

## Determination of Physiochemical Properties of Dikanut Shell

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### Abstract

This study was aimed at determining the physiochemical properties of Dikanut shell in two distinct forms, viz., unmodified Dikanut shell powder (UDSP) and carbonized Dikanut shell powder (CDSP). The Dikanut properties investigated in this study were density, pH value, moisture content, oil absorption capacity, ash content, metal oxides and functional group using the fourier transform infrared spectroscopy (FTIR) analysis. The results from this study showed that the Dikanut shell in the UDSP form contained a pH value of 4.8, bulk density of 0.49g/cm<sup>3</sup>, moisture content of 9.4, oil absorption capacity of 0.52g/g and ash content of 3.4%. The study further showed the presence of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, Fe<sub>2</sub>O<sub>3</sub>, aromatic hydrocarbons, and ring based compounds in the UDSP sample. The CDSP had a pH of 7.5, bulk density of 0.69g/cm<sup>3</sup>, moisture content of 5.5, oil absorption capacity of 0.54g/g, ash content of 6.5%. The FTIR analysis of Dikanut showed the presence of functional groups such as esters, amides, carboxylic acids and aromatic hydrocarbons.

## 1. Introduction

Everyday around the world, researchers are focusing on ways of utilizing agro-wastes as a source of raw materials for engineering new products for end users. Through history, materials have been used in the development of civilization. Thus, it has become important and necessary to seek new materials and ways of modifying existing materials to fit or solve challenges posed in the engineering world. Different engineering applications have become possible due to research into alternative materials and more suitable properties which in turn affects the economy of any given nation. It is true that the knowledge of any country about the potentials of its capabilities can be reflected in its development.

Many researches are currently ongoing in identifying new materials, examining these materials for inherent properties and the possible application in engineering fields. Before now, agricultural products which sometimes are treated as wastes have been studied and harnessed for engineering application. For example, palm kernel shell, coconut shell, periwinkle shell, etc have been characterized and used to reinforce concrete, metals, and purify waste water [1, 2]. The Dikanut shell is another agricultural by-product which by physical observation is light and tough and dictates usefulness in engineering application. In Tropical Africa, Dika tree is popularly grown and referred to as the bush mango. In Nigeria, about 150,000 tons of Dika is produced annually; almost all the Dika produced are in the south-east region of the country [3]. The Dikanut from the African bush

mango consist of a kernel embedded in a shell. Though the kernel has found much usefulness such as the oil that can be extracted from the kernel which finds application in soap making, cosmetics etc., there still exist much work and research to be done in analyzing the shell. The Dikanut shell can be obtained in large quantities at little or no cost as it rarely has a commercial value yet in Nigeria [3]. The lack of harnessing some of the agricultural by-products have led to low development of local product which in many areas can be used in composite particularly for low cost/high volume applications.

Ekebafé et al. [3] investigated the physico-mechanical and equilibrium swelling properties of natural rubber components filled with Dikanut shell pericarp powders. It was again established that Dikanut shell carbon (DNSC) exhibits better vulcanizing properties than the raw (uncarbonized) Dikanut shell (DNS) and concluded that the reinforcing potential of DNSC was comparable to that of carbon black (CB) in hardness and compression set properties and predicts potential applications of DNSC as low cost value added filler in natural rubber products and as a market substitute for CB.

Other methods to determine the materials physical and chemical properties using standard methods will show or create findings of its useful application in light weight construction as material filler and reinforcements.

## 2. Methodology

The materials used in the study include the Dikanut Shell (Figure 1), Muffle furnace, Digital Electronic Scale, Atomic Absorption Spectroscopy (AAS) Instrument, FTIR-8400S Fourier Transform Infrared Spectrophotometer, pocket pH meter and conductivity meter. The Dikanut shell was sourced from Agenebode in Edo State.



**Figure 1: The Dikanut Shell in Unprocessed State**

### 2.1 Preparation of the Dikanut Shell Powder Samples

After collection, the Dikanut shells were washed thoroughly using detergent and rinsed with distilled water, and spread in sun to dry for twenty hours during the dry season (i.e. early December). A portion of the dry shells were pulverized into powdery form. It was then sieved. The fine particles that passed through the mesh were collected as the unmodified Dikanut shell powder (UDSP) as shown in Figure 2. Some portions of the dry shells were taken to the muffle furnace for carbonization at 600°C for three hours [4]. The carbonized shells were pulverized into powdery form. The carbonized shell powder (CDSP) was sieved with a mesh size of 150 $\mu$ m. Figure 3 shows the CDSP sample.



**Figure 2: UDSP Sample**



**Figure 3: CDSP Sample**

Standard chemical procedure was used in determining the pH level, electrical conductivity, bulk density, oil absorption capacity, ash content of the UDSP and CDSP samples.

## **2.2 Determination of the pH Value and Electrical Conductivity of the Samples**

The pH values of the UDSP and CDSP samples were determined by putting 1g of each samples into 250 ml beaker 100ml of boiled out distilled water [5] and stirring the mixture for 20minutes. Thereafter, the electrode of the pocket pH meter which was initially calibrated with standard buffer 4 to 7 was introduced into the mixture and the digital value on the pH meter read-out was recorded. Just like in the determination of pH of the samples with a pH meter, the electrical conductivity of the samples was measured using a conductivity meter immersed into the mixtures.

## **2.3 Determination of the Bulk Density**

The bulk density of UDSP and CDSP samples were obtained by the tapping procedure [5]. The samples were put in a dried measuring cylinder and the cylinder was then carefully tapped on the bench for the powdered particles to fill the empty spaces. The volume obtained when continuous tapping was observed to yield no reduction in volume was taken and recorded. The bulk density was then calculated using the relation;

$$\text{Bulk Density}(B. D) = \frac{\text{weight of sample (g)}}{\text{volume of sample (cm}^3\text{)}} \quad (1)$$

This experiment was repeated three times and the mean bulk density was taken.

## **2.4 Determination of Oil Absorption Capacity (OAC) and Water Absorption Capacity (WAC) of the Samples**

In determining the OAC of the samples, 0.5g of the UDSP and CDSP samples were mixed with 10ml soya vegetable oil. The mixture was centrifuged at 3500rpm for 30 minutes [6]. The excess oil was decanted into a 10cm<sup>3</sup> graduated measuring cylinders and the volumes noted. The absorbed oil volume for the two samples were determined and converted to grams. The WAC of the samples

was determined by mixing 1g of the samples with  $10\text{cm}^3$  of distilled water and centrifuged for 30 minutes at 3500rpm [6]. The supernatant was decanted into a  $10\text{cm}^3$  graduated measuring cylinder. The volume noted was used to determine the volume of water absorbed.

## 2.5 Determination of Metal Oxides

The wet oxidation method was applied in the determination of the metal oxides. This was done by weighing 5g of the samples into macro kjeldahl digestion flasks with 20ml distilled water and concentrated Nitric acid added. The mixture was boiled until the volume was reduced to about 20ml. Then 10ml concentrated Sulphuric acid was added and the boiling continued with further addition of little quantities of Nitric acid immediately the liquid begins to blacken. The heating continued until dense white fumes evolved. The mixture was then cooled and 10ml of saturated ammonium oxalate solution was added until dense white fumes were again evolved. This was to facilitate the removal of coloured nitrous compounds. The digest was transferred into a 100ml volumetric flask and made work with distilled water. Blank was also prepared the same way. Atomic Absorption Spectrophotometer (AAS) was used in the determination of the trace metals in the digested sample including aluminium and silicon [7]. The metal oxides were determined using percentage conversion following normal stoichiometry.

## 2.6 Determination of the Functional Group by FTIR spectroscopy Analysis

The FTIR- 84005 fourier transform infrared spectrophotometer (SHIMADZU) was used to determine the surface functional groups present in the UDSP and CDSP samples. The analysis was done using spectrometer and detector, which is capable of measuring functional group to the predetermined minimum detectable level and spectral collection using FTIR system  $1\text{cm}^{-1}$  resolution, 22 meter path length, and a broad band MCT detector [8].

## 3. Results and Discussion

Table 1 shows the results obtained from the physical and chemical characterization of the UDSP and CDSP samples.

**Table 1: UDSP and CDSP Samples Characterization Results**

Investigated Parameters	UDSP	CDSP
pH	4.8	7.5
Bulk Density ( $\text{g}/\text{cm}^3$ )	0.490	0.690
Electrical conductivity (NS/CM)	14570	1230
Moisture content (%)	9.4	5.5
WAC (g/g)	2.4	1.4
OAC (g/g)	0.52	0.54
Ash content (%)	3.4	6.5
Pb(mg/kg)	0.00	0.00

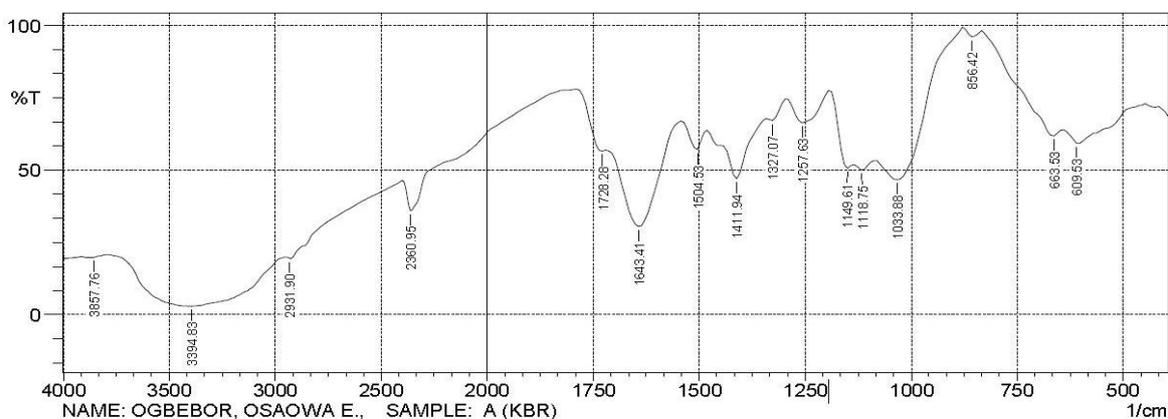
Cu(mg/kg)	0.00	0.30
Zn(mg/kg)	0.43	0.64
Cd(mg/kg)	0.00	0.00
Fe(mg/kg)	480	550
Ni(mg/kg)	100	110
Na(mg/kg)	580	850
K(mg/kg)	64200	60700
Mg(mg/kg)	2050	1300
Ca(mg/kg)	13800	13600
Al(mg/kg)	305	270
Si(mg/kg)	750	650

From Table 1, it is seen that the bulk density of the Dikanut shell is  $0.490\text{g/m}^3$  for UDSP and  $0.690\text{g/m}^3$  for CDSP, and this is within the range of values for other similar lightweight materials such as fly ash, bagasse, walnut shell, coconut shell, palm kernel shell, almond shell, as reported by other researchers [9, 10]. The pH content for the UDSP sample was tested to be acidic with a pH value of 4.8, while that of CDSP sample was alkaline with a pH value of 7.5. The moisture content obtained for the UDSP sample was 9.4% while it was 5.4% for the CDSP sample. These values of the moisture contents are in good range as compared to other agro waste product like walnut shell with moisture content of 4.18% and almond shell with moisture content of 7.21% [11, 12]. Table 2 shows the results obtained from the mineralogical analysis and chemical composition of the UDSP and CDSP samples using the atomic absorption spectrophotometer (AAS). The elements composition obtained from this process was converted to oxides by conversion factors from chemistry stoichiometry [13], and then the percentage (%) by weight was evaluated.

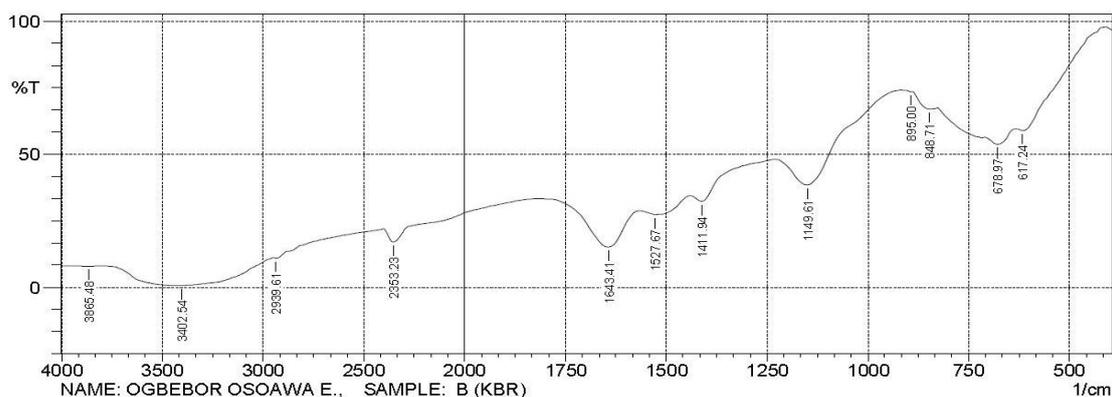
**Table 2: Oxide Compound obtained from Weight Based Analysis**

Oxide Compound	UDSP (Mg/kg)	% wt	CDSP (Mg/kg)	% wt
SiO <sub>2</sub>	1604.63	0.16	1390.68	0.14
Al <sub>2</sub> O <sub>3</sub>	576.24	0.06	510.11	0.05
Fe <sub>2</sub> O <sub>3</sub>	686.30	0.07	786.39	0.08
MgO	3399.11	0.34	2155.53	0.22
CaO	19308.96	1.93	19029.12	1.90
K <sub>2</sub> O	77328.90	7.73	73113.15	7.31

From Table 2, there is presence and trace of oxides such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, CaO, K<sub>2</sub>O in both the UDSP sample and the CDSP samples though at different percentage by weight. The presence of these oxides plays a vital role in the application of the Dikanut shell as particulate reinforcement material in other matrixes and as a good filler material. Figures 4 and 5 show the results of the FTIR spectroscopy analysis of the UDSP and CDSP samples respectively.



**Figure 4: FTIR spectrum of UDSP Sample**



**Figure 5: FTIR spectrum of CDSP Sample**

Tables 3 and 4 show the summary of the FTIR Spectroscopy result for the UDSP and CDSP samples respectively, as obtained in this study.

**Table 3: Summary of the FTIR Spectroscopy Results for UDSP Sample**

Spectral Frequency Range (cm <sup>-1</sup> )	Peak Values in UDSP Sample (cm <sup>-1</sup> )	Bonds and Compounds possibly Indicated
3500-3000	3394.83	O-H stretch (Alcohol and hydroxyl compounds group)

2500-2000	2360.95	C≡C stretch (Alkynes)
1750-1500	1728.28	C=O stretch (Carbonyl functional group, esters, ketones, aldehydes)
1750-1500	1643.41	C=C stretch, N-H bend (Amides, Amines, Alkenes)
1750-1500	1504.53	C=C stretch (Alkenes)
1500-1250	1411.94	C-H bend (Alkyl groups)
1500-1250	1257.63	C-O stretch (Carboxylic acid salts, Aromatic ethers)
1250-1000	1033.88	C-O stretch (Primary amines)
1000-750	856.42	C-H bend
750-500	663	Benzene

**Table 4: Summary of the FTIR Spectroscopy Results for CDSP Sample**

Spectral Frequency Range (cm <sup>-1</sup> )	Peak Values in CDSP Sample (cm <sup>-1</sup> )	Bonds and Compounds possibly Indicated
3500-3000	3402.54	O-H stretch (Alcohol and hydroxyl compounds group)
2500-2000	2353.23	C≡C stretch (Alkynes)
1750-1500	1643.41	C=C stretch, N-H bend (Amides, Amines, Alkenes)
1500-1250	1411.94	C-H bend (Alkyl groups)
1250-1000	1149.61	C-O stretch (Alcohol)
1000-750	848.71	C-H bend
750-500	678.97	Benzene

The FTIR analysis of the samples shown in Figures 4 and 5 indicated that 16 peaks were recorded for the UDSP sample while 12 peaks were recorded for the CDSP sample. It can also be seen that there is no much difference or variation between the bands of the UDSP spectrum and that of the CDSP spectrum. The intensity of the overall FTIR spectral features were reduced more in the CDSP sample as a result of carbonization.

The results of the FTIR spectroscopy analysis shown in Tables 4 and 5 of the two samples showed that the Dikanut shell contains functional groups such as hydroxyl group, esters, amines, carboxylic

acids and aromatics. These properties present in Dikanut shell make it appropriate for use as engineering material [14, 15]. Furthermore, the presence of aromatic bonds such as  $-C=C-$ ,  $-C\equiv C-$ , and  $-C-H-$  showed the polymeric nature of the Dikanut shell as raw material in fabrication of polymer matrix composites

#### 4. Conclusion

From the results obtained in this study, it is seen that the Dikanut shell which is usually treated as waste product actually possesses some properties that are useful for engineering discipline. The physiochemical properties of the Dikanut shell revealed pH value of 4.8, bulk density of  $0.49\text{g/cm}^3$ , moisture content of 9.4% for UDSP sample and pH value of 7.5, bulk density of  $0.69\text{g/cm}^3$ , moisture content of 5.5 for CDSP Sample. The shell also contained oxides of silicon, aluminium, calcium and iron. The FTIR spectroscopy analysis showed that the shell contained aromatic hydrocarbons, ring based compounds and carboxylic groups. The lightweight property Dikanut shell as revealed in this study, gives the shell significant consideration the selection of lightweight materials for engineering application.

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