

Studies on Physico-Mechanical Behaviour of Sawdust/Glass Fiber Reinforced Epoxy Hybrid Composites

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Abstract

In this study, various treated, untreated, and treated Sawdust/glass fiber composites were fabricated using epoxy resin. Wood saw dust were obtained and subjected to boiling at 100 °C, later washed, dried under shade for 24 *hours. The fiber were subjected to alkali, potassium permanganate, hydrogen peroxide treatment prior to composite fabrication. Composite of untreated and treated sawdust/glass fibers were fabricated using hand lay-up technique. The composite material was subjected to these treatments in order to achieve bond enhancement adjustment at the matrix-fiber bond interface. A part of the glass fiber was used to improve the hybrid composite's mechanical properties. The tensile strength, flexural, density, chemical resistance and water absorption of the composites were analyzed using standard methods. FTIR was conducted on the fiber to ascertain the chemical treatment on the fibers, SEM was used for the morphological study. The results obtained showed that the tensile and flexural strength improved from 17-225.5 kgf, and 13.9-158.4 kgf respectively in the composite samples. Chemical resistance of the Sawdust fiber treated potassium permanganate composite improved compared to the untreated fiber composites in both cases. Water absorption properties of the composites were found to decrease in the treated fibers compared to untreated fiber composites. The FTIR result revealed that reaction has taken place between the fiber and the treatment reagents. Hence, the fiber modification has improved the properties of the composites due to increase in fiber-matrix interaction.*

Keywords: *Chemical properties, Epoxy resin, Hybrid, Sawdust, Mechanical strength and Natural fiber*

1. Introduction

Recently, a significant increase is observed in the field of materials' research, both in academic and industrial sectors, to develop innovative "green materials "exploiting natural resources(Mohammed et al, 2015; Muralidharaet al., 2020; Pickeringet al., 2016) particularly, biodegradable fiber reinforced polymeric materials, alternatively known as bio composites, are of interest (Mohammed et al., 2015; Muralidhara et al., 2020; Nanda et al., 2020). Plant-based natural fiber is a high-potential region of reinforced polymer plastic material that is lightweight and cost-effective, the use of plant fibers in the central structure of composite materials has piqued the manufacturers' interest (Sakil et al., 2021). Although glass fiber reinforced polymer composites (GFRP) possess better mechanical properties and reduced cost compared to others, the wide use of these may cause serious negative impacts on human health and environment. Because of governmental emphasize on environmental legislations and sustainability as

well as customer concerns, the manufacturers of materials and end-products are on pressure to consider the environmental impact of the products' whole life cycle including manufacturing, recyclability and ultimate disposability in nature (Mohammed et al., 2015; Muralidhara et al., 2020; Nanda et al., 2020). Consequently, Many scientists are devoting time and effort to improving conventional fabrics and developing new polymer composites made of naturally occurring biodegradable materials (Mandal et al., 2020). A number of natural fibers such as hemp, jute, sisal, kenaf, bamboo, flax may be utilized in reinforcing polymer based matrices (Muralidhara et al., 2020;Nair et al., 2018). In a humid environment, all natural fiber reinforced polymer composites absorb moisture, and when immersed in water, moisture absorption by the natural fiber composites causes the fiber matrix interface area to degrade, resulting in low stress transfer efficiencies and a decrease in mechanical and dimensional properties (Mohammed et al., 2015). One of the major drawbacks of using natural fiber reinforced composite materials is their proclivity for moisture absorption, which has an effect on the composites' physical, mechanical, and thermal properties (Pickering et al., 2016). Thermosetting polymers cure irreversibly, which cannot be recycled, hence, in order to protect environment, natural decomposition of the products derived from thermosets is essential (Saba, et al., 2016; Atiqah, et al., 2020). In this regard, reinforcing these polymers with natural fibers may be a viable solution, which leads to the natural degradation of the discarded products. The use of natural fibers in replacement of inorganic materials has significantly reduced the consumption of non-renewable and non-biodegradable materials(Hu, et al., 2020). They have been seriously considered in reinforcing polymer matrix due to a number of reasons(Nair et al., 2018; Nanda et al., 2020; Jojibabu, Zhang, & Prusty, 2020). Natural fibers are readily available in nature, being usually waste material they are relatively cheaper as well (Nair et al., 2018). Researchers who have been able to build a sandwich structure with reduced water absorption and optimal mechanical strength using natural fibers have focused on fiber treatment and hybridization (Alsubari, et al., 2021). They have low abrasive properties and as consequence easier to process compared to those of harder inorganic fillers, such as glass fibers or mica. Low-density, low-cost, and recyclability are all advantages of natural fibers over man-made fibers (Nair et al., 2018; Nanda et al., 2020; Jojibabu et al., 2020;Prakash et al.,2019; Mazlan et al., 2020; Burda et al., 2019; Raajeshkrishna et al., 2020; Danuta Matykiewicz (2020). Furthermore, some commonly used natural fibers, such as jute, hemp flax, and sisal, have mechanical properties similar to glass fiber in terms of specific intensity and modulus, and are hence being used to replace synthetic fibers for some applications (Muralidhara et al., 2020; Pickering et al., 2016; Sathyaseelan et al., 2020).

Wood is a naturally occurring material that is used in a variety of applications such as agriculture, manufacturing, transportation, and furniture. As compared to the cost of other polymer, wood saw dust is significantly less expensive. If wood sawdust can be used as a reinforcement in polymer to create composites, the cost of polymer can undoubtedly be reduced (Hu et al., 2020; Nanda et al., 2020; Nair et al., 2018). Wood saw dust polymer matrix composites have physical and mechanical properties that make them suitable for industrial applications such as electrical and thermal insulators, mobile casings, multi connectors, adapters, switchboards, vehicle components, hardboards, utensil containers, and so on. (Mittal et al., 2016; Hu et al., 2020). Furthermore, addition of degradability of the polymer matrix composites, thus an environment friendly material is possible in a very low cost (Mohammed et al., 2015). The main aim of this paper is to fabricate conventional composite of modified wood sawdust glass fiber reinforced epoxy resin hybrid composites, compare the mechanical properties and to provide a substitute for synthetic fiber in the polymer processing industries.

2. Experimental

The epoxy resin Bis (phenol-A-diglycidyl-ether), cross linker HY-951 and the glass fiber were obtained from Nycil Nigeria Limited, the resin was used as a matrix material with the grade LM-556 and density of 1.3 ± 0.2 g/cm³. The sawdust fiber was obtained from a local market in Kumbotso local government, Kano State (Latitude 12° 01'05.55"N Longitude 8°58'05.55"E).

2.1 Method

Pre-Treatment

The Saw dust were collected and subjected to boiling at $100 \degree$ C for one (1) hour in order to remove impurities(Nair et al., 2018).

2.1.1 Alkaline Treatment of Sawdust Fiber

Initially, the fiber was cleaned several times with non-ionic detergent, a solution of 2 %. It was then soaked in 5 % solution of sodium hydroxide (NaOH) in thermostat system for three hours at 96°C. It was to add the sodium ion $(Na⁺)$ to the fiber backbone for the hydroxyl (OH-) activation cellulose group inside the fiber. Over-delignification of natural fibers can occur at concentrations above 5%, resulting in damaged or deteriorated fiber. The fiber was then thoroughly washed with purified water and dried in an air-flow at 70°C for 24 hours. The fiber was called Sawdust alkali-treated (Mittal et al., 2016).

2.1.2 Peroxidation

With 2% non-ionic detergent solutions, the fiber was washed and soaked for 1 hour in 2 percent NaOH solution. It was then soaked for 30 minutes in acetone in a solution of benzoyl peroxide (concentration of 6%). For comparison purposes, some portions of the material had been immersed in hydrogen peroxide instead of benzoyl peroxide. Full peroxide decomposition was accomplished by heating the solution at a temperature 180 ° C. The Sawdust fiber has been known as treated benzoyl peroxide (Abdullahi et al., 2020).

2.1.3 *Potassium Permanganate KMnO4 Treatment*

Potassium permanganate (KMnO₄) 0.125% was dissolved in 90ml of water and 6% of acetone was applied to the solution. 15 g Sawdust fiber treated with alkali was dipped within the solution and held for 30 minutes. The fiber was sieved after 30 minutes and dried under shade at room temperature for 7 days (Abdullahi et al., 2020).

2.1.4 *Polymer Matrix*

Resin of epoxy belongs to a family of thermosetting polymers that experience cross linking in the presence of a hardener (cross linker) when polymerized. The matrix material was prepared in a volume ratio of 2:1 with a mixture of the epoxy resin and hardener (Burak at al., 2020).

2.1.5 *Composite Fabrication*

The hand lay-up method was used to build this composite. To avoid air bubbles, the epoxy resin and cross connector (60:40) are carefully mixed together with gentle stirring. For fast and simple removal of the composite sheets, a mold release agent was added to the mold's inner side. Since holding the mold onto a glass tray, a thin coating of the matrix mixture was poured. The requisite amount of fiber was then spread on the mixture, then, as seen in Scheme 1, pour the remaining mixture into the mold. To keep the air bubbles from forming, a sonication device was used. After that, the mold was allowed to regenerate for two hours at room temperature., during which the samples were taken out of the mold and kept for further experimentation(Abdullahi et al., 2020;Birniwa et al., 2019; Yakubu, et al.,2010).

Scheme 1. Schematic representation of the mold used.

2.2 Moisture Absorption Test

To determine the amount of water absorbed under the specified conditions, water absorption test was used. As per ASTM D570-98 standard. (ASTM D570-98, 1998). Water absorption is represented as weight level rise. The strong ability of natural fibers to retain moisture negatively influences adhesion with a hydrophobic matrix and as a result may induce substrate deterioration and loss of resistance. The sample was pre-weight (W_1) and was submerged for

24hours in their respective tank of purified water. Pressing all sides of the polymer with tissue paper and re-weighing (W2) done after pressing all sides of the composite samples with tissue paper. The % weight loss/weight gained of the composite was evaluated as reported by (Mosadeghzada et al., 2009) using equation 1.

% weight =
$$
\frac{W^2-W^1}{W^4}
$$
 × 100%equation 1.

 $\%$ weight = $\frac{W2-W1}{W1} \times 100$
Where W₁ = initial weight, W₂ = final weigh of the sample.

2.3 Chemical Resistance Test

Acid and base were used to perform the chemical test of the composite (H₂SO₄, HCl, NH₄OH and KOH). In 90ml of water, the acid and base used was 10%, in accordance with ASTDM standard D-570 (Raajeshkrishna et al., 2020). Using each chemical, the composite samples were tested, the average values were evaluated. The composites was pre-weight (W_1) and were dipped into the respective cylinders for 24 hours at room temperature. The sample dried by pressing with tissue paper on the side of the sample and re-weighted immediately as if (W_2) . Similarly, the weight gain/ loss of the samples was calculated from equation 1.

2.4 Tensile strength Test

Following ASTM D3039 / D3039M-00 (2006) method, (Danuta Matykiewicz, 2020). Traction strength of dog bone shape specimens of 140 to 35 to 10 mm was performed using the standard ultimate testing machine (UTM), Shimadzu (Model AG-1) with 20 mm/min cross head speed (Saba et al., 2016; Jojibabu et al., 2020).

2.5 Flexural Test

An Instron IX 4302 unit, using the 3-point bending fixture according to ASTMD-790, was also used for flexural checks. A crosshead speed was used at 5 mm per minute. The test sample dimensions were typically 60x 6x2 mm. The composite findings were collected from 3 research specimens. The value of the flexural force (π) was calculated in accordance with equation $2 = 3FL/(2dh^3)$ (Birniwa et al., 2019).

2.6 Fourier transform infrared (FTIR)

The treated and untreated fibers were analyzed with Agilent Cary 630 FT-IR spectrometer at room temperature FT-IR spectra were recorded on the fiber samples. The spectra of the fibers were obtained within the frequency range of 4000-600 cm^{-1} .

2.7 Field Emission Scanning Electron Microscopy (FESEM)

The surface morphology was observed *via* field emission scanning electron microscopy (FESEM) [Hitachi Brand, Model SU 8220] with different magnifications.

3. Results and Discussion

By exposing more reactive groups, the chemical treatment of the fiber was meant to improve the surface of the fiber. The coupling smoothness between the fiber and the polymer resin is further facilitated by this. The resulting material would also have improved mechanical properties, allowing the creation of molecular cellulose sawdust fiber. This is intended to make reactive groups on the surface of the fiber more readily accessible. As such, interfacial properties will be strengthened, while the mechanical properties of the better performing natural fiberreinforced polymer composite will be created. Treatment with alkali of sawdust natural fibers have given rise to the growth of cellulose-O-Na⁺ groups in the material. While new reactive H-bonds are created between the cellular, molecular chains, and the normal crystalline structure of the cellulose, the H-bonding present in the network structure is broken. Therefore, fiber attachment of aqueous NaOH. The ionization of the hydroxyl group to the alkoxide was induced by sawdust natural fiber. Fiber washing later made it possible for $O₁$ to react with H₂O to produce more hydroxyl groups on the fiber surface. This formed a hydrogen bond and also made it possible to obtain a neutral pH (Birniwa et al., 2019). As a result of alkali treatment, the activation of hydroxyl groups and at the same time the cleaning of the surface of the cellulosic fiber were produced (Scheme 2). The removal of any proportion of pectin, hemicellulose, lignin, waxy epidermal skin, and oil wrapping adhesives resulted in a reduction

in fiber diameter and an improvement in aspect ratio. It also increases the surface area of the operative fiber for excellent matrix adhesion (Abdullahi et al., 2020).
 $P_{\text{H}_2\text{OH}}$

Scheme 2: Alkaline Treatment of Sawdust Fiber (Birniwa et al., 2019)

In nature, the hygroscopic natural fiber-reinforced composite is obvious; hence the composites are significantly affected by water. The natural fibers are absorbents of water and bulge. The fiber-resin interface is impaired by this, thereby damaging the mechanical properties. The natural fiber is penetrated by water and fibrils are split from the natural fiber. The characteristics of the loss of fiber are important. However, its moisture resistance after increased composite form adjustment of the fabric, as seen in Table 1. The observation of (Birniwa et al., 2019; Yakubu, et al.,2010) coincides with that result.

3.1 Chemical Resistance Test

The natural composite materials reinforced with fiber are hygroscopic in nature, so the composites are greatly influenced by water. Natural fabrics absorb water and bulge, respectively. The fiber resin interface is therefore weakened, so the mechanical strengths are lost. The water reaches the natural fiber and splits into fibrils of the natural fiber. Thus, the power of the fiber is substantially lost. However, as seen in Table 2, showed slightly reacted with hydrochloric acid, but improved after alteration of the fiber by composite forming. The chemical tolerance arising from Table 3 and Table 4 indicates that the composite species was resisted to chemical attack of H_2SO_4 and KMNO4 respectively. Cellulose, embedded in a matrix of hemicellulose and lignin, is the reinforcing part of the natural fiber. When the matrix stability is lost, the breakdown of the fiber happens, with the supporting fibrils and natural hydrogen bonding in the cellulose being impacted. Therefore, the lower the cellulose material, the lower the strength of the tensile(Birniwa et al., 2019; Yakubu, et al.,2010).

Alkaline fiber treatment helps to strengthen the chemical bond between the fiber and the solvent, resulting in improved mechanical properties. Chemical Sawdust fiber treatment induces fibrillation of fibers to tiny fibrils, increasing surface area with the decrease in fiber diameter, contributing to wet fiber capability within the matrix. For this purpose, the interfacial area is the most favorable aspect of the composite, as shown in Table 2-4, acids and base were used. The test of chemical resistance shows that some of the composite is resistant to chemical attack, i.e. they show an increase in acid and alkaline weight, some of the composite is discovered to dissolve in the acid and base. This is found in certain composites due to reduced weight.

The treatments, as stated by (Yakubu et al., 2010) led to enhanced surface roughening, thus facilitating mechanical bonding. It can be seen from the outcome in Scheme 1 and 2 that sawdust is handled with alkali epoxy/sawdust, by comparing those with the untreated sawdust/glass fiber/epoxy composites demonstrated enhanced qualities of mechanical properties tensile/flexural). Composites of sawdust/epoxy peroxidation reaction using benzoyl peroxide appears to quickly crumble to create free radicals of the form RO; RO• with subsequent reactions to form cellulose radical with the hydrogen group of cellulose fibers.

The free-radical reaction induced by peroxide is seen in Scheme 3.

$RO - RO \rightarrow 2RO \cdot$

RO• + Cellulose―H → ROH + Cellulose•

Scheme 3: Reaction of sawdust fiber with benzoyl peroxide.

This treatment decreases the propensity of the fiber to absorb moisture and increases dimensional stability, and it has been stated that benzoyl peroxide treatment has resulted in higher mechanical properties of composites(Birniwa et al., 2019). As seen in Figures 1 and 2, fiber was treated with peroxide. As compared to those of untreated sawdust fiber, sawdust/epoxy composite displayed improved mechanical properties. The (Tensile) mechanical test, the result also showed an improvement in almost all of the chemical treated samples as (a) untreated 17.0, (b) benzoyl peroxide with glass fiber 225.5, (c) potassium permanganate 171.8, (d) alkali 163.3 and (e) benzoyl peroxide without glass fiber 171.4 as shown in Figure (1). The stress strain curve was used to measure the composites' tensile modulus and elongation at split. For each collection of samples, five specimens were checked, and the average values were recorded. Figure 1 shows the total maximum tensile tension for the untreated, alkali, permanganate, hydrogen peroxide, and hydrogen peroxide for glass fiber composites. (Flexural) (a) untreated 13.9, (b) benzoyl peroxide with glass fiber 158.4, (c) potassium permanganate 138.9, (d) alkali 119.6 and (e) benzoyl peroxide without glass fiber 135.2 as shown in Figure (2). Composites of sawdust/epoxy findings of tensile strength reveal that the maximum tensile strength is in the fiber treated with glass. The morphology of the fiber also determines the natural fiber's tensile ability. It is predicted that the fiber with the smooth surface has high tensile strength, and the fiber with the rough surface finish has less strength. The addition of glass fiber into fibers (forming composites) has been shown to result in maximum tensile strength. It was found in the same manner, that composites of Nilotica mixtures can make full effect (Swain & Biswas, 2017).

3.2 Potassium permanganate Treatment on Saw dust fiber

Figure 2: Flexural Strength.

3.3 Fourier Transformed Infrared Spectrophotometric Analysis

Figure 3 shows the FT-IR spectra of (a) untreated, (b) treated benzoyl peroxide, (c) treated alkali, and (d) treated potassium per manganite Sawdust fibers respectively.

Figure 3: FTIR Analysis of Saw Dust Fiber.

Figure 3 (a), (b), (c) and (d) shows the FT-IR spectra of untreated, benzoyl peroxide, alkali, KMnO₄ Sawdust fibers respectively. All the studied fibers have O-H stretching vibrations in the range of 3630-3328 cm⁻¹. C-H stretch was located in the range of 2935 -2916 cm⁻¹ obtained from methylene, C=C was found in the range of 1655 -1639 cm⁻¹ obtained from lignin, C-O stretching vibrations from ether were observed in the range of 1033-1032 cm⁻¹ and C=C, bending for alkene in the range of 791- 661 cm⁻¹ were observed. It has been observed that all fibers exhibited similar peaks, but the intensity of the peaks increases upon treatment as seen in Figure 3 (b-d). Changes in the intensities of the treated sawdust fibers were found. The –OH functional group is responsible for the peaks in the region of 3328cm-1 to 3650cm-1, in the case of treated fibers, the intensities within this area were minimized. This may be because enzymatic activity reduces hydrogen bonding in cellulosic hydroxyl groups, lowering the concentration of –OH, this drop in severity was also due to the elimination of hemicelluloses, which revealed the cellulose's OH groups. Because of the increased visibility, the reinforcing fiber could be able to physically entangle with the polymer matrix more powerfully. All the FT-IR spectra of untreated, alkali, KMnO4, benzoyl peroxide treated fibers are similar, but the intensity of the peaks increases due to the treatment on the fiber by removing impurities, such as lignin, hemicellulose, pectin and waxes (Birniwa et al., 2019; Abdullahi et al., 2020).

3.4 SEM Analysis

Longitudinal views of the sawdust fibers that were untreated and processed are shown in Fig. 4 (a-d) As we can see in Fig. 4a, there are also impurities on the surface of the fibers, raw sawdust fibers with an irregular shape that arranges longitudinally in a circular pattern. It is found that because of therapy, the surface morphology of the untreated fiber distinctly varies. In fact, after potassium permanganate treatment, the least difference was observed on the fiber surface (Fig. 4b).

Fig. 4 (a-d) shows the FESEM images (a) untreated, (b) treated benzoyl peroxide, (c) treated alkali, and (d) treated potassium per manganite Sawdust fibers respectively. Longitudinal views of the sawdust fibers that were untreated and processed.

As a part of this procedure, it is noticed that the impurities are cleaned from the fiber surface and the tiny ridges are seen on the surface of the fiber. The surface roughness of the fiber is improved by this interaction. That can be an advantage because by interlocking between the fiber surface and the resin interface, the rougher fiber surface increases the mechanical properties of composite materials(Sultana et al., 2020). However, on the alkali treated fiber board, the highest improvement in roughness is obtained (Fig. 4c). Hemicelluloses and lignin, which are cement from the fiber body, are the primary cause for fibrillation (Saxena & Gupta, 2019). The enhanced surface roughness can be easily observed as a result of the appearance of multiple fibrils on the fiber surface. Furthermore, fragments linger on the surface of the fiber until treatment. A signature result very distinct from other treatment is found during treatment with benzoyl peroxide. As is visible from Fig. 4d, there was an unnecessary reduction in the gaps and pits on the fiber board. Interaction of the reactive site of benzoyl peroxide with the lignin components may be the cause for the reduction on the fiber surface(Jarukumjorn & Suppakarn, 2009; Mosadeghzada, et al., 2009).

4. Conclusion

This research demonstrates the efficient production of epoxy composites strengthened by Sawdust fiber and Sawdust/glass achieved by utilizing a direct hand lay-up technique. The surface modification is critical for decreasing the hydrophilicity of Sawdust fibers and for improving the contact force with the hydrophobic structure of the matrix. The surface treatments of alkalization, benzoylation, and potassium permanganate used in this work modifying the cellulose component of the fiber influenced the mechanical properties of the resulting composites. These were tested effectively using FTIR as shown in the research. The test indicates that tensile strength can be

determined for the treated fiber, the results showed that the treated blend Sawdust potassium permanganate composites had a highest tensile and flexural strength compared to the remaining composite samples. This showed promising property in improving fiber resin interaction and mechanical test implies strong resistance to deformations that may be encountered during usage of the composite for various applications.

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