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Characterization of Alkaline Treated Groundnut Shell Particulate for Reinforcement in Polymer Composite for Production of Prosthetic Sockets

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ABSTRACT
In this study, alkaline treated groundnut shell (GNS)was characterized using spectroscopy to evaluate its potential as reinforcement in polymer composites for prosthetic applications. The groundnut shell was crushed and sieved to a particle size of 300 µm. The GNSwas subjected to Fourier transform infrared (FTIR), electron dispersion X-ray fluorescence (EDXRF), and X-ray diffraction (XRD). FTIR spectra of treated GNS revealed that, the fibres are characterized with O-H stretching, C-H stretching, there is the presence of carboxylic acids, functional groups of methyl (CH_3), methylene (CH_2), and aliphatic saturated (CH) compounds. There was no observed peak at around 1000 cm^{-1} to 650 cm^{-1} bands characterized as C-H "oop" bond structure of functional group of aromatics. The absence of a peak in this range explains the disappearance of smell from the sample. Aromatic compounds may be impurities or contaminants, and their absence indicate that, the alkaline treatment has been effective.The EDXRF showed high concentration of Ca, K, and Bi. The XRD confirmed that, the alkaline treated GNS particulate is more amorphous than crystalline with the crystalline index (CI) of 0.34.These results suggest GNS is safe and suitable for deployment as a potential source of reinforcement in polymer composites especially, for the development of products for prosthetic applications.
Key Words: Groundnut shells, Particulate, biocompatible, amorphous, biodegradable

I. INTRODUCTION

As a result of environmental awareness and the concern for environmental sustainability, plant fibres such as jute, raffia palm, cotton, sisal, hemp, kenaf and bamboo have been used in the manufacturing industry for the production of composites for applications in many industrial sectors, such as textiles, automobiles, packaging, construction, sports equipment and medicine (Girijappa et al., 2019 and Sanjay et al., 2016). Natural fibre reinforced polymer composites are extensively used in prostheses such as upper and lower limb prostheses (Chandramohan and Marimuthu 2011). The reinforcement of polymer composites with plant fibres for prosthetic applications has attracted attention of researchers' due to health issues associated with the use of synthetic fibres such as fibre glass in the production of prosthetic devices. According to Ramakrishna et al., (2001), the development of polymer composites from plant fibres is encouraged due to the flexibility and the possibility to obtain composites having a wide range of mechanical and biological properties.

Over the years, the use of glass fibre in polymer composites for prosthetic applications has been discouraged due to high cost and health concerns; the use of glass fibres causes skin allergies when it comes in direct contact with the skin and allergic bronchitis leading to lungs cancer. Plant fibres are available at low cost and cause less health and environmental hazards for both users and workers producing the composites as compared to glass fibre composites (Jawaid and Abdulkhalil 2011).

According to Cheung et al., (2009), Materials for prosthetic applications should have sufficient strength, light weight, resistance to thermal conditions, durability and be biocompatible to human tissues; it should not cause allergic reactions to the body. This is anapplication where groundnut shells can be deployed;groundnut shells like other plant fibres have unique advantages over other materials such as; low cost, low density, high strength and stiffness to weight ratio, non-corrosive, processing flexibility, renewable, biodegradable, sustainable, non-toxicity and abundantly available (Chaudhary et al., 2018).

Groundnut botanically known as Arachis hypogeae belongs to Leguminosae family. Nigeria is one of the major producers of groundnut in the world after China and India. Groundnut shell is found in large quantities as agricultural farm wastes in Benue state and in some northern parts of Nigeria such as Sokoto, Kebbi, Zaria, Borno and Yobe States (Alabadan et al., 2005). Over the years, groundnut shell has constituted common solid waste especially in Nigeria. The utilization of Groundnut shell as reinforcement in polymer composite will promote waste management, reduce pollution therefore, enabling environmental sustainability, and increase the economic base of the farmer when groundnut shells are sold thereby encouraging more production.

The groundnut shell as reinforcing material is an agricultural product; eco-friendly, non-toxic, low cost, light weight and an easily available material as compared to conventional fibres like glass, kevlar, asbestos etc. Groundnut shells like other natural fibres is composed of cellulose, hemicellulose and lignin. Cellulose is very beneficial; plant cellulose has been used in clinical application in wound-healing research as a factor which stimulates granulation tissues in the wound (Morgan and Nigam 2013). Several options have been explored in researches for groundnut shell valorization including its use in the synthesis of nanocarbon (Berthet et al., 2015) bio-oil through pyrolysis, adsorbents, cellulose nanocrystals and solid fuel (Bano and Negi 2017) and (Lubwama and Yiga 2017).

In the area of composites, several studies have investigated the possibility of using groundnut shell as filler in various forms. Usman et al., (2016) used groundnut shells (both alkali-treated and untreated) as filler in a recycled polyethylene matrix. Kanayo et al., (2018) investigated the properties of a hybrid composite of groundnut shell ash and silicon carbide in an aluminium matrix. Nyior et al.,(2018) studied the mechanical properties of epoxy hybrid composite of raffia palm fibres and groundnut shells for automobile applications. Sesugh et al.,(2019) studied the dynamic mechanical properties of raffia palm fibres and groundnut shell particulate epoxy hybrid composites for interior panels in automobiles.

Though the results from these studies have been promising, there has not been a characterization of groundnut shell cultivated in Benue state Nigeria to establish its properties as potential reinforcements in polymers. This study has bridged this research gap by characterizing alkaline treated groundnut shell particulates obtained from Benue state Nigeria using Fourier transform infra-red (FTIR) spectroscopy, electron dispersion X-ray fluorescence (EDXRF) spectroscopy and X-ray diffraction (XRD) spectroscopy, to explore the possibility of using the groundnut shell particulate as a filler in polymer composites. According to Verma et al., (2013), particles enhance the stiffness of composites. Also, Particle fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, improve performance at elevated temperatures, reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage. Donald et al.,(2010), reported that, particles are used to increase the modulus of the matrix and to decrease the ductility of the matrix in composites.

II. METHODS

2.1 Preparation of groundnut shell particulate

The groundnut shells were collected from a groundnut processing centre in Ushongo Local Government area of Benue state. Cleaned and dried groundnut shells were initially washed with distilled water to remove sand and other impurities. Plate 1 shows clean groundnut shells while plate 2 shows the treated groundnut shell particulate.The groundnut shell was ground using a grinding machine and sieved using standard test sieves to a particulate size of 300 µm in the materials science laboratory Joseph Sarwuan Tarka University Makurdi, Nigeria.One gram of GSP was added to 10 ml of 10% sodium hydroxide solution for 2 hrs with continuous stirring, using stirrer at room temperature. Thereafter, the GSP was rinsed with distilled water until the solution turns neutral. The powder was filtered and then sun dried to obtain treated groundnut shell particulate.

Plate 1: Groundnut Shells

Plate 2: Alkaline Treated Groundnut Particulate

2.2 Characterization of groundnut shell particulate

A. Fourier Transform Infra-red (FTIR)

The alkaline treated groundnut shells were crushed to a particulate size of 300 µm. The sample was kept in a container at room temperature. The FTIR spectroscopy was carried out at the Umaru Musa Yar-adua University (UMYU) central laboratory Katsina State Nigeria using Agilent FTIR machine. The test was conducted with a sample scan of 64, a resolution of 16 and spectral range of 4000-650 cm^{-1}.

B. Energy Dispersive X-Ray Fluorescence (EDXRF)

An energy dispersive XRF spectroscopy machine with a tube voltage of 20 kV, tube current of 0.70 mA and a live time limits of 120 seconds was employed as a primary source to determine elemental concentrations in groundnut shell sample. This was done at the Umaru Musa Yar-adua University (UMYU) central laboratory Katsina State Nigeria.The groundnut shells were crushed to a particulate of size 300 µm. The powdered sample was placed into a plastic sample cup and manually pressed to ensure a reasonable flat sample surface and presented to the XRF spectrometer system. The spectra obtained as a result of x-ray excitation was transferred to a computer. The concentration of the elements present in the samples was obtained using AXIL-XRF software.

C. X-ray Diffraction (XRD)

The alkaline treated groundnut shells were crushed to particle size of 300 µm. 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-adua University (UMYU) central laboratory 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, X-ray characteristic of K-Alpha.The crystalline index was determined using the established Segalet al., (1959) empirical equationas shown in equation 1.

$$C.I = \frac{I_{200} - I_{AM}}{I_{AM}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

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.

III. RESULTS AND DISCUSSION

A. Results of FTIR spectroscopy on Groundnut shells (GNS) Particles

The FTIR spectroscopy graph of transmittance versus wave number for GNS is shown in Figure 1. The FTIR spectra of alkaline treated GNS particulate shows a peak at 3280.05730 cm^{-1}. The region of the broad absorption band at 3500 cm^{-1} to 3200 cm^{-1} for natural fibres are characterized with O-H stretching and H-bonded bond structure that mostly contains major functional groups of phenols, alcohols and waters. It observed that, the O-H stretching and H-bonded broad absorption band in the region in the GNS spectroscopy showed a low peak which could be attributed to alkaline treatment of the samples leading to a decrease of these functional groups in the fibre. Alcohols found in this fibre could be beneficial because; they are often used in products

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due to their antimicrobial properties and also, ability to reduce greasiness. According to Ramadevi et al., (2012), the O-H stretching, H-bonded and free hydroxyl found in natural fibres is due to the presence of 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 as well as absorption in biological systems. These can play a role in material design and engineering for specific applications such as in the deployment of GNS in biomedical applications. The results obtained from this study is in agreement with the results reported in Dwivedi and Mehta (2011).

An additional peak observed at 2914.77819 cm^{-1} in GNS 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 decrease in functional group of phenolic or aliphatic hydroxyl in the fibres due to reaction with sodium hydroxide that promotes free hydroxyl that caused the addition of extra peak in free hydroxyl bond structure at 2914.77819 cm^{-1} in the GNS. The presence of carboxylic acids in natural fibres influence biodegradability, hydrophilicity and adhesion.

According to Khalil et al., (2012), 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 present in GNS can impact material properties such as mechanical strength, flexibility and durability. The small peak at 1602.75527 cm^{-1} for GNS is characterized as the C=C stretching bond structure from the functional group of alkenes (lignin), the peak at 1423.84305 cm^{-1}, 1319.47759 cm^{-1} and 1259.84019 cm^{-1} for GNS is characterized as the C-H bending bond from the functional group of alkanes (cellulose, hemicellulose and lignin) and the peak at 1028.74524 cm^{-1} for GNS are characterized as the C-O stretching bond structure from the functional group of alcohol (cellulose, hemi-cellulose and lignin), carboxylic acids, esters and ethers. According to Cao and Tan (2004) and Hinterstoisser et al., (2001), the peak band located in the range of 1100 cm^{-1} to 1000 cm^{-1} are also characterized as the C-O stretching bond structure of the functional group of glycosides linkage. Glycosides are molecules that consist of a sugar molecule 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., (2013).

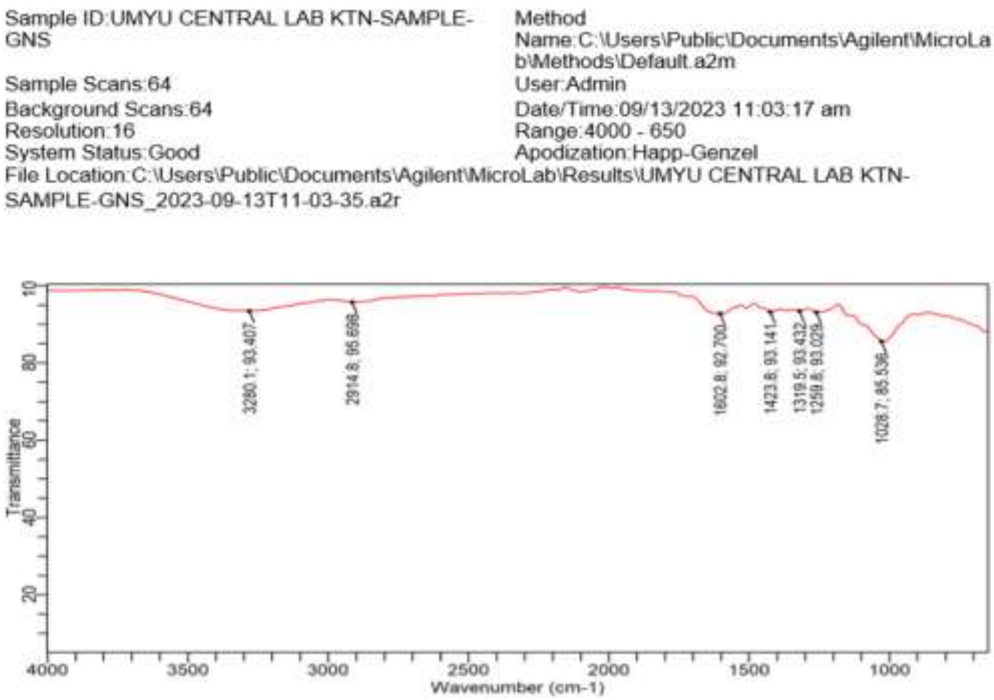

Figure 1: Groundnut shells FTIR Spectral

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Furthermore, there was no peak of an absorption observed at around 1000 cm^{-1} to 650 cm^{-1} in GNS spectroscopy. This bands characterized as C-H "oop" bond structure of functional group of aromatics. This is an indication of the absence or low concentration of the aromatic functional group or molecular vibrations associated with the sample due to alkaline treatment. The absence of a peak in this range accounts for the disappearance of smell from the groundnut shells. Aromatic compounds could also, 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.

B. Energy Dispersive X-ray Fluorescence on Groundnut Shells (GNS)

The elemental composition of groundnut shell (GNS) is revealed by the EDXRF as shown in Table 1 and the graph of the fluorescence is shown in Figure 2. The result showed that, major elements present in GNS in significant concentrations are; calcium (Ca) 0.9047 %, Bismuth (Bi) 0.392 %, and potassium (K) 0.21218 %.Trace amount of aluminium (Al), sulphur (S), chlorine (Cl), and manganese (Mn) were detected in small concentrations in the GNS sample. Iron, calcium, potassium and zinc are beneficial elements and are found in the sample in good concentrations.

Table 1: Electron Dispersion X-ray Fluorescence (EDXRF) of Alkaline Treated GNS Particles

Element	Concentration (%)	Peak(cps/mA)	Background(cps/mA)
Fe	0.07762	1317	-52
Cu	0.07858	4348	-111
Ni	0.00885	16	14
Zn	0.02446	1115	-43
Al	0.02673	558	1595
Mg	0.02931	184	172
Na	0.0642	37	20
S	0.10806	1612	1334
P	0.05866	582	702
Ca	0.9047	5052	-233
K	0.21218	1173	-65
Mn	0.010215	572	178
Rb	0.000686	3	2
Sr	0.002605	19	1
Br	0.00022	0	2
Cl	0.02938	54	6
Cr	0	0	111
V	0	20	92
W	0.1204	75	-6
Bi	0.392	4	1
Sn	0.14	1	52
Si	0	0	21
As	0	1	3
Nb	0.0141	1	2
Ta	0.0181	26	2615
Ag	0	0	1299
Pb	0.070	2	2

Typical toxic elements such as; Arsenic (As), Cadmium (Cd), Mercury (Hg), Chromium (Cr), Vanadium (V), Cobalt (Co), Tantalum (Ta) and Ag were absent in the GNS grown in Benue state.

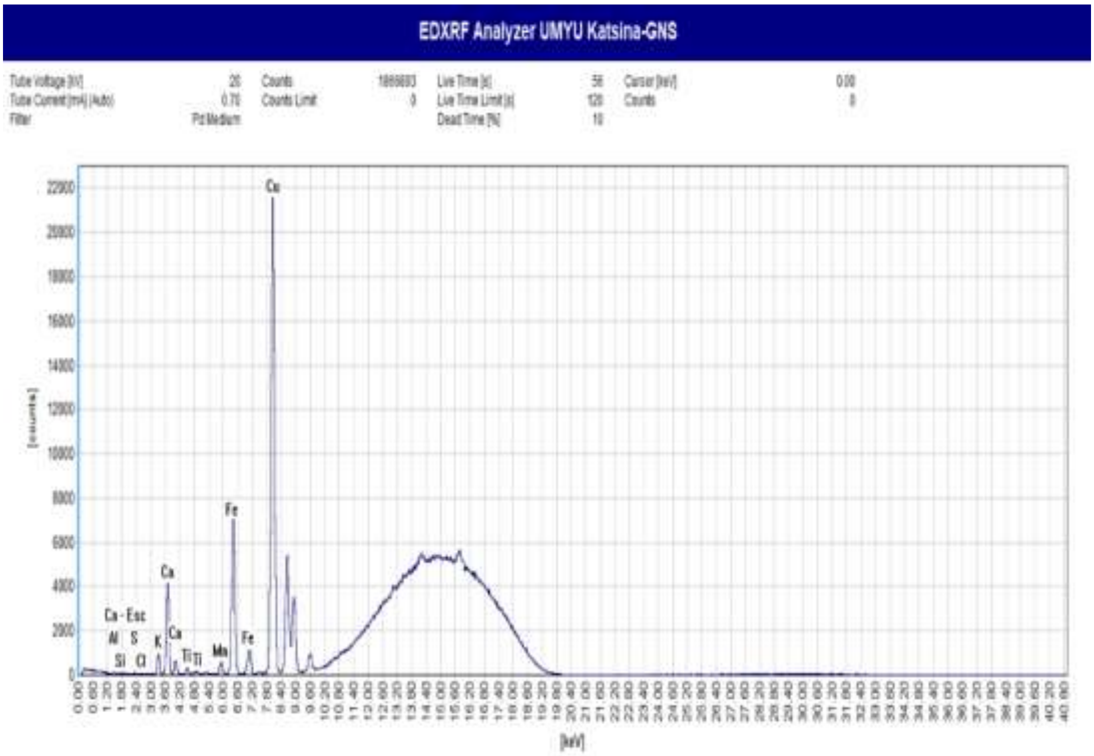

Figure 2: Groundnut shell EDXRFSpectral

C. X-ray Diffraction (XRD) Spectroscopy

The x-ray diffraction of the alkaline treated groundnut shell particulate is presented in Figure 3. The intensity of the diffraction band was used to establish the crystalline and the amorphous phase of the GSP. Using Eq.,1 the crystallinity index of the groundnut shell particulate is 0.34 (34 %). Crystallinity index is a measure of the amount of crystalline material present in a sample. It is typically expressed as a value between 0 and 1, where a value of 0 indicates a completely amorphous material and a value of 1 indicates a completely crystalline material. A crystallinity of 0.34 indicates that, the sample is predominantly amorphous, with some crystalline regions present.According to Jayamani et al., (2015), the alkaline treatment causes the removal of hydrogen bonding in the fibre network thus, promote the formation of the amorphous cellulose at the expense of the crystalline cellulose. Amorphous materials are soft and flexible, potentially useful in wide range of applications from electronics, automotive andmedicine.

The result obtained in this study, agrees with the study by Usman et al. (2021); in which,untreated groundnut shell powder (UGSP), treated groundnut shell powder (TGSP), as well as ash groundnut shell powder (AGSP) was subjected to XRD. The result was that, UGSP showed higher crystallinity than the TGSP while the AGSP displayed the highest crystallinity, this was explained that, the alkaline treatment removed hydrogen bonding in the fibre thereby increasing the formation of the amorphous cellulose.The study concluded that, sodium hydroxide treated groundnut shell powder will be a suitable reinforcement material in the development of biodegradable polymer composites while the ash GSP is the best candidate material for the reinforcement of polymer composites for structural applications, although ash-reinforced polymer composite might not be biodegradable due to low or absence of amorphous cellulose. According to van Kuijk et al., (2015), cellulose consist of two components (crystalline and amorphous) and the chemical treatment is effective in changing the chemistry of the cellulose. Natural fibres with high crystallinity according to Mokhtari et al., (2015), have shown to exhibit high tensile strength as the extraction of the non-cellulosic substance (hemicellulose and lignin) enhance the tensile strength of the natural-lignocellulosic fibres. The results of this study also, confirmed the results ofde Oliveira et al., (2017),who conducted a study and reported a lower crystallinity index for treated sisal fibre compared with untreated sisal fibre. This was attributed to an infiltration of the species

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(Na^+/H2O) into the crystalline domain causing decreased crystallinity of treated fibre.

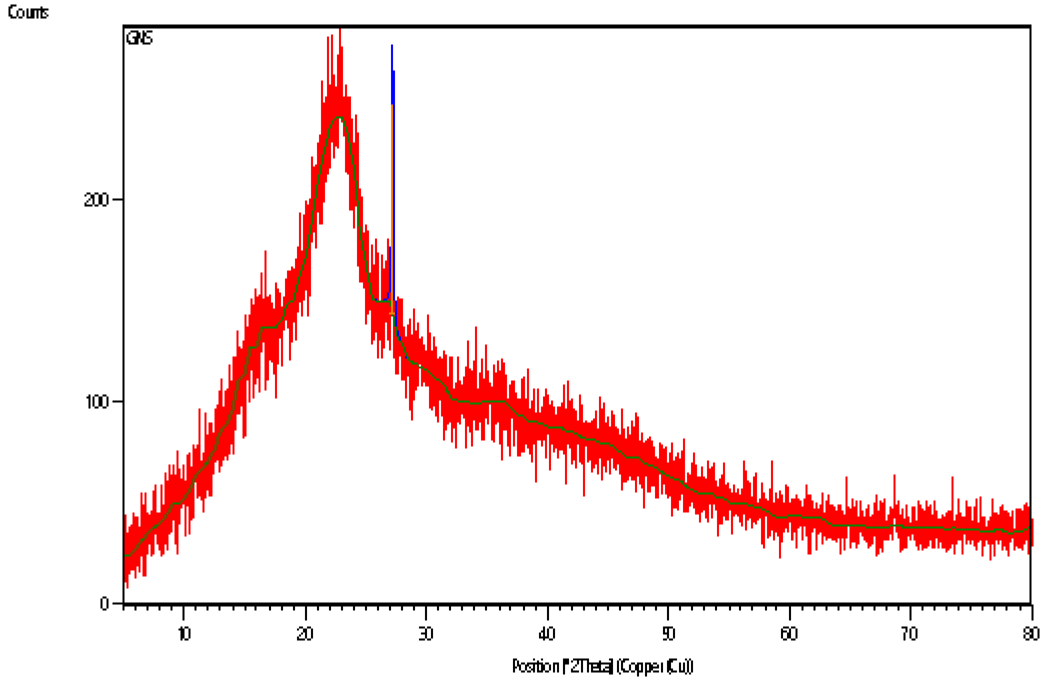

Figure 3: Groundnut shell XRD

IV. CONCLUSION

Groundnut shells which is an agricultural bye product is cheap and in abundance in Nigeria, it is considered as agricultural waste by farmers. However, the FTIR spectroscopy results of GNS shows beneficial functional groups in groundnut shell particulate. The Free hydroxyl groups found in GNS can impact the physical and chemical properties of materials; they can increase the polarity and hydrophilicity of a compound, adhesion and biocompatibility as well as absorption in biological systems. These can play a role in material design especially using GNS as reinforcement in polymer composites for biomedical applications. Also, the presence of carboxylic acids in GNS influence biodegradability, hydrophilicity and adhesion. Methyl groups present in GNS can impact material properties such as mechanical strength, flexibility and durability which are requirements of materials for prosthetics applications. The absence of aromatic compounds in the alkaline treated GNS as confirmed by the FTIR suggest the samples purity. Aromatic compounds could be impurities or contaminants, and their absence can suggest that, the sample is of high purity or has been effectively purified. This is an indication that, NaOH treatment on the GNS is effective and actually enhances the purity of the GNS. This is particularly relevant for products that come into contact with the human body.

The EDXRF shows that, the major elements present in GNS in significant amounts are;calcium (Ca), bismuth (Bi), and potassium (K). Beneficial elements such iron (Fe), calcium (Ca) and potassium (K) are in high concentrations in the shells. Calcium is an essential macro nutrient that provides structural support to 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 considered to be important constituent for the human body.

The XRD confirmed that, the alkaline treated GNS particulate contain more amorphous phase than crystalline phase. The amorphous cellulose which was increased in the treated GSP favours the degradation of the fibre by enzymes and this makes it the right candidate filler material in the development of biodegradable natural fibre polymer composites.

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Based on the results of the spectroscopy in this study, it can be concluded that, groundnut shells grown in Benue state when treated with NaOH is biocompatible, biodegradable, amorphous, good strength, flexible and durable as well as possess good quantity of beneficial elements and no toxic elements of concern. The soft and flexible nature of amorphous materials may reduce the incidence of skin irritation and breakdown, which is a common problem with the traditional prosthetic sockets made from rigid synthetic fibres materials. These results suggest GNS is safe and suitable for deployment as a potential source of reinforcement in polymer composites especially, for the development of products for prosthetic applications.

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