



Characterization of alkaline treated raffia palm fibres as reinforcement in polymer composite



Ipilakyaa T, Daniel^{*}, Tile S. Emmanuel, Nyior G. Bem, Gundu D. Terfa

Mechanical Engineering Dept., Joseph Sarwuan Tarka University-Nigeria, Benue State- Nigeria.

*Corresponding author Email: ipilakyaa@yahoo.com

HIGHLIGHTS

- This research characterizes raffia palm fiber (RPF) for use in specialized polymer composite reinforcement.
- The study found that the alkaline-treated RPF presented a tensile strength of 195 MPa and Young's modulus of 91.76 MPa.
- The XRD results show that RPF is semi-crystalline, with the crystalline index estimated at 57.3%.

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ABSTRACT

The characterization of raffia palm fibre (RPF) for reinforcement in polymer composite for specialized applications was studied. The fibres were treated with NaOH solution and subjected to a tensile strength test. The fibres were also subjected to spectroscopy using Fourier transform infrared (FTIR), energy dispersive x-ray fluorescence (EDXRF), and x-ray diffraction (XRD). The study found that the alkaline-treated RPF presented a tensile strength of 195 MPa and Young's modulus of 91.76 MPa. The strain at maximum stress in the fibre was found to be 2.125%. FTIR spectra of treated raffia palm fibres revealed that the fibres are characterized by O-H and C-H stretching. There is the presence of carboxylic acids, functional groups of methyl (CH₃), methylene (CH₂), and aliphatic saturated (CH) compounds. There was no observed peak at around 1000 cm⁻¹ to 650 cm⁻¹ bands characterized as the C-H "oop" bond structure of a functional group of aromatics. The absence of a peak in the range indicates that the alkaline treatment of RPF has been effective, as evidenced by the absence of smell. EDXRF showed significant Ca, Fe, and K concentrations in raffia palm fibres. The XRD results show that RPF is semi-crystalline, with the crystalline index estimated at 57.3%. Therefore, RPF can be used as potential reinforcement in polymer composite applications where moderate strength and stiffness are required. From the spectroscopy, RPF is safe for deployment as an alternative source of reinforcement in polymer composites, especially for developing biomedical applications such as prosthetics and wheelchairs.

1. Introduction

Fibres are the principal load-carrying component of a composite. The characteristics of the fibre significantly influence the mechanical properties of the composite produced from it. Due to environmental concerns, the use of natural fibre in the composite is encouraged. Natural fibres can replace synthetic fibres in polymer composite materials because of their low cost, low density, easy production, high specific properties, biodegradability, and environmentally friendly Samuel et al., [1]. One type of natural fibre with the potential to be developed is the raffia palm fibre, which is abundant in Nigeria. Raffia palm fibre is a natural vegetable fibre extracted from the raffia palm tree leaflets. According to Elenga et al. [2], there are about 28 different species of raffia palm trees that grow in the tropics, with the fibre having different properties due to geographical locations and growing and harvesting conditions. Fadele et al. [3] studied the properties of raffia palm fibres (RPFs) derived from the raffia palm tree (*Raphia farinifera*). The results revealed that the main constituents of RPFs are cellulose, hemicellulose, and lignin. The study also reported that, like many other natural fibres, raffia fiber has good specific strength, is lightweight, readily available, renewable, environmentally friendly, low cost, skin-irritation free, and has good adhesion with resin. Also, Elenga et al. [4] reported that Raffia palm fibre has high tensile strength, which is a potential reinforcement for composites. There are few studies on raffia palm fibers' mechanical properties and spectroscopy analysis Elenga et al. [4] Elenga et al., [2]. Sandy and Bacon [5], and Fadele et al., [3]. This study fills the gap in the current knowledge of these fibres

by providing additional information on the properties of the raffia palm fibres grown in Benue state, Nigeria, which will help assess their potential use as reinforcements in polymer composites.

Recently, natural fibre composites have been applied in the production of biomedical devices such as prosthetic materials because the products are structural and external to the body. The requirements of prosthetic materials, which are good strength, low weight, durability, size reduction, safety, and energy conservation, have made natural fibre-reinforced plastics very attractive in this area.

This study aims to characterize alkaline-treated raffia palm fibres from palm trees grown in Benue State, Nigeria to assess their potential use as alternative reinforcement materials in composite materials as a substitute for glass fibres in biomedical applications. The use of glass fibres in biomedical applications is a concern; according to the US Department of Health (2011) [6], fibre glass causes sensitivity to the skin (skin itch) when it comes in direct contact with the skin. For the workers who produce the devices, it causes skin allergies and allergic bronchitis, which may lead to lung cancer due to material volatility in the air during processing (U.S. Department of Labour. 2002) [7]. Also, the high carbon composition of glass fibres causes problems for the environment as they are non-degradable and high-density materials [8]. Consequently, with the advantages of low cost, light weight, non-toxicity, and friendly to the environment, raffia palm fiber will be characterized to assess its properties as an alternative material for producing prosthetic devices. Characterization of the raffia palm fibres in this study will involve studying the tensile properties of the fibre and spectroscopy analysis of RPF to determine the elemental composition of the RPF, identifying specific functional groups or compounds present in RPF aiding in a detailed understanding of their structure and properties for engineering design applications.

2. Methods

2.1 Raffia fibre preparation

The raffia palm fibres used in this study were collected from raffia palm trees around the Chia-Aku stream in Ikov, Ushongo Local Government of Benue State. The pinnate leaves of the raffia palm (*Raphia*) were pulled out from the leaf stalks. After that, the raffia fibres were taken off from the pinnate leaves. The fibres were washed thoroughly and allowed to dry under the sun. The fibres were treated using alkali by soaking one gram of clean raffia fibres in 10 ml of 10% sodium hydroxide solution for 1 hour at room temperature. The alkaline treated fibres were washed thoroughly in distilled water to remove the excess NaOH (or non-reacted alkali). The fibres were allowed to dry under the sun. Plate 1 shows raffia palm fibres. Individual long fibres were cut for the tensile test, and the fibres for spectroscopy were crushed to a particulate size of 300 μm .



Plate 1: Raffia palm fibres

2.1.1 Tensile test

The experimental procedure to determine the ultimate tensile strength and Young's modulus of single fibres of raffia palm was carried out using a digital Tensile testing machine in the Polymer and Textile Engineering laboratory Ahmadu Bello University (ABU) Zaria Nigeria in accordance with the ASTM D638 procedure. The test was conducted at room temperature. The diameters and cross-sectional area of each fibre used in the test were measured and recorded. The fibres were mounted individually into the grips of the tensile testing machine with the help of cellophane tape. A cellophane tape was used to fasten both ends of the fibres to prevent slippage and fracture of the fibre at the points of grip. The dimensions of the fibres were $60 \times 6.24 \times 0.06 \text{ mm}^3$ (length x width x thickness). The gauge length was 40 mm, as shown in Plate 2. The test was conducted by gripping each end of the fibre and slowly pulling it with a cross-head speed of 2 mm/min until catastrophic failure occurred. Five samples were tested, and the average value was taken with a standard deviation 2.61. The graph of tensile stress (MPa) versus strain (%) is shown in Figure 1. Based on the tensile test, tensile strength and strain at maximum tensile stress were obtained, and Young's Modulus of the fibre was calculated using Equation (1). The results are presented in Table 1.

$$\text{Young's modulus (MPa)} = \frac{\sigma}{\varepsilon} \quad (1)$$

where: σ is the stress (MPa), ε is the strain

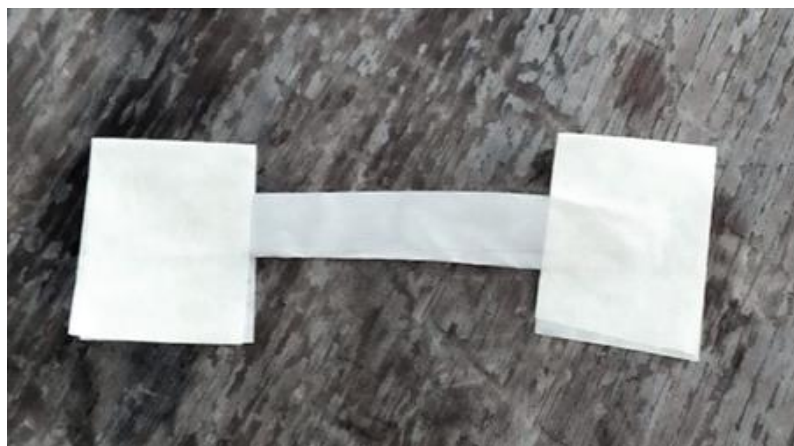


Plate 2: Prepared sample of Raffia palm fibre for tensile testing

2.1.2 Fourier transform infra-red (FTIR) spectroscopy

The raffia palm fibres were crushed to a particulate size of 300 μm . The sample was kept in a container at room temperature. Using an Agilent FTIR machine, the FTIR spectroscopy was performed at the Umaru Musa Yar-adia University (UMYU) central laboratory in Katsina State, Nigeria. The test was conducted with a sample scan of 64, a resolution of 16, and a spectral range of 4000-650 cm^{-1} through a sample, with some radiation absorbed and some passed through. The sample molecules convert the absorbed radiation into rotational and/or vibrational energy. The resulting signal at the detector presents as a spectrum, representing a molecular fingerprint of the sample. Each molecule or chemical structure will produce a unique spectral fingerprint, making FTIR analysis a great tool for chemical identification.

2.1.3 Energy dispersive X-Ray fluorescence (EDXRF) spectroscopy

An energy-dispersive XRF spectroscopy machine with a tube voltage of 20 kV, tube current of 0.70 mA, and a live time limit of 120 seconds was employed as a primary source to determine trace element concentrations in the RPF sample. This was done at the Umaru Musa Yar-adia University (UMYU) central laboratory in Katsina State, Nigeria. The raffia palm fibres were crushed to a particulate of size 300 μm using a tabletop harmer mill, as shown in Plate 3 (a,b). The powdered sample was placed into a plastic sample cup, manually pressed to ensure a reasonable flat sample surface, and presented to the EDXRF spectrometer system. The spectra obtained from x-ray excitation were transferred to a computer where the concentration of the elements in the samples was obtained using AXIL-XRF software.



Plate 3: (a) Table top harmer mill for crushing of raffia palm fibres (b) Crushed powdered sample of raffia palm fibre

2.1.4 X-ray diffraction (XRD)

The raffia palm fibres were crushed to a particle size of 300 μm . Using a split-able pressing die with an internal diameter of 20mm and height of 100 mm, with a maximum pressing force of 15 kN, the sample was pressed into a standard sample holder so that a smooth flat surface was obtained. This was carried out at the Umaru Musa Yar-adia University (UMYU) central laboratory in Katsina State, Nigeria, using an X-ray diffractometer with a Cu target, having a maximum voltage of 30 kV and a resolution of 1 and scan axis of 2θ configuration. The current used was 10 mA, an x-ray characteristic of K-Alpha

using a continuous scanning speed. The crystalline index was determined using the established empirical equation of Segal et al. [9], shown in Equation (2):

$$C.I = \frac{I_{200} - I_{AM}}{I_{AM}} \quad (2)$$

where: I_{200} is the maximum intensity of reflection by the crystalline plane of the cellulose at 2θ , and I_{AM} is the maximum intensity of the amorphous part at 2θ .

3. Results and discussion

3.1 Tensile properties of alkaline treated raffia palm fibres

Conducting a tension test on individual fibers is valuable for assessing their tensile strength, elongation, and other mechanical characteristics. Single fiber tests yield more precise outcomes than tests conducted on bundles of fibers, as the behavior of an individual fiber is less intricate and more foreseeable than that of a bundle. This methodology can be employed to ascertain and choose fibers that possess optimal mechanical characteristics suitable for particular applications. The results obtained from the tensile testing of single raffia palm fibers are presented in Table 1.

Table 1: Tensile Properties of Alkaline Treated Raffia Palm Fibres in the present study

Sample	Tensile Strength (MPa)	Max Force (N)	Area (mm ²)	Strain at max stress (%)	Young's modulus (MPa)
Raffia palm Fibre	195	80.317	0.4160	2.125	91.76

Figure 1 shows the stress (MPa) graph against strain (%). The RPF presented a tensile strength of 195 MPa and Young's modulus of 91.76 MPa. It was also observed that the strain at maximum stress in the fibre is 2.125%.

Raffia palm fibre with a strain of 2.125% can withstand significant stretching without breaking, which might be suitable for applications with relatively low loads. With a Young's modulus of 91 MPa (0.10 GPa), the RPF can be used as a potential reinforcement in polymers in applications requiring moderate strength and stiffness.

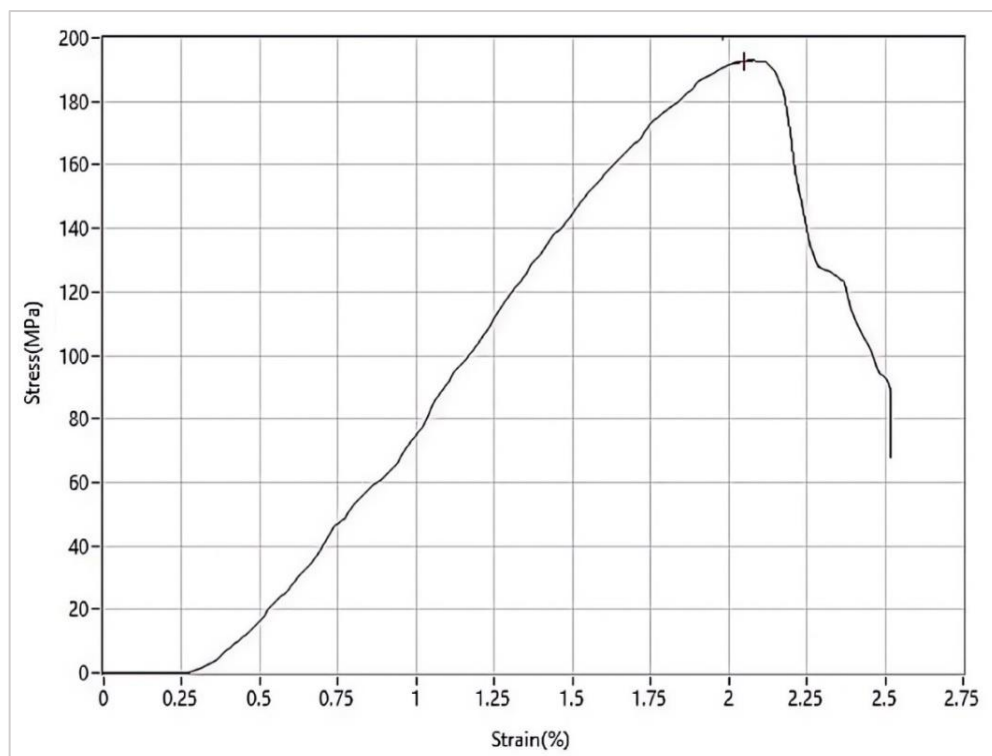


Figure 1: Stress-Strain Curve of Alkaline Treated Raffia Palm Fibres

The tensile properties of the alkaline-treated raffia palm fibre in this study compare well with the results given by Fadele et al. [3] and several other works, as shown in Table 2. Where the tensile strength of raffia fibre obtained in southern Nigeria gave average tensile strength ranging from 152 ± 22 to 270 ± 39 MPa, which is better than the tensile strength reported for other natural fibres, such as 175 MPa reported for coir fibre in Bledzki and Gassan [10] and 126.6 MPa reported in Arib et al., [11] for pineapple fibre but less than those of the other leaf fibres.

Although, the result of raffia palm fibre obtained in this study is also lower than the tensile strength value of 500 ± 97 MPa reported for *Raphia textilis* by Elenga et al. [4] and 500 ± 80 MPa reported for *Raphia farinifera* by Sandy and Bacon [5]. The differences in the species, harvest time, geographical location, and internal structures of raffia fibres investigated in the different works compared to the raffia palm fibre in this study could be the reasons for the large differences.

The average percent elongation of the raffia fibre investigated in this study, which is 2.125%, compares well with values of elongation obtained for *Raffia textilis* 2% Elenga et al. [4], *Raffia farinifera* 4% Sandy and Bacon [5], raffia palm fibre obtained from southern Nigeria $2.5 \pm 1.9\%$ Fadele et al., [3]. Pineapple fibre 2.2% Arib et al., [11], sisal fibre 2–2.5% Bledzki and Gassan [10], and flax fibre 2.3–3.0% Chinga et al., [12].

Table 2: Tensile Properties of alkaline-treated raffia Palm Fibres obtained from Benue State Nigeria Compared with Raffia Fibres in other regions of the world

Fibre	Ultimate tensile stress (MPa)	Strain at max stress (%)	Ref.
Raffia palm fibre grown in Benue Sate Nigeria	195	2.125	Present study
Raffia palm fibre obtained from Southern Nigeria	152 ± 22 to 270 ± 39 MPa	2.5 ± 1.9	[3]
<i>Raphia textilis</i>	500 ± 97 MPa	2.0	[4]
<i>Raphia farinifera</i>	500 ± 80	4.0	[5]

3.2 Fourier transform infrared (FTIR) spectroscopy results

FTIR spectroscopy was conducted on raffia palm fibres to identify the functional groups in the sample. The result of the graph of transmittance versus wave number is shown in Figure 2. The FTIR spectra of treated raffia palm fibres (RPF) show a peak at $3294.96665 \text{ cm}^{-1}$; the range of the broad absorption band at 3500 cm^{-1} to 3200 cm^{-1} for plant fibres is characterized by O-H stretching (the vibration of the bond between oxygen and hydrogen atom in a molecule), and H-bonded bond structure that mostly contains major functional groups of phenols, alcohols, and waters. Alcohols found in this fibre could be beneficial because they are often used in products due to their antimicrobial properties and ability to reduce greasiness. According to Ramadevi et al. [13], the O-H stretching, H- bonded, and free hydroxyl found in natural fibres are due to carbohydrates (hemicellulose and cellulose). Free hydroxyl groups can impact the physical and chemical properties of materials; they can increase the polarity and hydrophilicity of a compound, adhesion and biocompatibility, and absorption in biological systems. These can play a role in material design and engineering for specific applications, such as deploying this fibre in biomedical applications.

The results obtained from this study agree with the results reported in Dwivedi and Mehta [14]. An additional peak observed at $2922.23286 \text{ cm}^{-1}$ for the raffia palm fibre spectroscopy was attributed to the C-H stretching (vibration of the bond between carbon and hydrogen in a molecule) and O-H stretching bond structure that contains a functional group of alkanes (cellulose and lignin) and carboxylic acids. These could be attributed to a decrease in the functional group of phenolic or aliphatic hydroxyl in the fibers due to a reaction with sodium hydroxide that promotes free hydroxyl that caused the addition of extra peak in free hydroxyl bond structure at $2922.23286 \text{ cm}^{-1}$ for the raffia fibers. Carboxylic acids in natural fibres influence biodegradability, hydrophilicity, and adhesion.

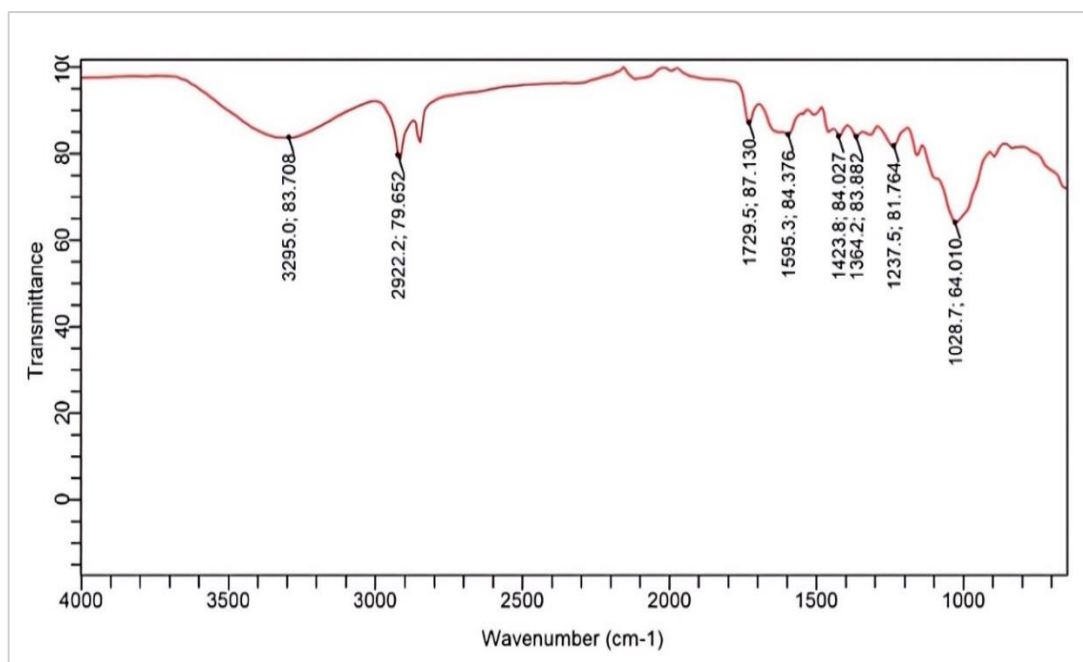


Figure 2: FTIR Spectral of Raffia Palm Fibres

According to Khalil et al. [15], a small peak in the region of the C-H stretching bond structure, as observed in this fibre, can also include a functional group of methyl (CH_3), methylene (CH_2), and aliphatic saturated (CH) compounds. Methyl groups in this fibre can impact material properties such as mechanical strength, flexibility, and durability. The small peak observed at $1729.48476\text{ cm}^{-1}$ and $1595.30059\text{ cm}^{-1}$ for RPF is characterized as the C=C stretching bond structure from the functional group of alkenes (lignin), the peak at $1423.84305\text{ cm}^{-1}$ and 1364.20565 for RPF is characterized as the C-H bending bond from the functional group of alkanes (cellulose, hemicellulose, and lignin) and the peak at $1237.47616\text{ cm}^{-1}$ and $1028.74524\text{ cm}^{-1}$ for RPF are characterized as the C-O stretching bond structure from the functional group of alcohol (cellulose, hemicellulose and lignin), carboxylic acids, esters and ethers. According to Hinterstoisser et al. [16], the peak band located in the range of 1100 cm^{-1} to 1000 cm^{-1} is also characterized as the C-O stretching bond structure of the functional group of glycosides linkage. Glycosides are sugar molecules bonded to a non-sugar molecule, such as phenol or terpene. The result obtained in this study also agrees with the study reported by Xu et al.,[17].

Furthermore, no peak at around 1000 cm^{-1} to 650 cm^{-1} bands was observed, characterized as the C-H “oop” bond structure of a functional group of aromatics. The absence of a peak in this range explains the disappearance of smell from the alkaline-treated raffia palm fibres. Aromatic compounds may be impurities or contaminants, and their absence can suggest that the sample is of high purity or has been effectively purified. This indicates that NaOH treatment of the raffia fibres was effective and enhanced the purity of fibres.

3.3 Energy dispersive X-Ray fluorescence (EDXRF) spectroscopy results

Energy dispersive X-ray fluorescence (EDXRF) was carried out on the alkaline-treated raffia palm fibres to determine the elemental composition in the fibre. The technique identified elements in the sample, offering insights into the plant fibre nutritional content and potential contaminants. The elemental composition of the RPF is revealed by the EDXRF, as presented in Table 3, while the graph of the fluorescence is shown in Figure 3. The result showed that the major elements present in raffia palm fibres (RPF) in significant concentrations are; Calcium (Ca) 0.5979%, Potassium (K) 0.11893%, and Bismuth (Bi) 0.097%.

Table 3: Electron Dispersion X-ray Fluorescence (EDXRF) of Alkaline Treated RPF

Element	Concentration (%)	Peak(cps/mA)	Background(cps/mA)
Fe	0.016190	275	15
Cu	0.001930	107	8
Ni	0.00239	4	8
Zn	0.000955	44	12
Al	0	0	1572
Mg	0.00713	45	223
Na	0.02605	15	25
S	0.04934	736	1674
P	0.07565	751	879
Ca	0.5979	3338	-171
K	0.11893	658	-28
Mn	0.033724	1887	111
Rb	0.000434	2	1
Sr	0.001694	12	1
Br	0.00026	1	1
Cl	0.01404	26	8
Cr	0	0	85
V	0	2	42
W	0.0036	2	12
Bi	0.097	1	1
Sn	0	0	49
Si	0	0	20
As	0	0	2
Nb	[0.0044]	0	2
Ta	[0.00054]	1	74
Ag	0	0	1123
Pb	[-0.060]	0	1

Calcium is an essential macronutrient that supports plant cell walls, influencing their strength, rigidity, and permeability. It contributes to the mechanical properties of plant tissues. Calcium in humans can promote bone health and reduce the risk of osteoporosis. Potassium (K) is an important constituent of the human body.

Typical toxic elements such as lead (Pb), Arsenic (As), Cadmium (Cd), Mercury (Hg), Chromium (Cr), Vanadium (V), Cobalt (Co), Tantalum (Ta) and Nickel (Ni) were absent in raffia palm fibres grown in Benue state.

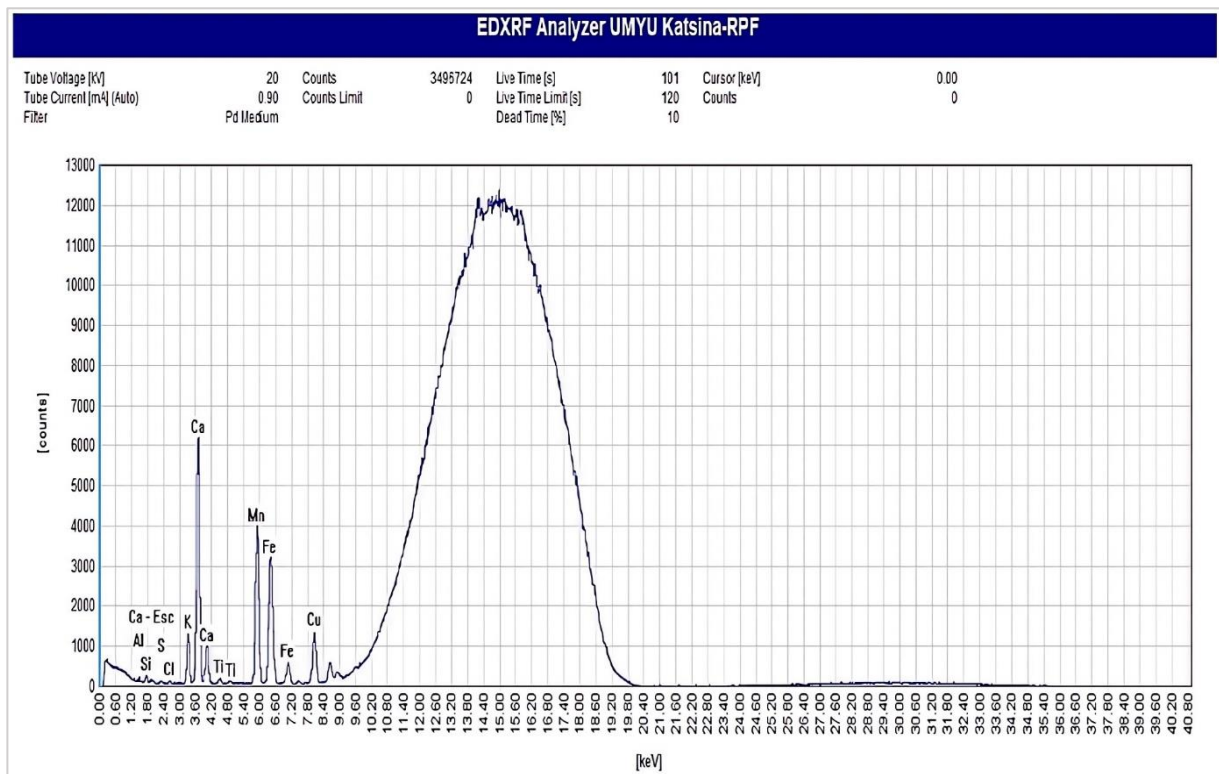


Figure 3: EDXRF Spectral of alkaline-treated Raffia Palm Fibres

3.4 X-Ray diffraction (XRD) spectroscopy results

The XRD diffractogram of raffia palm fibre shown in Figure 4 exhibits peaks commonly found in natural fibres. The most prominent peaks appear at scattering angles (2θ) of approximately 32.4° and 44.2° . RPF's crystallinity index (CI) was estimated to be 57.3% using Equation (2). This value of the crystallinity index indicates that RPF is semi-crystalline. The fibre has a mixed phase containing crystalline and amorphous regions with a higher percentage of the crystalline phase. A semi-crystalline material can have variable mechanical properties depending on the relative proportions of crystalline and amorphous regions. The crystallinity index of RPF in this study is less than the CI value of 66.6% reported by Fadele et al. [3] from a study of raffia palm fibre grown in Southern Nigeria. Also, less than a CI value of 64% was reported by Elenga et al. [4] for *Raffia textilis* fibre. This confirms the observed differences in the results of the tensile properties of the RPF fibre in this study compared with the RPF obtained in other regions. However, the CI value of the raffia palm fibre in this study is higher than the CI value of 45.8% obtained from kapok (cotton) fibre by Mwaikambo et al., [18]. A significant crystalline phase in the RPF can provide some degree of mechanical strength and stiffness. In contrast, the amorphous phase can provide flexibility, ductility, durability, biodegradability, biocompatibility, and toughness, making the fibre suitable for reinforcement in polymer composite for prosthetic applications.

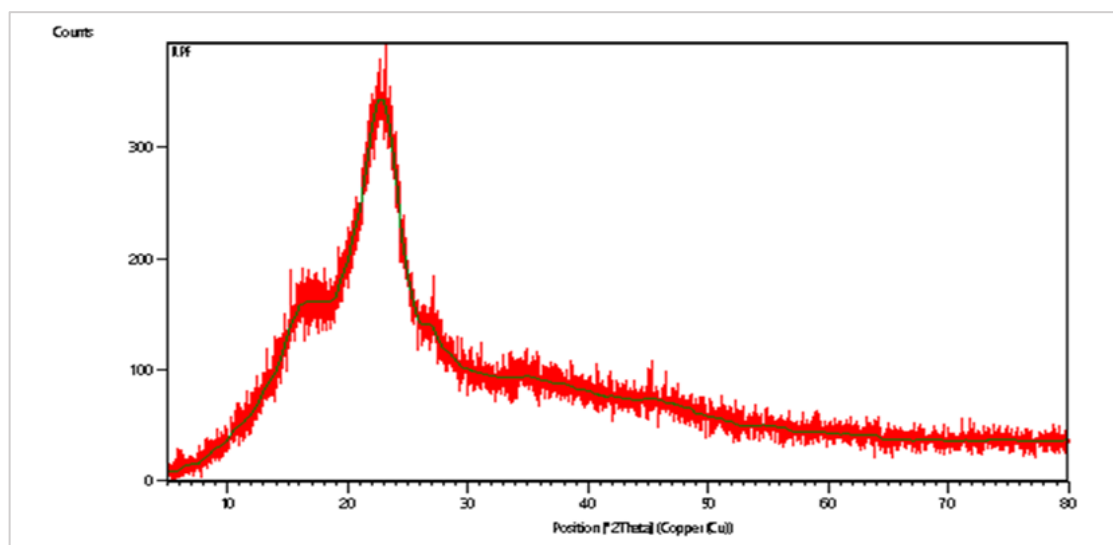


Figure 4: X-ray Diffraction Spectral of Raffia Palm Fibres

4. Conclusion

Raffia palm fibres are abundant in Nigeria and are used for various purposes, such as robes for weaving baskets in agriculture and construction. However, raffia palm fibres for reinforcement in polymer composites have not been promoted in Nigeria. The presence of beneficial metals Ca, Fe, and K in significant RPF concentrations suggests the fiber's safety. The good tensile properties obtained from this study indicate that raffia palm fibre can be further developed as an alternative material in the manufacturing industry as a substitute for fiberglass-reinforced polymer composites in some applications requiring moderate stiffness and strength. The absence of aromatic compounds in alkaline-treated raffia palm fibre confirms the fibre's purity. Aromatic compounds could be toxic, allergenic, or irritating, and their absence can make a material safe for use in products or materials where the absence of aromatic compounds is critical, such as food products, pharmaceuticals, or health care. This is particularly relevant for products that come into contact with the human skin, especially products used by vulnerable populations, as in the production of biomedical devices such as prosthetics. The presence of a significant crystalline phase in the RPF, as revealed by the XRD result, shows that the fibre is more crystalline than amorphous, as confirmed by the good tensile strength obtained from the fibre.

Based on the results of the spectroscopy in this study, it can be concluded that raffia palm fibre grown in Benue state, Nigeria, is a semi-crystalline fibre, has significant concentrations of beneficial elements and no toxic elements of concern when used as reinforcements in polymer composites in the development of products external to the body for human use. This suggests RPF is safe for deployment as a potential source of reinforcement in polymer composites, especially for developing products for biomedical applications such as prostheses and wheelchairs.

Author contributions

Conceptualization, I. Daniel and T. Emmanuel; methodology, N. Bem; software, I. Daniel; validation, G. Terfa, N. Bem and I. Daniel; formal analysis, T. Emmanuel; investigation, T. Emmanuel; resources, T. Emmanuel; data curation, I. Daniel; writing—original draft preparation, T. Emmanuel; writing—review and editing, N. Bem and I. Daniel; visualization, I. Daniel; supervision, N. Bem; project administration, T. Emmanuel. All authors have read and agreed to the published version of the manuscript.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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