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Characterization of Dikanut Shell using Scanning Electron Microscopy and X-Ray Diffractometry

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ABSTRACT

In Nigeria today, Dikanut shell is an agro waste that has the tendency of becoming an environmental pollutant in the near future. There is currently a dearth of literature with regard to Dikanut characterization with a view to its utilization in science and engineering fields. This paper is focused on the characterization of the surface morphology and crystalline structure of Dikanut shell using scanning electron microscopy (SEM) and X-ray Diffraction (XRD) respectively. Dikanut shell samples that were used in this study were ground into powdery form and sieved with a mesh to a size of 150 µm. The samples were in two forms - natural state termed unmodified Dikanut shell particles (UDSP) and carbonized state at (600 °C for three hours) termed carbonized Dikanut shell particle (CDSP). The result of the SEM analysis showed that the UDSP orientation was scattered with visible porous structures and appeared dense. Also, its irregular shaped organic particles formed isolated aggregate of an amorphous matrix. The SEM result for the CDSP showed more developed porous structure indicating high surface area with more compact and well distributed structure. The XRD analysis of UDSP showed the presence of amorphous structure of carbon in the UDSP with a peak of 16.64° and 21.95°. The XRD analysis of CDSP showed a peak of 19.88° and sepiolite phase (magnesium silicate hydroxide hydrate) which are hard phases. The presence of the hard phases as represented by the sepiolite indicates that the material is useful as material filler and composite reinforcement.

1. INTRODUCTION

The Dikanut shell is an agricultural by-product which by physical observation is light and tough and has some usefulness in engineering applications. These include, filtering of effluent, filler material in infrastructure, buildings and road-making, reinforcing of concrete, light weight-structural applications, etc. (Okpala, 1990; Okafor, 1998; Alengaram et al., 2008). In Tropical Africa, Dika tree is popularly referred to

as the bush mango. In Nigeria, about 150,000 tons of Dika is produced annually and almost all the Dika is produced in the south-east region of the country (Ekebafe et al., 2012). Dikanut consists 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 (Ekebafe et al., 2012).

Awono et al. (2009) noted that in developing countries, the problem of developing local products is compounded by the lack of knowledge on their potentials and undervaluation of their impact on national economy. In Nigeria and other African countries today, many researches are being done to identify new materials, examine them for inherent properties and applying in various engineering discipline (Anuar et al., 2017; Ebhojiaye et al., 2018). Before now, local product such as agricultural products which sometimes are treated as wastes have been studied and harnessed for engineering applications such as automobile disk brake pads, internal combustion engine block, particle board production, etc. (Dagwa and Ibhadode, 2008; Ebhojiaye et al., 2018; Abdullahi and Sara, 2015). Various agro waste products have been characterized and thus their usefulness seen in many engineering applications. Many of these products are in abundance all around us and enhancing their potentials increases its usability in the engineering world (Dagwa and Ibhadode, 2008; Ebhojiaye et al., 2018).

Other agricultural waste products such as coconut shell ash have been characterized for potential utilization in metal matrix composites for automotive application using SEM and XRD (Madakson et al., 2012). In the study, hard phase such as silicon oxide was found present and its particle morphology were observed to be solid in nature, but irregular in size. Nitin and Singh (2013) and Mohammed (2014) also characterized walnut shell using SEM and XRD analysis and reported its potential in light weight applications by improving its mechanical properties. Atuanya and Ibhadode (2011) characterized wood sawdust using XRD, FTIR and SEM analysis as a potential reinforcement in polymer matrix. This paper therefore, focuses on characterizing the Dikanut shell using SEM and XRD to ascertain its crystallinity and morphology.

2. MATERIALS AND METHODS

2.1. Materials

Dikanut shell, shown in Figure 1 was sourced from one of the agricultural farms in Edo State, Nigeria. The Dikanut shell samples were ground to powdery form and sieved with a mesh to a size of 150 µm. The samples were in two forms - natural state termed unmodified Dikanut shell particles (UDSP) as shown in Figure 2, and carbonized state at (600 ^{o}C for three hours), according to Tenebe et al. (2013), and termed carbonized Dikanut shell particle (CDSP) as in Figure 3. Other materials used in the study included the JOEL JSM-6400v (lanthanum hexaboride electron emitter) scanning electron microscope (SEM) equipped with an oxford INCA^{TM} Energy dispersive spectroscopy system and Philip X-ray Diffractometer (XRD) machine.

Figure 1: The Dikanut shell

Figure 2: The UDSP sample

Figure 3: The CDSP sample

2.2. Methods

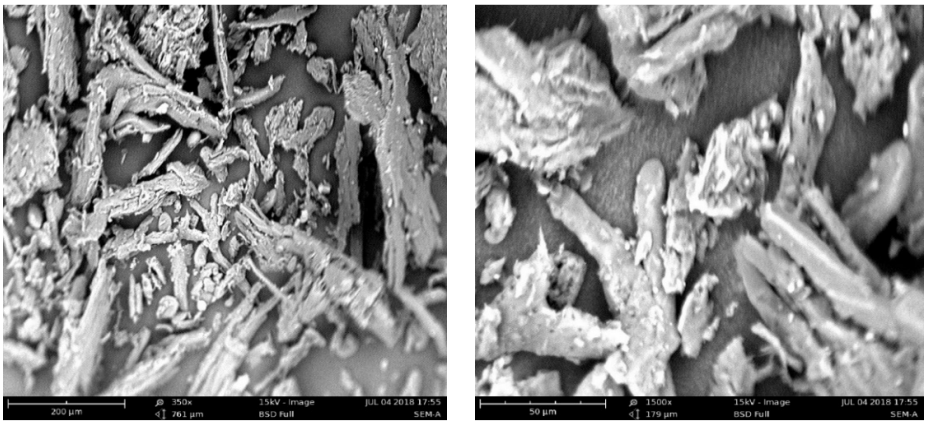
In carrying out this study, the prepared UDSP and CDSP samples were placed on sample holder and the images were captured under various magnifications. Prior to this, the prepared samples were applied with gold coating to avoid charge effect in order to obtain clear images. The SEM was operated at an accelerating voltage of 5 to 20 kV as suggested by Aigbodion et al. (2010).

The XRD analyses of the UDSP and CDSP were carried out using Philips X-ray diffractometer. The X-ray diffractograms were taken using Cu kα radiation at a scan speed of 3º/min. The samples were rotated at precisely one and half (i.e. 1.5) of the angular speed of the receiving slit, so that constant angle between the incident and reflected beams was maintained. The receiving slit was mounted in front of the counter on the counter tube arm, and behind it. A scatter slit was fixed to ensure that the counter received the radiation only from the portion of the specimen illuminated by the primary beam. The intensity diffracted at the various angles was recorded automatically on a chart and appropriate (θ) and (d) values were obtained (Rajan et al., 2007; Aigbodion, 2010).

3. RESULTS AND DISCUSSION

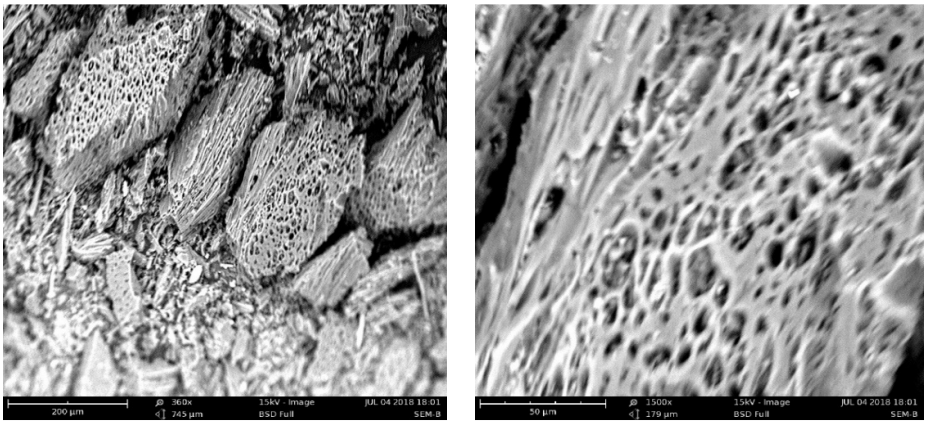
The SEM images as shown in Figures 4 and 5 show the particle morphology with respect to the different magnifications. Figure 4 showed the particle morphology for the UDSP and Figure 5 showed that of CDSP. From Figure 4, it was seen that the tendency of the particles to clump together is at lower magnification while variations in size and morphology became clearer at higher magnification. The light region corresponds to carbon wall and the black region corresponds to pores. The orientation was more or less scattered with minimum visible porous structures and appeared dense. Irregular shaped organic particles, heterogeneous mixture of different particle sizes and shapes were observed to form isolated aggregate or dispersed in an amorphous carbon matrix (Shakuntala, 2005).

The image of the CDSP samples in Figure 5 showed a more developed porous structure which indicated a high surface area (Ekebafe et al., 2012). The enlarged pores observed from the conversion of UDSP to CDSP by subjecting it to high temperature of 600 ºC, may have been due to the release of volatile matter from the raw particles (Okoroigwe et al., 2014). Although the exact surface area or pore size could not be obtained from this particular image in Figure 5, but it was observed that the CDSP had more compact and distributed surface area.



Scale of 200 µm at 350 magnification Scale of 50 µm at 1500 magnification

Figure 4: SEM image of the UDSP samples



Scale of 200 µm at 350 magnification Scale of 50 µm at 1500 magnification

Figure 5: SEM image of the CDSP samples

This enabled the interaction between the filler and base material, which readily explains the reason why larger the surface area, the more reinforcing the filler would become. From the study therefore, the CDNS exhibited a surface area enhancement. This singular property gives the CDNS the reinforcing potential and will be useful as reinforcement agents in composite fabrication (Ekebafe et al., 2012).

In carrying out the XRD analysis, the structures were measured by scanning the particle samples in the range of 5°-70° of 2θ angle with a scan rate of 12°/min and a step size of 0.03° by a Powder X-ray Diffractometer (Shimadzu XRD-6000 using Cukα radiation) with a wavelength of 1.540562 Å. Figures 6 and 7 show the XRD pattern of UDSP and CDSP samples respectively.

Figure 6: XRD of UDSP sample

It is observed from Figure 6 that the powder samples contain large amount of noises, and this behavior confirmed the presence of amorphous structure of carbon in the UDSP sample (Shakuntala, 2005). The XRD pattern of the UDSP showed two major broad peaks that suggested that the material is most likely an amorphous, at peak 16.64° and 21.95° (Madakson et al., 2012). The phases present as obtained from the sleeve report are antimony oxide hydrate, silicon oxide and iron molybdenum oxide.

The XRD pattern for the CDSP in Figure 7 showed a broad peak at 19.88° which is usually a carbon phase. A Sepiolite (or magenesium silicate hydroxide hydrate) phase is also most likely present in the sample. This Sepiolite is similar to a Cordierite ($Mg_2Al_4Si_5O_{18}$) phase present in coconut shell (Madakson, et al., 2012).

The existence of these hard phases presents the usefulness of this material as material filler and a composite reinforcing material, especially as particulate reinforcement in metal matrix composites for automobile applications (Madakson et al., 2012; Okoroigwe et al., 2014).

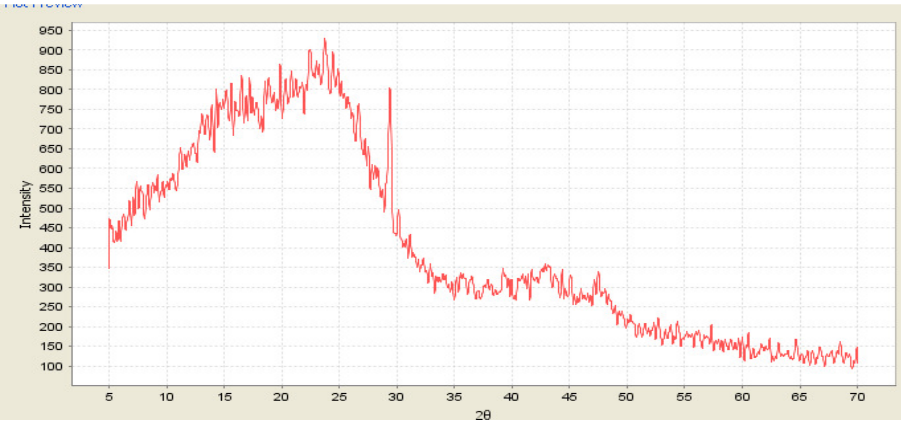


Figure 7: XRD of CDSP sample

4. CONCLUSION

As carbonization occurred, a well-developed pore structure was seen in the morphology of CDSP. From this study, it is acknowledged that the Dikanut shell is amorphous and contains phases such as antimony oxide hydrate, silicon oxide, iron molybdenum oxide, carbon and sepiolite. The up-to-date technique employed in this study further provides information on the material and its behavior and potential use to scientist and engineers, who may be interested in harnessing the properties of this material.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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