DURABILITY STUDY OF FLUTED PUMPKIN STEMS FIBER (FPSF) FOR THE DEVELOPMENT OF NATURAL FIBER REINFORCED PLASTICS (NFRP)

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Abstract
This research article investigated the development of Fluted Pumpkin stem fiber (FPSF) for Natural Fiber Reinforced Plastics (NFRP). Emphasis was on the fiber development process, fiber extraction, surface treatment and characterization. In this study we look into micro structural properties, the density and tensile test of Fluted Pumpkin stem fiber (Telfairia occidentalis fiber), with the finer detail application of Fourier Transform Infrared spectroscopy (FTIR) and Scanning electron microscope (SEM); SEM was used to characterize the surface, inter surface and other dynamic properties of Telfairia occidentalis treated and untreated fibers. While the spectral analysis using Infra-red spectroscopy gives broad information on the qualitative and quantitative analysis of these fibers, that leads to elucidation and identification of chemical groups and interferes structure property relationship of the fibers. Using FTIR-8400S, by clamping 2.5cm sample in a compressed disc, the sample scanned between 4000 to 620.1559 cm^{-1}, and the spectra was recorded and studied.

1. Introduction

The amount of agricultural waste generated in the African region annually demonstrates the inadequacies of various African countries to follow the global trend of recycling [15]. [13], reported that large quantities of waste produced in Nigeria are underutilized yearly. The burning of agrowastes and the inappropriate disposal of wastes (such as open dumping) are some of the regular practices of waste disposal and management in Nigeria [13]. The growing ecological concern and governmental laws have lead to a rise in the demand for natural fibers as a substitute for synthetic fibers in the production of modern materials [8-10]. Generally, materials developed from agrowastes are considered environment-friendly and biodegradable materials [13]. These materials are now becoming the centre of attraction because of the numerous advantages offered by them [21]. Apart from providing a clean environment for production, these materials are also inexpensive, which further increases the interest of the scientific and engineering community to explore the possibilities of using these materials in various engineering applications [15].

Hence, the search for better engineering materials has intensified efforts towards the development of composite materials as an alternative or replacement for ceramic and metallic materials [14]. Composite materials can be classified as particle-reinforced, fiber-reinforced, and structural [22]. Their properties depend basically on the properties and relative amount of the constituent phases, the shape, size, orientation, and distribution of the dispersed phase [19-22]. In this research study, a new variant of natural fiber called Fluted Pumpkin Stem Fiber has been introduced, characterized, and model for the development of Fluted Pumpkin stem fiber (FPSF) for Natural Fiber Reinforced Plastics (NFRP). The importance of understanding these fiber surfaces and micro-structures helps in designing and engineering a particular material product.

1.1. Review Of Related Literature

The constant advancing of computer and computing science has made infrared spectroscopy techniques striding further: The availability of a dedicated computer, which is required for the FTIR instrumentation, has allowed the digitized spectra to be treated by sophisticated data processing techniques and increased the utility of the infrared spectra for qualitative and quantitative purposes [1]. Cellulose, which acts as the reinforcing material in the cell wall, is the main constitute in natural fibers [12]. The cellulose molecules are laid down in microfibrils in which there is extensive hydrogen bonding between cellulose chains, producing a strong crystalline structure [20].

Fourier Transform Infrared spectroscopy (FTIR), is nowadays one of the most important analytical techniques available to scientists [28]. One of the greatest advantages of infrared spectroscopy is that virtually any sample in any state may be analyzed [23-29]. For example, liquids, solutions, pastes, powders, films, fibers, gases and surfaces can all be examined with a judicious choice of sampling technique. While, The Scanning Electron Microscopy (SEM), is routinely used to generate high-resolution images of shapes of objects and to show spatial variations in chemical compositions: (1) acquiring elemental maps or spot chemical analyses, (2)discrimination of phases based on mean atomic number (commonly related to relative density) [18; 21-24] The review by [2], demonstrates the applicability of dispersion infrared spectroscopy for natural fibers studies Fourier transform infrared spectroscopy (FTIR) has facilitated many different IR sampling techniques, including attenuated total reflection and diffuses reflectance infrared Fourier transform (DRIFT) spectroscopy. It has dramatically improved the quality of infrared spectra and minimized the time required to obtain data. The increased speed and higher ratio of signal-to-noise of FTIR relative to dispersion infrared has lead to a substantially greater number of applications of infrared in natural fibers research.

[1], Study the Qualitative and Quantitative characterization of textile material by Fourier transform infrared. International Journal of innovative research in science, engineering and technology. The mechanical properties of linear low-density polyethylene (LLDPE) reinforced with dodecyl amine-modified graphene were investigated by Kuila. Their study showed improvements in the storage moduli and thermal stability of the nanocomposites with increasing dodecyl amine-modified graphene content [24]. [27], Investigated the Quantitative fiber mixture analysis by scanning electron microscopy: Part VI: Possibility and limitations of the analysis of binary speciality fiber/wool blends in view of test method IWTO-58. Textile Research Journal 73(9): 781-786. [26] Studied the Quantitative analysis of fiber mixture analysis by scanning electron microscopy: Part V: Analyzing pure fiber samples and samples with small admixtures according to test method IWTO-58. Textile Research Journal 73(8): 727-732.

2. METHODS

2.1. Raw Material source

Fluted pumpkin stems of about seven to eight months old species of Telfairia occidentalis were harvested from the farm garden, at the lower Anambra/Imo River basin in the south-east region, Nigeria. Telfairia occidentalis is a tropical vine grown in West Africa as a leaf vegetable and for its edible seeds. Otherwise known as fluted gourd, fluted pumpkin stem, okporo-ugu, and ikong-ubong, in their native dialect [12]. It belongs to the Cucurbitaceae family and is indigenous to southern Nigeria. The fluted pumpkin stems were collected after used as an agricultural waste material near a market dump-site source in Ehime Mbano council area, Imo State, Nigeria. The collected fluted pumpkin stems were washed with water to remove any dirt and then cut between anti-nodes into nodes as in Plates 3.3 and 3.4.

2.2. Fluted Pumpkin Stem Fiber Extraction and Treatment Method

In this research study, the retting process involves the use of longitudinal sections (NODES) of Fluted pumpkin stem between the ages of seven to eight months. Fluted pumpkin stem was sliced into small slabs (dimension) of about 10 to 20cm in length with a slicer. The Fluted pumpkin stems were soaked in water for 6 days. After which the fiber strands were soaked in separate concentrations of 2% and 3% mass over volume of sodium hydroxide (NaOH), respectively, for a minimum duration of twenty-four (24) hours and press with a roller lesser, washed in diluted acetic acid solution to influence the cellulosic and non-cellulosic parts. These steps are intended to dissolve the cellulosic parts, leaving lignin to float on top as a black substance, as in Plates 3.5 and 3.6, and

finally, the fibers were rinsed and cleansed with distilled water and dried under high sun intensity for 48 hours, as in Plates 3.7 and 3.8. Considering the high amount of lignin contained in Fluted pumpkin stem as compared with other natural stem fibers, the chemical method or alkaline retting process was employed to extract the Fluted pumpkin stem fibers. This method aids in removing the amorphous regions of the Fluted pumpkin stem and reduces the lignin content of the elementary fiber [24-25]. The steps for the development and extraction of the Fluted pumpkin stem fibers for Natural Fiber Reinforced Plastics (NFRP) were outlined as in plates 3.1 to 3.9 below.

Figure 1. Morphology of Fluted Pumpkin Stem

Figure 1 and 2 depict the fiber Cellulose, which acts as the reinforcing material in the cell wall. It is the main constituent in natural fibers. The cellulose molecules are laid down in microfibrils, in which there is extensive hydrogen bonding between cellulose chains, producing a strong crystalline structure.

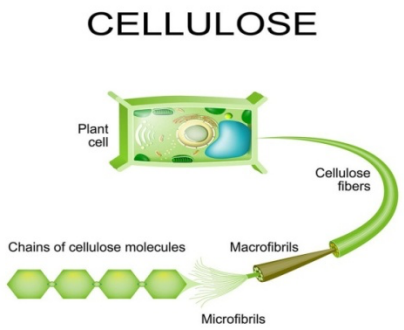


Figure 2. Structure of cellulose cell wall and microflbris of Fluted pumpkin stem

Figure 3. Fluted pumpkin stem from dump-site

Figure 4. Fluted pumpkin stems cut into nodes

Figure 5. Fluted pumpkin stems soaked in water

Figure 6. Fluted pumpkin stems of untreated and treated fibers

Figure 7. Fluted pumpkin stem treated and untreated fibers on intensity sun

Figure 8. Ground fluted pumpkin stem fibers treated and untreated

2.3. Fiber Characterization

This research test experiment is in accordance with the provisions of the American Society for Testing and Materials (ASTM). In this research study, a Tensile test experiment was conducted on the Fluted pumpkin stem fiber using a Universal Materials Testing Machine (2500 KGF Capacity-Cat. PWG25W**,** M500 to 250T) at Turret Engineering Services Ltd, Port Harcourt, Rivers State, Nigeria. While Fourier Transform Infrared spectroscopy (FTIR) and Scanning electron microscope (SEM) were conducted at DICON Research and Development centre, Kakuri, Kaduna State, Nigeria.

2.3.1. Tensile Test

The tensile tests were performed according to ASTM D638 standard using a Universal Testing Machine at a crosshead speed of 5 mm/min. Fluted pumpkin stem fiber samples (Treated and untreated) were characterized through which the tensile strength and tensile modulus were expressed as in equations (1) and (2), see table 3.1 and table 3.2, for the results [4-5] Tensile strength (MPa) = $\frac{P}{bh}$Tensile modulus (MPa) = $\frac{\rho}{\varepsilon}$.

Where P = Pulling force (N), b =Sample width (m), h = Sample thickness (m), ρ =Stress (N/m^2), ε = Strain

2.3.2. Determination of the Moisture Content of Telfairia occidentalis Fiber

Moisture contents of Telfairia occidentalis fiber samples were tested by Humidity Cabinet LHL-112 equipment using ASTM D2495-07 standard [7]. 2g of each of the sample Telfairia occidentalis fiber was weighed, transferred into a petri dish, and then dried in an oven for 3 h at 105°C to a constant weight. The moisture content (M%) was then computed based on the initial air-dried weight using equation (3) [8-9]:

$$M\% = \frac{Wt - Wo}{Wo} x100$$

Where Wt is the weight of the sample at time t, and Wo is the weight of the initial sample after drying.

2.3.3. Determination of the True Density of Telfairia occidentalis Fiber

The volume of the grinded Telfairia occidentalis fiber was accurately determined using the Bulk and Tapped density principle

Bulk density (Bd)

5g of the Telfairia occidentalis fibers was weighed and transferred into a 50 ml clean, dry measuring cylinder. The occupied volume by the sample without tapping was noted as the bulk volume, while the bulk density was computed by dividing the mass of the sample by its bulk volume as expressed in (4) [6-9]:

$$\mathbf{B_d} = \frac{M}{VB}$$

Where M is the mass of the Telfairia occidentalis fiber, V_B is the bulk volume of the Telfairia occidentalis fiber.

Tapped density (Td)

The measuring cylinder containing the Telfairia occidentalis fiber was then tapped on a wooden platform by dropping the cylinder from a height of one inch at 2-second intervals until there was no observable change in volume reduction. The volume occupied by the material was recorded as the tapped volume. The tapped density was determined using the expression in equation (5) [9-10]:

$$\mathbf{T_d} = \frac{\mathrm{M}}{\mathrm{VT}}$$

Where M is the mass of the Telfairia occidentalis fiber, V_T is the tapped volume of the Telfairia occidentalis fiber.

True density (Ttd)

The liquid displacement method was adopted in determining the true density. This was carried out using the immersion fluid xylene and immersing the sample completely in a pycnometer bottle 25 ml capacity. The volume of the liquid displaced was measured, and the density of the Telfairia occidentalis fiber was computed as in equation (6) [9; 12-17];

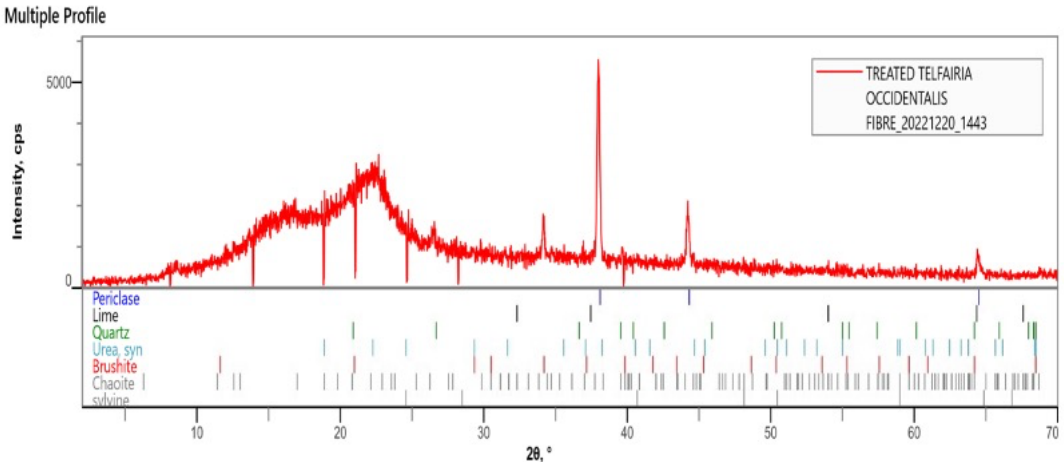
$$\mathrm{Ttd} = [\mathrm{w}/\{(\mathrm{a} + \mathrm{w}) - \mathrm{b}\} \times \mathrm{SG}]$$

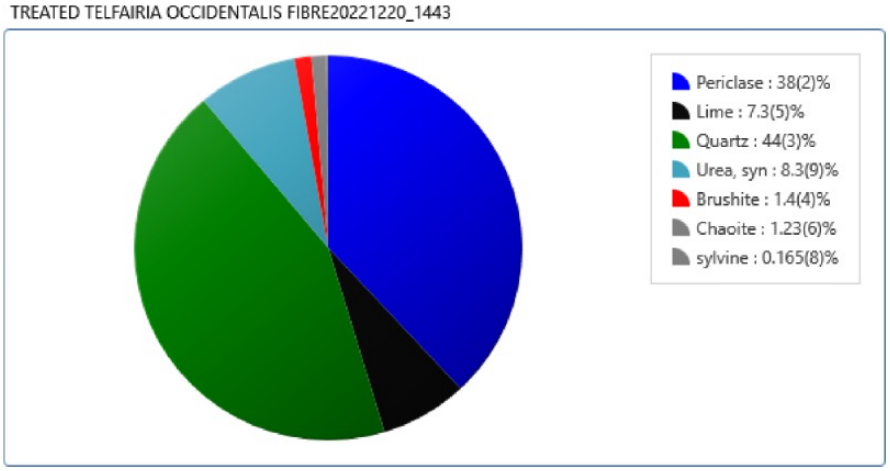
Where w is the weight of Telfairia occidentalis fiber (Filler), **SG** is specific gravity of xylene, **a** represents the sum of weights of bottle and solvent, and **b** represents the sum of weights of bottle, solvent, and the Telfairia occidentalis fiber (Filler).

Scanning Electron Microscopy (SEM) and Fourier Transform (FTIR) Spectroscopy

SEM was carried out to study the micro surface morphology, inter surface, and other dynamic properties of Telfairia occidentalis fibers, which can image these fiber surfaces at different magnifications which thus facilitating the observations to be examined at varying conditions as presented in figures 3.10 to 3.11. While spectral analysis using FTIR gives broad information on the qualitative and quantitative analysis of these fibers, as presented in figures 3.1-3.2 and figures 3.3-3.4, which helps in elucidation and identification of chemical groups and interferes structure-property relationship of the fibers.

Analysis date	2022-12-22 14:40:17	Measurement start time	2022-12-20 14:44:27
Analyst	Administrator	Operator	Administrator
Sample name	TREATED TELFAIRIA OCCIDENTALIS FIBRE	Comment	
Measured data name	C:\Users\Rigaku pc\Desktop\ANALYSIS\OGA\20-12-2022\TR...	Memo	





Dataset / Weight Fr	Value,	Periclase	Lim	Quartz	Urea, syn	Brushite	Chaoite	sylvin
TREATED ..	0	38(2	7.3(5	44(3	8.3(9	1.4(4	1.23(6	0.165(8

Figure 9. Plot and Table showing chemical compositions of Telfairia occidentalis treated fibers

Analysis date	2022-12-22 14:40:17	Measurement start time	2022-12-20 14:44:27
Analyst	Administrator	Operator	Administrator
Sample name	TREATED TELFAIRIA OCCIDENTALIS FIBRE	Comment	
Measured data name	C:\Users\Rigaku pc\Desktop\ANALYSIS\OGA\20-12-2022\TR..	Memo	

Qualitative Analysis Results

Phase name	Formula	Figure of merit	Phase reg. detail	Space Group	DB Card Number
Periclase	Mg O	0.830	S/M(PDF-4 Minerals 2021)	225 : Fm-3m	01-085-5633
Lime	Ca O	3.321	S/M(PDF-4 Minerals 2021)	225 : Fm-3m	00-003-1123
Quartz	Si O2	2.922	S/M(PDF-4 Minerals 2021)	154 : P3221	00-001-0649
Urea, syn	C H4 N2 O	1.784	S/M(PDF-4 Minerals 2021)	113 : P-421m	00-008-0822
Brushite	Ca H P O4 ·2 H2 O	1.439	S/M(PDF-4 Minerals 2021)		00-001-0395
Chaoite	C	3.106	S/M(PDF-4 Minerals 2021)	147 : P-3	00-022-1069
sylvine	K Cl	3.142	Import(PDF-4 Minerals 2021)	225 : Fm-3m	01-076-3364

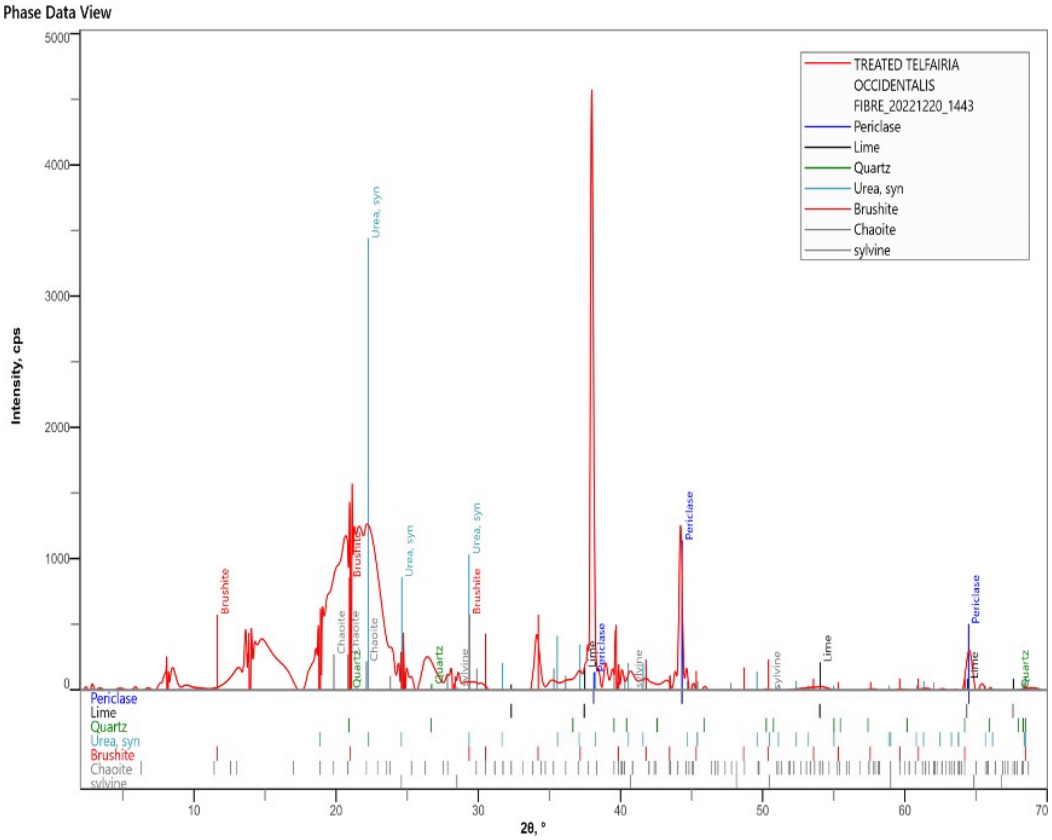
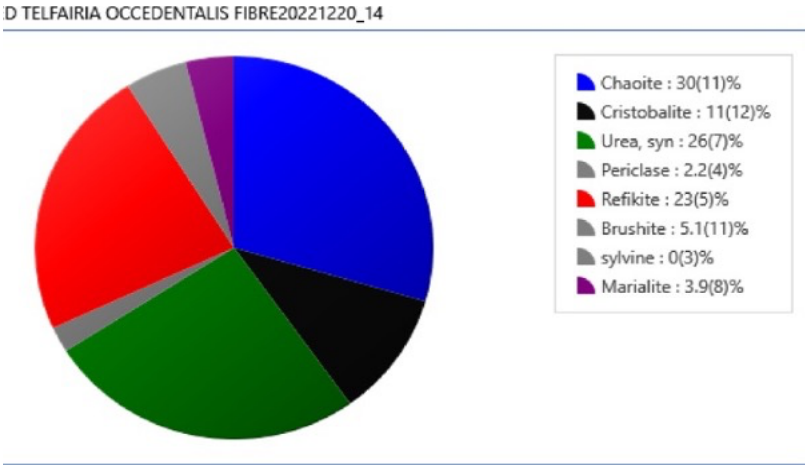
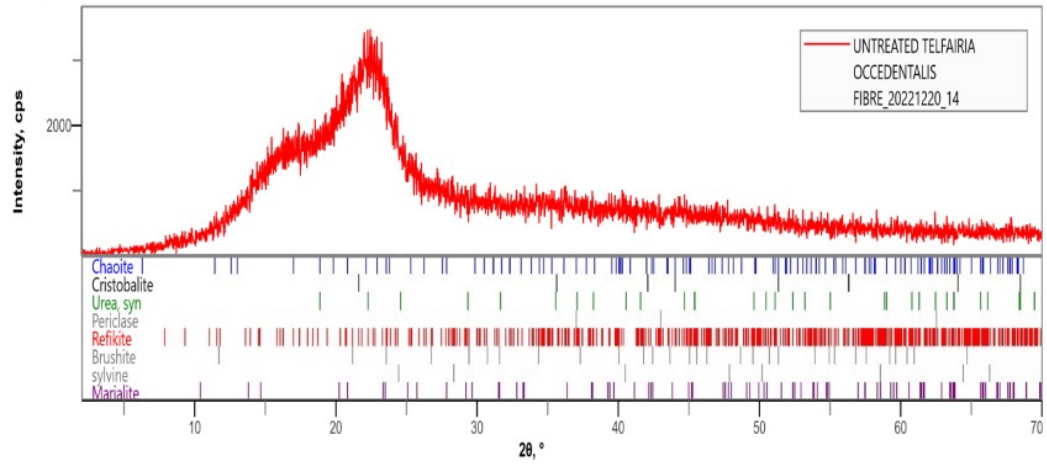


Figure 10. Quantitative analysis report (Solution) on treated Telfairia occidentalis fiber.



Analysis date	2022-12-22 14:46:52	Measurement start time	2022-12-20 14:48:18
Analyst	Administrator	Operator	Administrator
Sample name	UNTREATED TELFAIRIA OCCEDENTALIS FIBRE	Comment	
Measured data name	C:\Users\Rigaku pc\Desktop\ANALYSIS\OGA\20-12-2022\UN...	Memo	

Multiple Profile



Dataset / Weight	Value,	Chaoit	Cristobalit	Urea,	Periclas	Refikit	Brushit	sylvin	Marialit
UNTREATED ..	0	30(11	11(12	26(7	2.2(4	23(5	5.1(11	0(3	3.9(8

Figure 11. Plot and Table showing chemical compositions of Telfairia occidentalis untreated fibers.

Analysis date	2022-12-22 14:46:52	Measurement start time	2022-12-20 14:48:18
Analyst	Administrator	Operator	Administrator
Sample name	UNTREATED TELFAIRIA OCCEDENTALIS FIBRE	Comment	
Measured data name	C:\Users\Rigaku pc\Desktop\ANALYSIS\OGA\20-12-2022\UN...	Memo	

Qualitative Analysis Results

Phase name	Formula	Figure of merit	Phase reg. detail	Space Group	DB Card Number
Chaoite	C	1.276	Import(PDF-4 Minerals 2021)	147 : P-3	00-022-1069
Cristobalite	Si O2	1.510	Import(PDF-4 Minerals 2021)	227 : Fd-3m:2	00-001-0424
Urea, syn	C H4 N2 O	0.685	Import(PDF-4 Minerals 2021)	113 : P-421m	00-008-0822
Periclase	Mg O	2.850	Import(PDF-4 Minerals 2021)	225 : Fm-3m	00-002-1207
Refikite	C20 H32 O2	0.489	Import(PDF-4 Minerals 2021)	18 : P21212	00-028-2009
Brushite	Ca H P O4 ·2 H2 O	1.704	Import(PDF-4 Minerals 2021)		00-002-0085
sylvine	K Cl	3.141	Import(PDF-4 Minerals 2021)	225 : Fm-3m	01-076-3361
Marialite	(Na3.21 Ca0.68 K0.11) (Si8....	1.450	Import(PDF-4 Minerals 2021)	87 : I4/m	01-070-6156

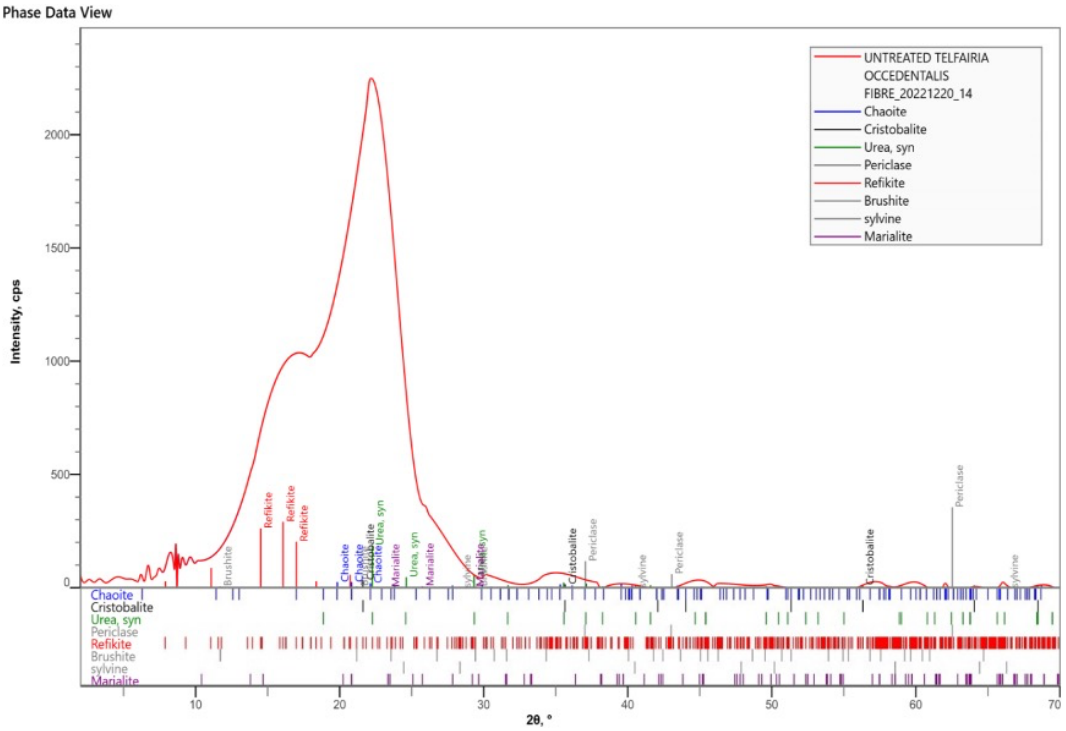


Figure 12. Quantitative analysis report (Solution) on untreated Telfairia occidentalis fiber

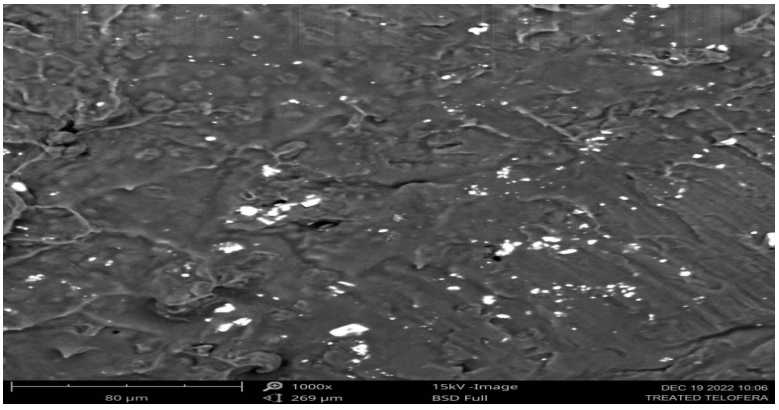


Plate 3.10: Scanning electron micrograph images of a treated fluted pumpkin stem fiber

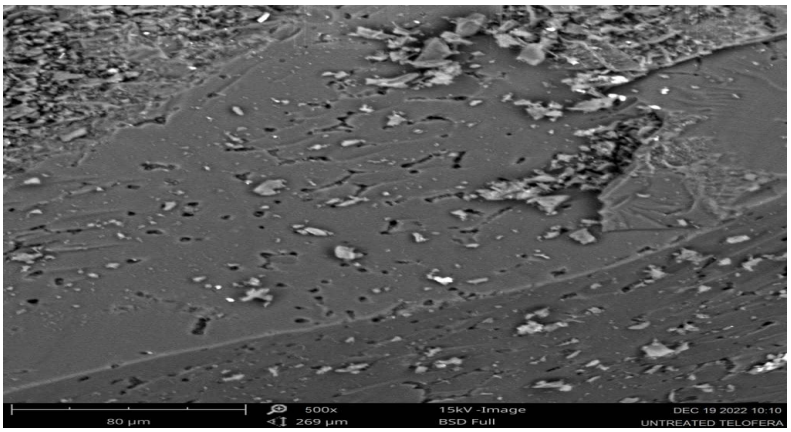


Plate 3.11: Scanning electron micrograph images of untreated fluted pumpkin stem fiber

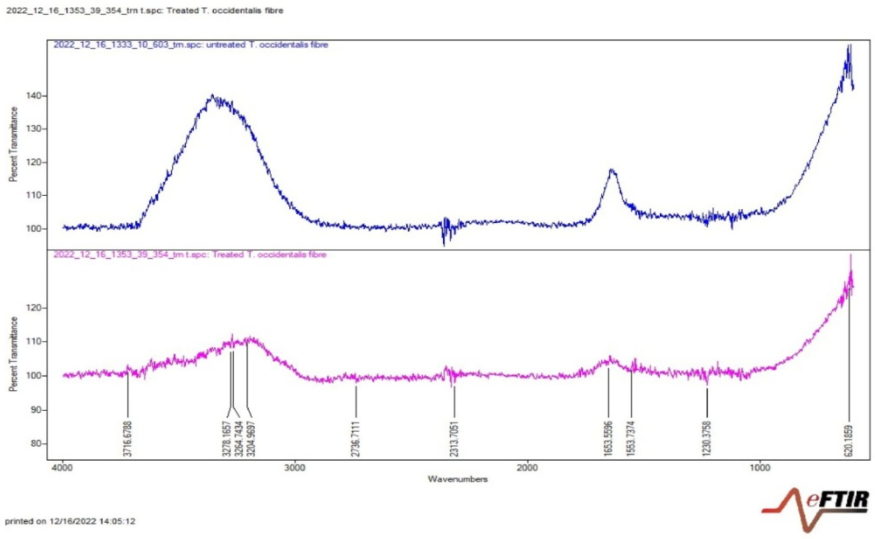


Figure 13. Fourier transform infrared (FT-IR) of treated and untreated fluted pumpkin stem fiber.

Table 1. Physical properties of the fluted pumpkin stem fiber.

Parameters	Treated	Untreated
Moisture content (%)	5.26	6.10
Bulk density (g/ml)	0.20	0.25
Tapped density (g/ml)	0.45	0.54
True density (g/ml)	1.42	1.45

Table 2. The tensile test result of the fluted pumpkin stem treated fiber

Comments: The tensile test result of fluted pumpkin stem treated fiber using 2% concentrations over volume of sodium hydro-oxide (NaOH).

Test No	Force @ Peak (N)	Elong. @ Peak (mm)	Stress @ Peak (N/mm)	Strain @ Peak (%)	Strain @ Break (%)	Youngs Modulus (N/mm)	Width (mm)
1	124.2	2.008	124.2	0.802	14.846	28219.82	1.0
Test No	Thickness (mm)	Elong. @ Yield (mm)	Elong. @ 0.000 % Proof	Force @ Break (N)	Force @ 0.000 mm (N)	Force @ Yield (N)	Force @ 0.000 % (N)
1	1.0	1.96	0.217	0.0	5.0	122.5	5.0
Test No	Force @ Peak (N)	Plastic Strain @ Break (%)	Poisson's Ratio	Stress @ Yield (N/mm)	Percent Reduction of Area (%)	Strain @ Yield (%)	Secant Stiffness 0.000 to 0.000 % (N/mm)
1	124.2	14.846		122.5	0.0	0.783	
Test No	Stress @ 0.000 % Proof (N/mm)	Stress @ 0.000 mm (N/mm)	Stress @ Break (N/mm)	Elong. @ Break (mm)	Strain after Fracture (%)	Energy to Break (N.m)	Elong. @ Peak (mm)
	28.349	5.0	0.0	37.15	-0.096	0.541	2.008
Test No	Energy to Peak (Nm.)	Strain @ Upper Yield (%)					
1	0.152	0.783					

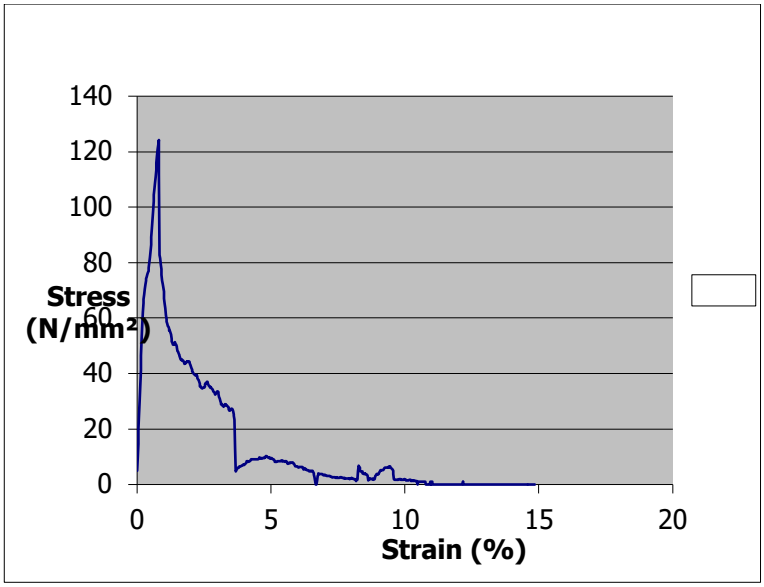


Figure 14. Tensile test result of treated fluted pumpkin stem fiber

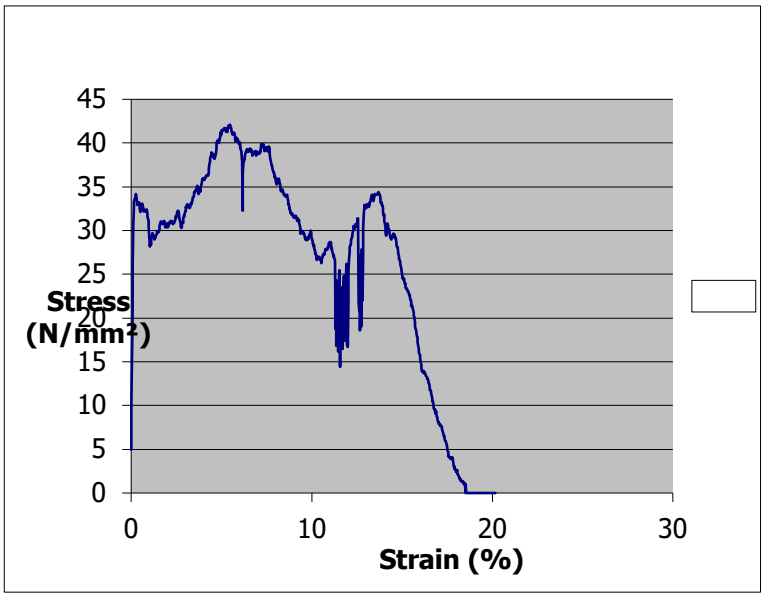


Figure 15. Tensile test result of untreated fluted pumpkin stem fiber

Table 3. The tensile test results of the fluted pumpkin stem untreated fiber.

Comments: The fluted pumpkins stem untreated fiber using 2% mass over volume of sodium hydro-oxide (NaOH).

Test No	Force @ Peak (N)	Elong. @ Peak (mm)	Stress @ Peak (N/mm²)	Strain @ Peak (%)	Strain @ Break (%)	Youngs Modulus (N/mm²)	Width (mm)
1	42.1	10.969	42.1	5.481	20.168	21439.936	1.0
Test No	Thickness (mm)	Elong. @ Yield (mm)	Elong. @ 0.000 % Proof (mm)	Force @ Break (N)	Force @ 0.000 mm (N)	Force @ Yield (N)	Force @ 0.000 % (N)
1	1.0	0.53	0.033	0.0	5.0	34.2	5.0
Test No	Force @ Peak (N)	Plastic Strain @ Break (%)	Poisson's Ratio	Stress @ Yield (N/mm²)	Percentage Reduction of Area (%)	Strain @ Yield (%)	Secant Stiffness 0.000 to 0.000 % (N/mm)
1	42.1	20.168		34.2	0.0	0.265	

Test No	Stress @ 0.000 % Proof (N/mm^2)	Stress @ 0.000 mm (N/mm^2)	Stress @ Break (N/mm^2)	Elong. @ Break (mm)	Strain after Fracture (%)	Energy to Break (N.m)	Elong. @ Peak (mm)
	8.203	5.0	0.0	40.363	-0.068	1.054	10.969
Test No	Energy to Peak (N.m)	Strain @ Upper Yield (%)					
1	0.369	0.265					

3. RESULTS AND DISCUSSIONS

3.1. The chemical composition of the fibers was determined by using FTIR.

From Figure 3.5, the samples displayed broad absorption within the band region of 3716.6788 to 620.1859 cm^{-1} of Telfairia occidentalis fiber. Bands between 3278.1657 cm^{-1} to 3264.7434 cm^{-1} depict O-H stretching vibration due to intermolecular hydrogen bonding. The spectra band at 3204.9697 cm^{-1} to 1633.5596 cm^{-1} is due to S_iO_2 and MgO being dominant, and the occurrence of this band is attributed to the strong interaction between cellulose and the water molecules. A peak was observed at 326.434 cm^{-1} and 1653.5596 $cm^{-1,}$ which indicates a trace of hemicellulose for the Telfairia occidentalis fiber samples. Also, the studies have shown that the band at 2736.7111 to 2313.7051 cm^{-1} corresponds to either the acetyl or uronic ester groups of CH_4 N_2O or the ester linkage of the carboxylic group of C_2OH of lignin and/or hemicellulose. The CH deformation vibration was at 2313.7015 $cm^{-1,}$ while the CH_2 rocking vibration was denoted by bands at 1230.3758 cm^{-1}. The band at 1230.3758 cm^{-1} is a symmetric in-phase ring stretching, C-C and C-O stretching. The regions assigned to 620.1859 cm^{-1} are due to the C-H out-of-plane asymmetric ring stretching in cellulose of the Telfairia occidentalis.

3.2. Morphologies of natural fibers by using SEM

Figures 3.10 and 3.11 are the scanning electron micrograph images of Telfairia occidentalis treated and untreated fibers at 1000 and 500 magnifications, respectively. Telfairia occidentalis treated fiber images showed a smooth surface, long individual rod-like shaped fiber structures with a few bundled crystal-packed forms. In contrast, the Telfairia occidentalis untreated fiber has less smooth and coarse, shorter fibers, which exist as irregular individual flat-shaped plank-like fibers. The unevenness of their surfaces may be due to the processing and treatment conditions that led to the elimination of some binding materials around their fiber bundles. The surface coarseness assists in nanocrystals production through hydrolysis. These are reflected in the physical properties of the Telfairia occidentalis fiber samples.

In this study, FTIR and SEM have proved to be powerful scientific techniques. Furthermore, FTIR can provide researchers with further information on the supermolecular structure. FTIR can also be used to determine the chemical linkage compositions of native natural fibers and modified natural fibers.

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