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CHARACTERIZATION OF UNTREATED AND TREATED GUINEACORN (*SORGHUM BICOLOR*) HUSK PARTICULATES

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Abstract

Growing global environmental awareness has raised the utilization of natural fibers as reinforcements in polymers due to government regulations globally that encourage the development of eco-friendly, sustainable and biodegradable materials. This study aims to characterize guineacorn (Sorghum bicolor) husk particulates (GHP) to assess its suitability as reinforcement in production of bio-composites. Untreated (UNTRD), alkali (ALK), silane (SIL) and the combination of alkali and silane (ALKSIL) treated GHP were considered in the study; taking into cognizance the density, thermal stability and surface morphology of the GHP; using helium gas pycnometer, Perkin Elmer thermogravimetric analyzer and JOEL-JSM 5600 series scanning electron microscope respectively. The density of the UNTRD GHP was found to be 1.47 g/cm^3, while those treated compared with the UNTRD GHP recorded percentage reduction in densities to the tune of 0.46 %, 1.19 % and 1.75 % for ALKSIL, ALK and SIL treated GHP respectively. The derivatives of thermogravimetric analysis (TGA) revealed that both untreated and treated GHP were stable up to 340 °C. Scanning electron microscopy images showed that surface treatments removed surface impurities resulting in a clean, rough and coated GHP surface. It was concluded that GHP is suitable for reinforcement in bio-composites with the surface treatments further improving its surface roughness; a quality that could enhance the interfacial bonding of GHP with polymer matrices.

Keywords: *Guineacorn-husk, treatments, density, thermogravimetry, scanning electron microscopy*

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1 Introduction

Processing of polymer matrix composites reinforced with natural fiber is one of the fastest growing industries (Zafar and Siddiqui, 2020). Growing global environmental awareness has raised the utilization of natural fibers as reinforcements in polymers on both research and industrial scale (Singh and Singh, 2019; Fiore *et al.*, 2016). A number of lignocellulose fibers such as jute, hemp, sisal and abaca have been used as reinforcements for biodegradable composites due their good mechanical properties and low specific mass (Shehu, 2015). These green fibers are now being sought as a result of the concerns about the fast depletion of limited petroleum reserves as well as the government regulations globally that encourage the development of eco-friendly, sustainable and biodegradable materials (Zafar and Siddiqui, 2020). The advantages of using lignocellulosic fibers as fillers/reinforcements in polymeric materials are numerous and include their low cost, availability, biodegradability, low density, high specific strength and stiffness, excellent absorbance properties and high impact energy absorption (Omole and Dauda, 2016). Lignocellulosic fibers are also abundant and renewable, their main advantage over synthetic fillers is their environmental friendliness (Shehu, 2015).

The effectiveness of composites materials depends on their ability to transfer stresses from the matrix to the reinforcement (Boussehel *et al*., 2018). However, the hydrophilic nature of the natural fiber due to large number of hydroxyl groups it possesses usually results in incompatibility with the hydrophobic polymers which causes poor interfacial bonding between the matrix and the reinforcement (Hu *et al*., 2020). Therefore, natural fiber treatment is necessary to modify its surface for improved adhesion with different matrices (Azizah *et al*., 2019). Interfacial interactions can be improved by different kinds of physical (Corona, Plasma, γ-radiation) and chemical (esterification, silane and sodium hydroxide) treatments (Shehu, 2015; Fiore *et al*., 2016). The chemical methods are usually based on the use of reagents which contain functional groups that are capable of being bonded to the hydroxyl groups of the lignocellulosic fiber, while maintaining good compatibility with the matrix. Robust interfacial compatibility improves the stress transfer between the two components and leads to the overall improvement of mechanical and physical properties of the produced composites (Khakalo *et al*., 2018).

Chemical modifications are usually applied to optimize the interface of fibers in that they may activate hydroxyl groups or introduce new moieties that can effectively interlock with the matrix so as to induce the required properties in the polymer (Shehu, 2015; Rahman *et al*., 2019; Amiandamhen *et al*., 2020).

Generally, chemical coupling agents are molecules possessing two functions; (1) they react with hydroxyl groups of cellulose and (2) they react with functional groups of the matrix. Quite a number of researchers have investigated the chemical modifications of lignocellulosic materials which is aimed at improving the adhesion with polymer matrices. One of the chemical methods used for surface modification of natural fibres is Alkali (NaOH) treatment, which is also known as mercerization. It disrupts the hydrogen bonding in the network structure of the lignocellulosic fibres thereby increasing surface roughness (Wang *et al*., 2019). This treatment removes a certain amount of lignin, pectin, wax and oils covering the external surface of the fibre cell wall, depolymerizes cellulose and exposes the short length crystallites (Teli and Jadhav, 2017; Vignesh, 2018; Nascimento *et al*., 2018). The equation of reaction is as adopted from the work of (Balaji and Nagarajan, 2017):

$$\text{Fibre–OH} + \text{NaOH} \longrightarrow \text{Fibre–O–Na} + H_2O \quad (1)$$

In mercerization, fibres are immersed in NaOH solution for a given period of time, removed, washed and subsequently dried.

Another chemical fiber modification is silane treatment. Silane is a coupling agent, which is capable of reducing the number of cellulose hydroxyl groups in the fibre– matrix interface. In the presence of moisture, hydrolysable alkoxy group leads to the formation of silanols. The silanol then reacts with the hydroxyl group of the fibre, forming stable covalent bonds to the cell wall that are chemisorbed onto the fiber surface (Okoronkwo and Madumelu, 2016; Amiandamhen *et al*., 2020). The chemical composition of silane coupling agents (bifunctional siloxane molecules) allows forming a chemical link between the surface of the cellulose fibre and the polymer matrix through a siloxane bridge (Liu *et al*., 2019). This co-reactivity provides molecular continuity across the interface region of the composite. It also provides the hydrocarbon chains that restrain fibre swelling into the matrix. Natural fibers exhibit micropores on their surfaces and silane coupling agent act as a surface coating which penetrates into the pores and develop mechanically interlocked coating on their surface (Mehesha *et al*., 2018; Wang *et al*., 2019; Gholampour and Ozbakkaloglu, 2019). As reported in the work of Shehu, (2015), the reaction schemes are:

$$CH_2CHSi(OC_2H_5)_3 \longrightarrow CH_2CHSi(OH)_3 + 3C_2H_5OH \quad (2)$$

$$CH_2CHSi(OH)_3 + H_2O + \text{fibre-OH} \longrightarrow CH_2CHSi(OH)_3O - \text{fibre} \quad (3)$$

Silane coupling agents have been found to be effective in modifying natural fibre–polymer matrix interface and increasing the interfacial strength (Lin *et al*., 2020). Three-aminopropyltrimethoxy silane with concentration of 1 % in a solution of acetone and water (50/50 by volume) for 2 h was reportedly used to modify flax surface (Naveen and Naidu, 2017).

Sorghum is a cereal crop cultivated as a major food in several countries in South Asia, Africa, and Central America (Visarada and Aruna, 2019). Nigeria is the largest sorghum producer in West Africa, accounting for about 71 % of the total regional sorghum output (Zalkuwi *et al*., 2014; Shehu, 2015). Nigeria's sorghum production also accounted for 35 % of the African production in 2007 (Sorghum production, 2014; Iloh *et al*., 2014; Garba *et al*., 2017). Studies on the use of sorghum wastes as reinforcement in polymer matrices have not been widely reported in literatures.

This research is aimed at the characterizing of UNTRD, ALK, SIL and ALKSIL treated guineacorn (*Sorghum bicolor*) husk particulates. Density, thermal stability and surface morphology were the properties investigated in this work.

2 Materials and Method

2.1 Materials and equipment

The materials and equipment used in this work include: Guineacorn husks from Zaria, Nigeria, 3-Aminopropyltriethoxysilane (3-APS) cat.44,014-0 Lot: 12100 from Sigma Aldrich, Germany, Sodium hydroxide (NaOH) BP pellets, Acetic acid (glacial) 100 %, Plastic containers, Laboratory trays, Hand gloves, Nose masks, Desiccator, Silica gel, Laboratory sized mini-crusher, Micromeritics Helium-gas pycnometer (Accupuc1330), Precision weighing balance (0.00001 g), Perkin Elmer Thermogravimetric Analyzer model-Pyris6, JOEL-JSM 5600 series Scanning Electron Microscopes (SEM).

2.2 Method

2.2.1 Preparation of guineacorn (*Sorghum bicolor*) husk particulates (GHP)

All The Guineacorn (*Sorghum bicolor*) stalk after the removal of the edible cereals were gathered from the local farmers in Zaria, Kaduna State, Nigeria after the harvest period (November - December) and the husks removed manually from the stalks. The husks were then washed thoroughly with water and detergent solution to remove all forms of dirt including stones, dried and subsequently ground in a mini crusher having a sieve of 0.5 mm attached to its outlet. The particulates were then oven dried at 105 °C for 24 hours and then stored in a desiccator before embarking on further processes. To remove volatile materials, the particulates were soaked in hot water for 24 hours, washed and dried in air and subsequently oven dried at 105 °C for 24 hours. The particulates were designated as untreated (UNTRD). The untreated particulates were treated with sodium hydroxide (NaOH) solution, 3-Aminopropyltriethoxysilane (3-APS) coupling agent and a combination of both treatments. The untreated particulates were used as control in this study.

2.2.2 Alkali (ALK) treatment

The untreated (UNTRD) particulates were treated with 5 % sodium hydroxide (NaOH) solution for 24 hours at room temperature after which it was washed with distilled water until all the NaOH was eliminated (Shehu, 2015). A little quantity of acetic acid was added to help in the quick neutralization process. This was confirmed by using a pH meter which showed neutral state of 7.2. After washing, the particulates were kept in air for 2 days to dry before oven drying at 105 °C for 6 hours and then stored.

2.2.3 Silane (SIL) treatment

The untreated particulates were treated with 3 % of 3-Aminopropyltriethoxysilane (3-APS) silane coupling agent. This was achieved by first hydrolysing the silane coupling agent in a methanol/water mixture in the ratio 50/50. The pH of the mixture was adjusted to 4.5 by adding a small quantity of acetic acid. The whole mixture was stirred before introducing the particulates into the mixture and left for 3 hours. The particulates were then removed, washed and kept in air for 2 days before oven drying at 105 °C for 6 hours and then stored.

2.2.4 Combination of Alkali and Silane (ALKSIL) treatment

The untreated particulates were first treated with sodium hydroxide solution as described earlier in the alkali treatment section, followed by treatment with silane coupling agent as described in the silane treatment section above. After the treatments, the particulates were

placed in plastic bags, sealed and stored in a desiccator before further analyses.

2.2.5 Density measurement

Untreated and treated guineacorn husk particulates (GHP) were subjected to density measurements using a helium-gas pycnometer. Each of the sample was weighed and introduced into a 10 cm^3 capacity sample holder of the pycnometer which was then inserted into the machine and tightly covered. The measured weigh of each sample to be tested was recorded in the machine before pressing the start button on the machine.

On starting the machine, a number of cycles involving purging and determination of volume of the samples was initiated. This was done for about 15 minutes before the calculated densities were displayed on the screen of the machine. 3 values were given for each sample and the average values were recorded.

2.2.6 Thermogravimetric analysis (TGA)

The TGA analysis was carried out in a Perkin-Elmer Pyris 6 TGA thermogravimetric analyzer.

Nitrogen was used as the purge gas at the rate of 20 ml/ minute. The samples (5-10 mg) were heated from 30 to 600 ºC at a rate of 10 ºC/ minute. The instrument was computer controlled and the calculations performed on the curves were done using Pyris 6 software.

2.2.7 Scanning electron microscopy (SEM)

The samples were studied by using JOEL-JSM 5600 series Scanning Electron Microscopes (SEM). The specimens were sputter coated with a thin layer of gold to avoid electrostatic charging during sample examination.

2.3 Results and Discussion

2.3.1 Density

The results for the density values of GHP (untreated and treated) are as shown in Figure 1.

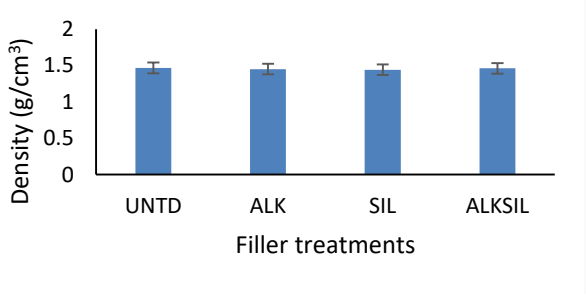

Figure 1: Density of Guinea-corn husks particulates against various treatments

From Figure 1, it was observed that the UNTRD GHP has the highest density value of 1.47 g/cm^3. The treated GHP on the other hand, has slightly lower density values of 1.46 g/cm^3, 1.45 g/cm^3 and 1.44 g/cm^3 for ALKSIL, ALK and SIL treatments respectively.

The density values obtained in this study which lied between 1.44 and 1.47 g/cm^3 were higher than those of some lignocellulosic fillers such as Rice husk flour (RHF) with a density value of 1.30 g/cm^3 (Kim, 2020), 1.068 g/cm^3 for wood apple shell particulate (Ojha *et al*., 2014) but lower than other lignocellulosic fillers such as coconut shell particulates with a density value of 1.60 g/cm^3 (Gayatri *et al*., 2016; Ojha *et al*., 2014), 1.5-1.6 g/cm3 for cotton fibers, 1.5 g/cm^3 for abaca, flax and sisal fibers (Mukhtar *et al*., 2018; Barba *et al*., 2020; Bakar *et al*., 2020) and 1.13 – 1.9 g/cm^3 for date palm fibers (Alshammari *et al*., 2019).

The reduction of density values with surface treatment containing alkali could be attributed to the fact that alkali treatment removes surface impurities and makes the surface of fiber rough (Thao tran *et al*., 2014); which is contrary to the findings of (Ouarhim *et al*., 2018; Hashim *et al*., 2017; Tenazoa *et al*., 2019) that reported increase in the densities of lignocellulosic fibers after alkali treatment. The reduction of density with silane treatment could be attributed to the removal of waxes from the filler as reported by Thao tran *et al*., (2014). The reduction of density value with surface treatment has been reported by Suardana *et al*., (2011) who stated that the density value of untreated hemp fiber as 1.249 g/cm^3 but on treatment with NaOH and silane coupling agent, the density values reduced to 1.127 g/cm^3 and 1.150 g/cm^3 respectively.

2.3.2 Thermogravimetric Analysis (TGA)

This The thermal analysis curves together with their derivative (DTG) curves which show the stages of decompositions and maximum decomposition temperatures are shown in Figures 2, 3 and the TGA data are summarized in Table 1.

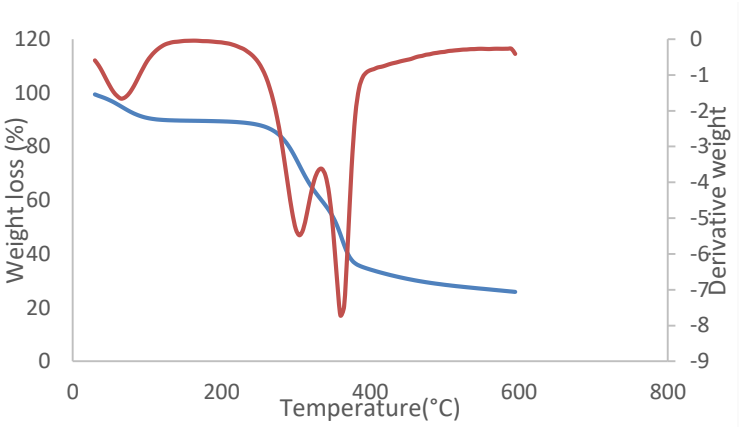

Figure 2: Weight loss and derivative weight of UNTRD GHP against temperature

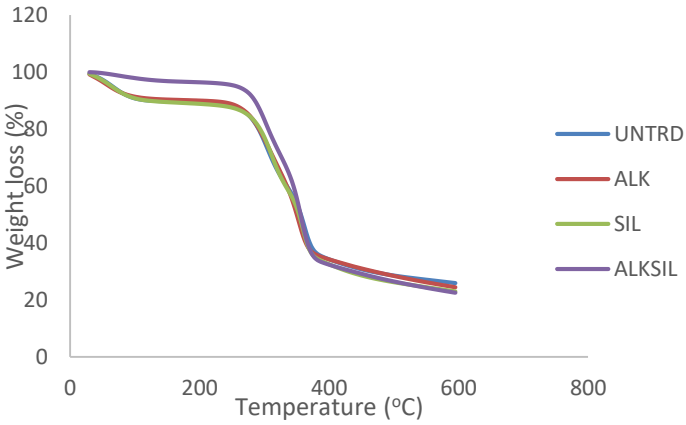

Figure 3: Weight loss of treated and untreated GHP against temperature

TGA is used in obtaining the thermal stability of fillers at their processing temperatures (Battegazzore *et al*., 2014). The TGA and DTG curves (Figure 2) show that the GHP exhibited 3- step decomposition. This was also reported for other fillers such as Luffa cylindrical fruit (NagarajaGanesh and Muralikannan, 2016), sugarcane bagasse (Mohomone *et al*., 2017) and sisal fiber (Chaitanya and Singh, 2017). The decomposition stages include:

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Table 1: Summary of TGA data for GHP

GHP	Onset (°C)	Endset (°C)	Dmax (°C)	Residue (%)
UNTRD	250	380	360	25.82
ALK	250	375	355	24.37
SIL	265	370	360	22.93
ALKSIL	260	380	355	22.45

i. 30 °C – 110 °C - due to the evaporation of moisture content in the filler (NagarajaGanesh and Muralikannan, 2016; Mohomone *et al*., 2017; Teli and Terega, 2017; Kale *et al*., 2018; Najeeb *et al*., 2020).
ii. 240 °C – 340 °C – due to the decomposition of cellulosic substances such as cellulose and hemicellulose (Eksiler *et al*., 2016; Eksiler *et al*., 2018; Najeeb *et al*., 2020).
iii. Above 340°C – due to the decomposition of non-cellulosic substances in the filler (Teli and Terega, 2017; Chaitanya and Singh, 2017).

From the combined curves (Figure 3), it was observed that the ALKSIL treated GHP exhibited the highest thermal stability while the others which include the UNTRD, ALK and SIL treated GHP overlapped.
The derivatives of the thermogravimetric curves (DTG) of treated and untreated GHP show the main decomposition temperatures of the GHP (dmax) presented in Table 1. It indicated that GHP, whether treated or untreated is stable up to 340 °C with a maximum rate of decomposition at 350 °C.
From Table 1, the onset decomposition temperature of UNTRD and ALK treated GHP was 250 °C while it was 265 °C for SIL treated GHP and 260 °C for ALKSIL treated GHP. Also, the maximum decomposition temperature for UNTRD and SIL treated GHP was 360 °C while that of ALK and ALKSIL treated GHP was 355 °C.
The residues or ash contents at 600 °C was highest for the UNTRD GHP with a value of 26 % while the least residue was obtained for the ALKSIL treated GHP with a value of 22 %. The reason for relative high residue for the UNTRD GHP and lower values for the treated GHP could be due to the presence of impurities with regards to the UNTRD that were probably removed with various surface treatments.

2.3.3 Morphological study of GHP

The SEM images for both the treated and untreated GHP fillers are shown in Figure 4a – d.

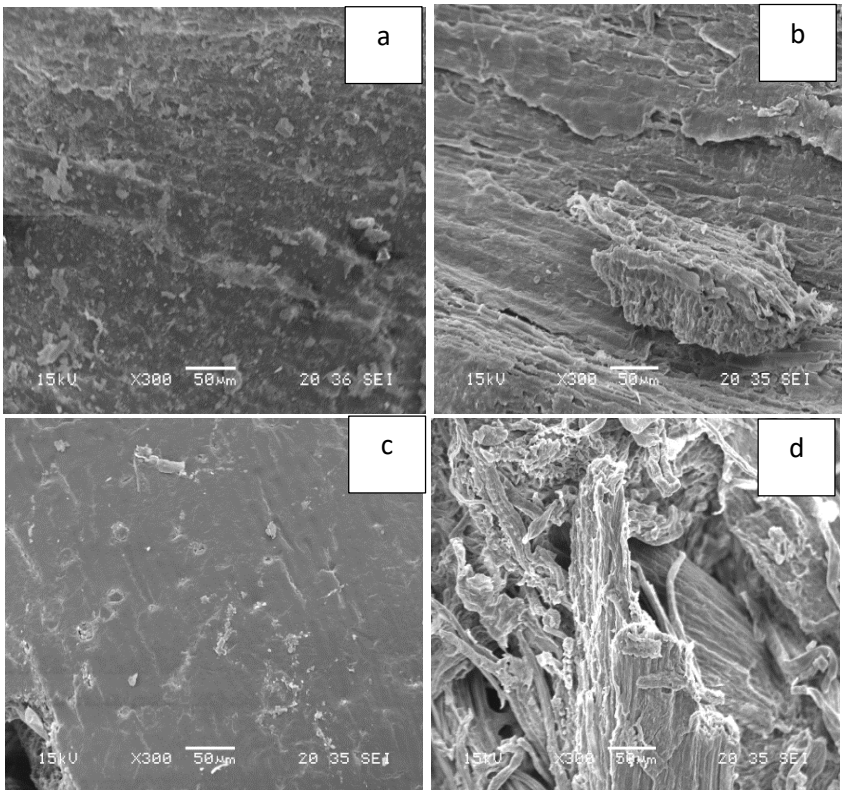

Figure 4: SEM micrographs of untreated and treated GHP (a). UNTRD (b). ALK treated (c). SIL treated (d). ALKSIL treated

From Figure 4a which shows the SEM image for UNTRD GHP, it was observed that the filler exhibited some scaly substances that could be waxes which cover the surface of the filler. The scaly covering was said to be a membrane layer called pellicle (Shehu, 2015; Kumar Singha *et al*., 2017).

The SEM image for ALK treated GHP (Figure 4b) showed a rough and cleaner image as compared with the UNTRD. Treatment with alkali removes surface impurities such as lignin, pectin, hemicellulose, oils covering the external surface of the filler cell wall and makes the surface of fillers rougher (Thao tran *et al*., 2014; Shehu, 2015; Mukaida *et al*., 2016; Kumar Singha *et al*., 2017; Bartos *et al*., 2020). The membrane that was observed in the UNTRD GHP was removed after alkali treatment.

The SIL treated GHP in Figure 4c looks a bit similar to that of UNTRD GHP but with little difference which could be as a result of removal of waxes from the surface of the GHP. As reported by Thao Tran *et al*. (2014), silane treatment removes waxes from husks surfaces which does not really lead to very noticeable change of the GHP surface after SIL treatment.

Liu *et al*. (2019) reported that SIL treatment resulted in the coating of natural filler surfaces as it was deposited on the filler surface which suggests the similarity between the UNTRD and SIL treated GHP.

Figure 4d shows the SEM image of ALKSIL treated GHP. The image showed a clean, rough as well as a coated surface which resulted from the alkali treatment followed by silane treatment.

2.4 Conclusion

The research focused on the characterization of GHP to be used as filler in bio-composites production. At the end of the study, the following conclusions were drawn based on the results obtained;

1. UNTRD GHP recorded the highest density of 1.47 g/cm^3, while the treated GHP recorded 1.46 g/cm^3, 1.45 g/cm^{3}and 1.44 g/cm^3 for ALKSIL, ALK and SIL treatments respectively.
2. The combined TGA curves revealed that ALKSIL treated GHP exhibited the highest thermal stability, while the UNTRD, ALK and SIL treated GHP curves overlap; the derivatives of the TGA curves showed that GHP, whether untreated or treated is stable up to 340 °C.
3. The residues after thermal degradation at 600 °C was highest for the UNTRD GHP with a value of 26 %, while the least residue was obtained for the ALKSIL treated GHP with the value of 22 %.
4. SEM images revealed that treatment with alkali removes surface impurities such as lignin, pectin, hemicellulose and oils. It shows that silane treatment resulted in coating of filler surface while the combination of alkali and silane treatments reveal a clean, rough as well as coated GHP surface. This quality could enhance the interfacial bonding of GHP with polymer matrices.

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