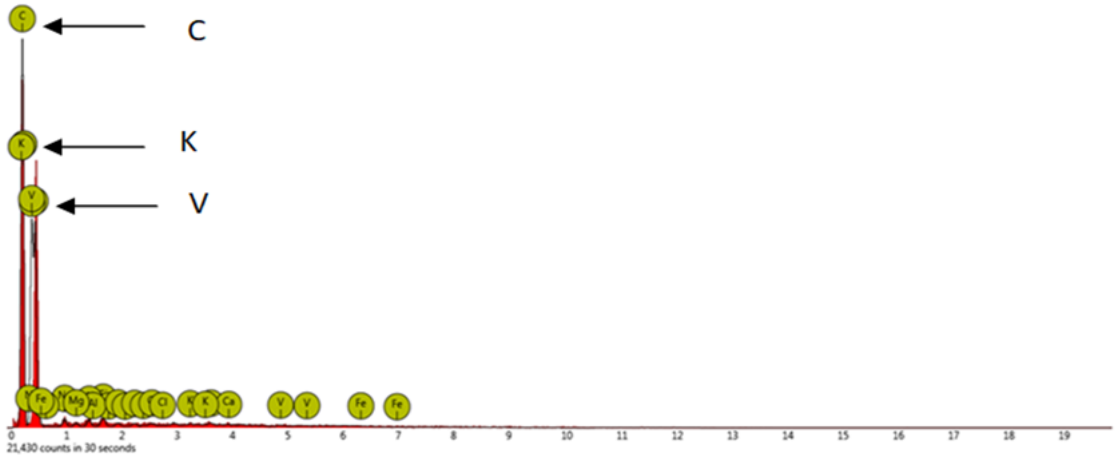

Fig. 7. EDS analysis of JFL C = Carbon, K = Nitrogen and V = Sodium

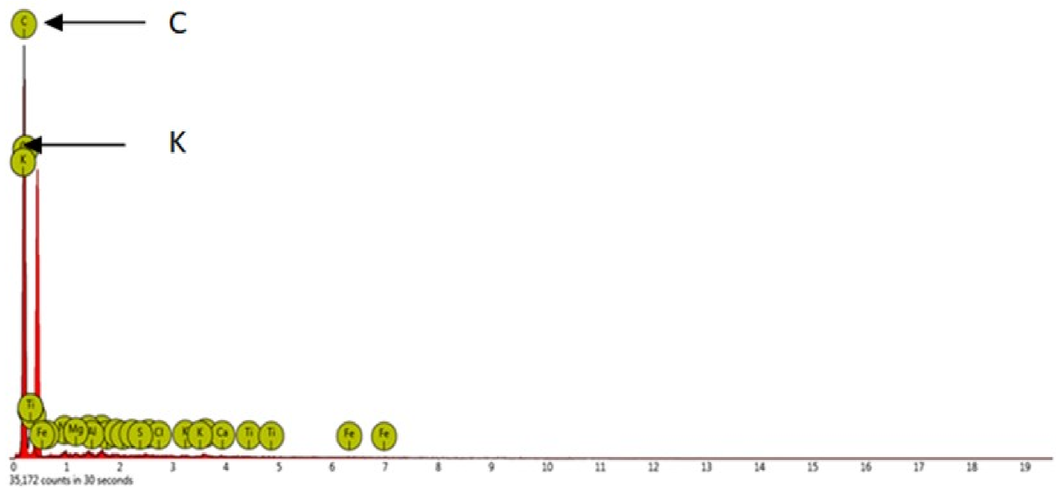

Fig.8. EDS analysis of JFP, where C = Carbon, and K = Nitrogen

6. XRD ANALYSIS

In fig.7 a and b the XRD patterns showed high presence of peaks at 2θ angle around 18.3o, 22°, and 34. 7o for both JFP and JFL which correspond to the 110, 200 and 004 planes of crystals [30][18][36]. The crystalline index (CI) was around 39.3% for JFL cellulose and 41.2% for JFP. These values equally confirm the earlier FTIR results in this work. The difference in cellulose CI has been attributed to difference in cellulose source and isolation method [37]. The crystalline index of JFP is higher compared to the jackfruit peel cellulose crystalline index (30%) reported by Brahma, R and Ray, S. [35]. This difference is attributed to our use of AHP for cellulose purification (bleaching). It has been identified that pH plays an important role in cellulose purification hence hydrogen peroxide under alkaline condition (high pH) produced a reactive oxygen which gave the bleaching effect [38]. Apart from the fact earlier stated that AHP is environmentally friendly it is also more cost effective than some other bleaching reagents [38]. This however cannot be said concerning the cellulose extraction method here used considering the time, energy and reagents required vis-à-vis another method we used for cellulose extraction from other waste plant biomass [39] and those used by some other researchers for extraction from jackfruit [18][35] According to Trilokesh, C. et al [18], the mechanical (especially reinforcement capabilities) and thermal properties of cellulose depend on its crystallinity. Hence fibrous cellulose extracts can function adequately as reinforcing materials for composites.

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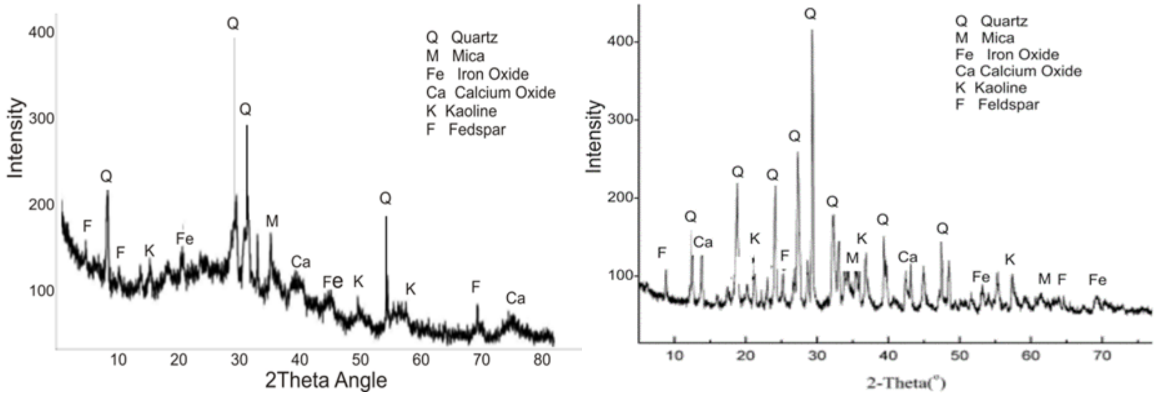

Fig 9. XRD images of (a) jackfruit leaf cellulose and (b) Jackfruit Peel cellulose

COMPARISON

The differences noticed from the cellulose extracted from JFL and JFP using same method of material preparation and extraction method are as follows:

1. Cellulose from JFL appears as separate single fibres while that of JFP are compacted fibers forming separate tiny sheets.
2. JFL yielded more cellulose after AHP bleaching than JFP
3. The JFL cellulose had slight Hemicelluloses presence while JFP had slight lignin content
4. Bleaching with AHP method had a greater observable visual effect on JFP cellulose than on JFL cellulose.
5. The initial bleaching and purification method is not suitable for JFP cellulose.
6. Extracted cellulose from JFP is more crystalline than cellulose from JFL
7. Purity of JFP extracted cellulose is more than that of JFL

CONCLUSION

The extraction of cellulose from jackfruit leaves and peels represents a sustainable and eco-friendly approach to cellulose production. This research paper explored the potential of extracting cellulose from jackfruit leaves and peels, an agricultural byproduct. FTIR result confirmed the JFP and JFL extracts to be cellulose and we have shown that AHP bleaching solution which is environmentally friendly and cost effective can be used to achieve a high level of purity for extracted cellulose from jackfruit peel and leaf. Our research also revealed that jackfruit leaves yielded more cellulose, 33.21% after AHP bleaching than the peel (17.4%) but the JFP cellulose with 84.93% carbon weight concentration is purer. Also the cellulose extracted from the peel was more crystalline (CI =41.2%) than that from the leaves (CI = 39.3%). Both plant parts are a promising source of cellulose, with properties comparable to commercial sources. With the high research interest in extraction of cellulose from jackfruit peel we recommend the use of less energy consuming and green extraction methods. We also recommend the use of green bleaching methods for the purification of extracted cellulose as established by our research findings on the use of AHP bleaching solution. There's a wide information gap on jackfruit cultivation and waste generation and valorization in Africa and Nigeria in particular hence the urgent need for more research. Focus should also be shifted toward Jackfruit leaf as a sustainable source of cellulose.

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DECLARATION OF CONFLICTING INTEREST

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CONCLUSIONS

This research explored the influence

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