



Study on Physico-Mechanical Behaviour of *Acacia nilotica* (Gum Tree) and Glass Fiber Blend Reinforced Epoxy Resin Composite

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ABSTRACT

The mechanical properties of *Acacia nilotica* were investigated by chemical modification of the fiber-reinforced composite. For this, the bast fiber was retted enzymatically, washed, dried, combed and alkali treated using a mixture of benzoyl peroxide and sodium hydroxide. This treatments on the composite material was undertaken in order to achieve improved modification of the interface between the matrix and fiber bond. A portion of the glass fiber was used for enhancing the mechanical properties of the hybrid composite. Epoxy resin and hardener were also used being 60:40 respectively in composition, to fabricate the composite. The results show an improvement in all the properties tested in relation to the alkali and the acid treatments of the *Acacia nilotica*/glass fiber blend composites. Tensile strength of the *Acacia nilotica* treated with the glass fiber by benzolation improved from (40 to 240 MPa) the flexural strength, of the *Acacia nilotica* treated by benzolation also significantly improved from 8 to 28 MPa strength value. FTIR analysis confirmed that the chemical modification of the fiber bond was carried out.

Keywords: *Acacia nilotica*, Composite, Epoxy, Glass fiber, Natural fiber

INTRODUCTION

A composite material is a 'material system' composed of a combination of two or more micro or macro constituents that differ in form and chemical composition where the components are essentially insoluble in each other (Sumit *et al.*, 2018; Mathur, 2006). The two phases are the reinforcing phase and matrix phase. Composites are one of the most advanced and adaptable engineering materials known and used for various application (Şahin and Mehmet, 2018). Progress in the field of materials science and technology has given birth to these fascinating and wonderful materials. A composite material can provide superior and unique mechanical and physical properties because it combines the most desirable properties of its constituents while suppressing their least desirable properties (Montenegro, 2019).

At present, composite materials play a key role in the aerospace industry, automobile industry, and other engineering applications as they exhibit outstanding strength to weight and modulus to weight ratio (Azadi, *et al.*, 2019). Composites are essential materials because of their strength and stiffness, yet they can be very light in weight. Therefore, ratios of strength to weight and stiffness to weight can be several times stronger than steel or aluminum. It is also possible to achieve combinations of properties not attainable with metals, ceramics, or polymers alone (Kafi *et al.*,

2006). Interestingly, all these advantages enable composite materials to be widely applicable in aerospace and automobile industries (Azedi *et al.*, 2019) In recent times, there has been a growing interest towards using natural fibres as composite re-enforces (Lei *et al.*, 2018; Xie *et al.*, 2010). This is particularly common in the case of synthetic polymers due to the attractive properties of the resulting materials. For instance, epoxy resin is one of the polymer matrix subjected to such modification due to its high strength, high hardness, chemical stability and its low cost (Dadrasi, *et al.*, 2019; Gang *et al.*; 2018).

In this work, a reinforced epoxy resin composite material will be prepare using natural fibre obtained from *Acacia nilotica*. Consequently, change in the physico-mechanical properties of the fiber reinforced composite will be investigated. Prior to the composite preparation, the natural fiber was subjected to chemical treatment in order to enhance its surface chemistry.

MATERIALS AND METHODS

Materials

The epoxy resin Bis (phenol-A-diglycidyl-ether), crosslinker HY-951 and the glass fiber were obtained from NYCIL Nigeria Limited, Lagos. The resin was used as a matrix material with the grade LM-556 and density of $1.3 \pm 0.2 \text{ g/cm}^3$. The *A. nilotica* fiber was obtained from a local farm in

Gada village, Kazaure local government, Jigawa state.

Fiber Extraction

The *A. nilotica* fiber was extracted using water retting process. Thus, the bark of *A. nilotica* fiber which was cut from its tree was soaked in a trough of water at room temperature for seven days. After which the water fed on the pectin, lignin and other impurities contained in the fiber. It produced an unpleasant odor with gummy materials. The fiber appeared fresh and milky in a sheath of networked structure. The resulting fiber were removed by hand scratching and carded with a soft nylon brush. This was then washed with tap water several times and allowed to dry for three days under shade (Gumel and Tijjani 2017; Velmurugan, *et al.*, 2012;). The dried fiber was brushed using a hand nylon brush resulting in a fine strand of the material ready for treatment and composite fabrication.

Alkaline Treatment

The fiber was first washed with non-ionic 2% detergent solution several times. It was later immersed in 5% sodium hydroxide solution (NaOH) for three hours at 96 °C, in a thermostat machine. This was to activate the hydroxyl (OH⁻) groups on the cellulose within the fiber and to introduce the sodium ion (Na⁺) on the fiber backbone. At higher concentration above 5%, excess delignification of natural fibers can occur, resulting in a weaker or damaged material. The fiber was then washed thoroughly with distilled water and dried in an air oven at 70°C for 24 hours. The fiber was designated as alkali-treated *A. nilotica* (Sumit *et al* 2018).

Peroxidation

The fibre was washed with 2% non-ionic detergent solutions and soaked in 2% NaOH solution for 1 hour. It was subsequently immersed in benzoyl peroxide (around 6% concentration) solution in acetone for 30 minutes. Some portions of the material were immersed in hydrogen peroxide instead of benzoyl peroxide for the purpose of comparisons. Complete decomposition of peroxide was achieved by heating the solution at higher temperature 180°C. The *A. nilotica* fiber was designated as benzoyl treated (Lingtong *et al.*, 2018).

Polymer Matrix

The epoxy resin belong to a class of thermosetting polymers that undergo cross linking when polymerized in the presence of a hardener and a crosslinker. The matrix material was prepared with a mixture of the epoxy resin and hardener in the volume ratio of 2:1 (Niharika and Acharya, 2013).

Composite Preparation

The hand lay-up technique was used for the preparation of this composite. Thus the epoxy resin and hardener of (60:40 respectively) were carefully mixed with gentle stirring for the purpose of removing air bubbles. For quick and easy removal of the composite sheets, a mold release agent was applied at the inner surface of the mold. After keeping the mold on a glass sheet, a thin layer of the matrix mixture was poured. The required amount of fiber was then distributed on the mixture. The remaining mixture was then poured into the mold. Sonication machine was used to avoid the formation of air bubbles. The mold was then allowed to cure at room temperature for two hours after which the samples were taken out from the mold and kept for further experimentation (Abubakar, 2004).

Moisture Absorption Test

Water absorption is used to determine the amount of water absorbed under specified condition. The test specimen was manufactured as per ASTM standard D570. The water absorption is expressed as increasing weight percent. The high moisture absorption capacity of natural fibers adversely affects adhesion with a hydrophobic matrix and, as a result, it may cause material degradation and loss of strength.

The composite was pre- weight (W_1) and immersed in their respective cylinder contained distilled water for 24hours. After the sample was dried by pressing both side of the composite with tissue paper and re-weight (W_2). The % weight loss/weight gained of the composite was evaluated as reported by Venkateshwaran *et al.* (2011) using equation 1.

$$\% \text{ weight} = \frac{w_2 - w_1}{w_1} \times 100\% \quad (1)$$

Where W_1 = initial weight, w_2 = final weigh of the sample.

Chemical Resistance Test

The chemical test of the composite was conducted using acid and base (H₂SO₄, HCl and KOH). The acid and base used was 10% in 90ml of water. In accordance with ASTM standard D - 570. The composite sample was tested using each chemical, the average value was evaluated, composite was pre-weight (w_1) and dipped into the respective cylinders at room temperature for 24 hours. After sample has been washed with distilled water and dried by pressing with side of the sample with tissue paper and re-weighted immediately as (w_2).The weight gain/ loss of the samples was similarly calculated from equation 1.

CHARACTERIZATION OF SAMPLES

Tensile Strength Test

Samples of the composite were tested for their tensile strength using an Instron Universal Testing Machine, IX, Model 4302, Instron, A crosshead speed of 5mm per minute was used. An ASTM D-638 standard was adopted for this purpose (Gang *et al.*, 2018).

Flexural Strength Test

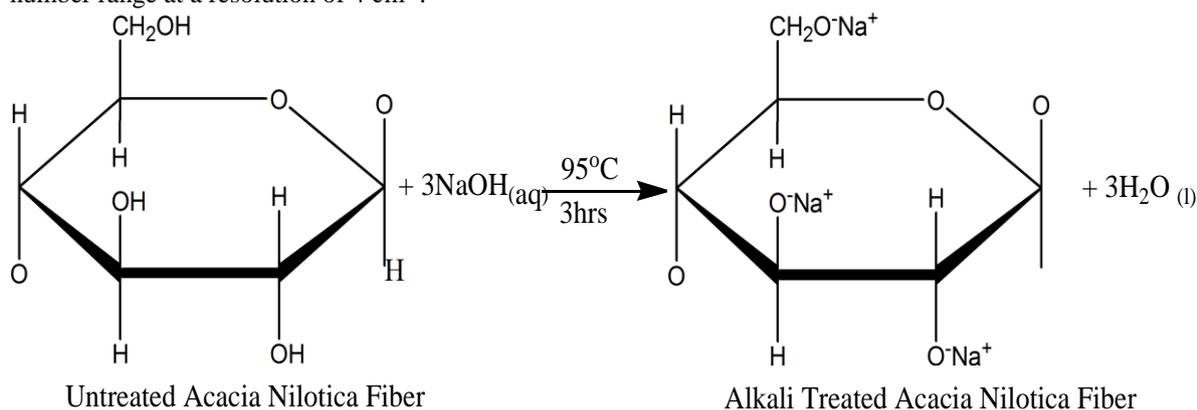
An Instron IX 4302 machine was also used for the flexural tests, using the 3-point bending fixture according to ASTM D-790. Across-head speed of 5 mm per minute was used. The dimension of test samples was typically 60 x 6 x 2 mm. The average results were obtained from 3 specimens that were tested. The value of flexural strength (σ) was calculated according to equation 2.

$$\sigma = \frac{3FL}{2dh^3} \quad (2)$$

In equation 2, F is the maximum load recorded (N), L is the sample span (mm), d is the sample diameter (mm), and h is the width (mm). (Gang *et al.*, 2018; Bai *et al.*, 1999).

FTIR Analysis

Fourier Transform Infrared spectroscopic (FTIR) analysis was performed on the *Acacia nilotica* natural fiber using an Agilent infrared spectrophotometer. The unmodified and modified fibers were analyzed over a 650-4000 cm^{-1} wave number range at a resolution of 4 cm^{-1} .



Scheme 1: Alkali treatment of *A. nilotica* fiber

Activation of hydroxyl groups and at the same time cleaning of the surface of the cellulosic fiber was achieved as a result of the alkali treatment (Scheme 1). Removing some percentage of adhesive pectin, hemicelluloses, lignin, waxy epidermal tissue, and oil wrapping, led to reduced fiber diameter and a corresponding increase in aspect ratio. This also increases the operative fiber surface area for excellent adhesion with the matrix (Xue *et al.*, 2007). As reported by (Yakubu *et al.*, 2010), the treatments led to improved surface roughening, hence encouraging mechanical bonding. It can be seen from the result in Figures 1 and 2 that the

RESULTS AND DISCUSSION

The chemical treatment of the fibre was aimed at enhancing the surface of the fibre by revealing more reactive groups. This further facilitated the smoothness of coupling between the fiber and the polymer resin. Hence, the resulting material would have improved mechanical properties. *A. nilotica* fiber allowed the formation of molecular cellulose. This is expected to make reactive groups more readily available on the surface of the fiber. As such, interfacial properties can be enhanced, while the mechanical properties of the natural fiber-reinforced polymer composite with better performance will be produced. (Maya and Anandjiwala, 2008). The alkali treatment of the *A. nilotica* natural fibers gave rise to the development of cellulose-O⁻Na⁺ groups in the material. The H-bonding existing in the network structure is broken while new reactive H-bonds are formed between the cellular, molecular chains, and the natural crystalline structure of the cellulose relaxed. Hence, the addition of aqueous NaOH to the *A. nilotica* natural fiber stimulated the ionization of the hydroxyl group to the alkoxide (Scheme 1). Washing of the fiber later permitted a reaction of Oxygen with H₂O to create more hydroxyl groups on the surface of the fiber. This created hydrogen bond and also allowed a neutral pH to be attained (Montenegro *et al.*, 2019; Caia *et al.*, 2012).

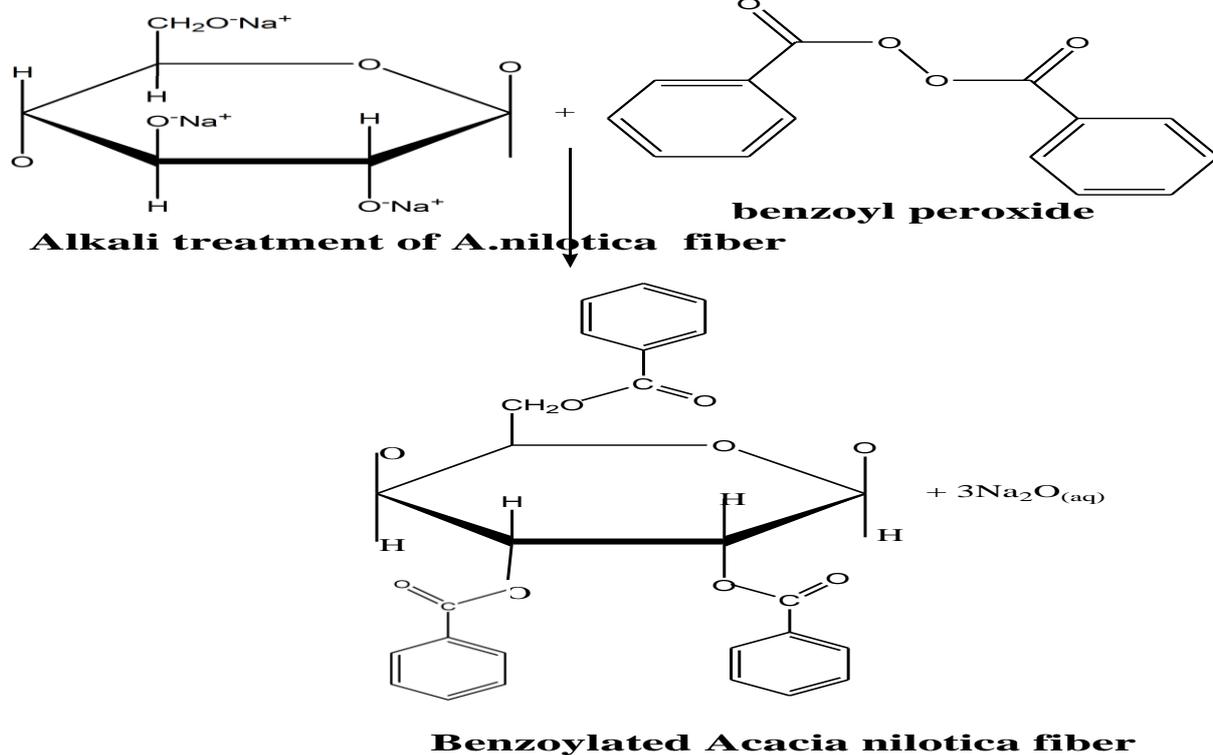
alkali treatment of *A. nilotica*/epoxy and *A. nilotica*/glass fiber/epoxy composites showed improved values of mechanical properties (tensile/flexural) by comparing those with the untreated *A. nilotica*/epoxy composites.

Peroxidation reactions using benzoyl peroxide tend to breakdown easily to form free radicals of the form RO•; RO• with subsequent reactions occurring with the hydrogen group of the cellulose fibers to form cellulose radical. The peroxide initiated free-radical reaction is shown in equation 3 (Sumit *et al.*, 2018; Paul *et al.*, 1997; Joseph, *et al.*, 1996).



This treatment reduces moisture absorption tendency of the fiber and improves dimensional stability, it was reported that treatment with benzoyl peroxide led to higher mechanical

properties of composites Fu *et al.*, 2018; Lingtong *et al.*, 2018 and Wang *et al.*, 2007. It was shown already in respect to Figures 1 and 2 that peroxide treated *A. nilotica*/epoxy composite (Scheme 2) showed increased mechanical properties (Tensile/Flexural) when compared with that of untreated *A. nilotica*/epoxy composites.



Scheme 2: Benzoyl peroxide treatment of *A. nilotica* fiber

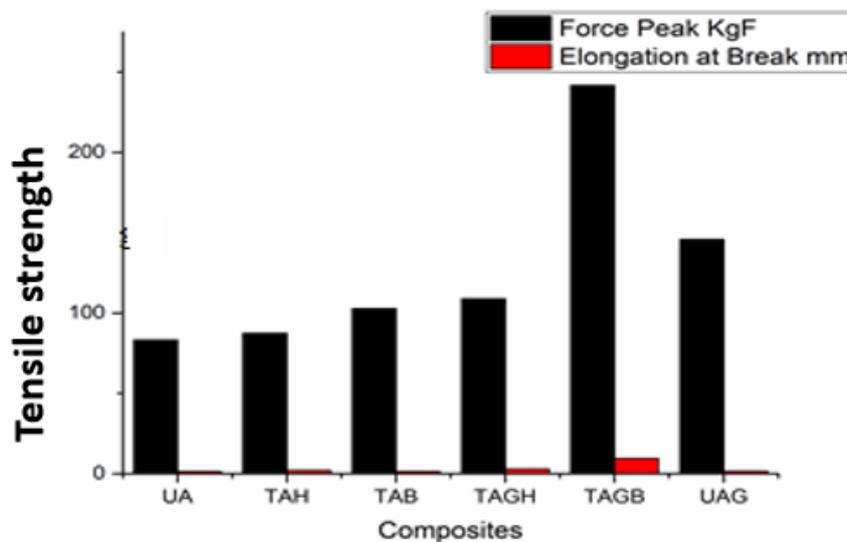


Figure 1: Tensile strength of the samples.

Key: UN - Untreated Acacia fiber; TAH - Treated Acacia with H₂O₂
 TAB- Treated Acacia with (C₆H₅CO)₂; TAGH- Treated Acacia + Glass fiber with H₂O₂
 TAGB- Treated Acacia + Glass fiber with (C₆H₅CO)₂
 UAG - Untreated Acacia and glass fiber

The tensile strength results show that the fiber treated with glass has the highest tensile strength. The fiber morphology also influences the tensile strength of the natural fiber. The fiber with the smooth surface is expected to have good tensile strength, while the fiber with the rough surface finish is expected to possess less strength. The mechanical properties of *A nilotica* composites

reinforced by glass fiber in polyester matrices have been investigated by many researchers. It was shown that the addition of glass fiber into composite resulted in maximum tensile strength. In the same way, it was observed that the *A. nilotica* mixture composites are capable of having a high impact strength (Vimalanathan, *et al* 2017).

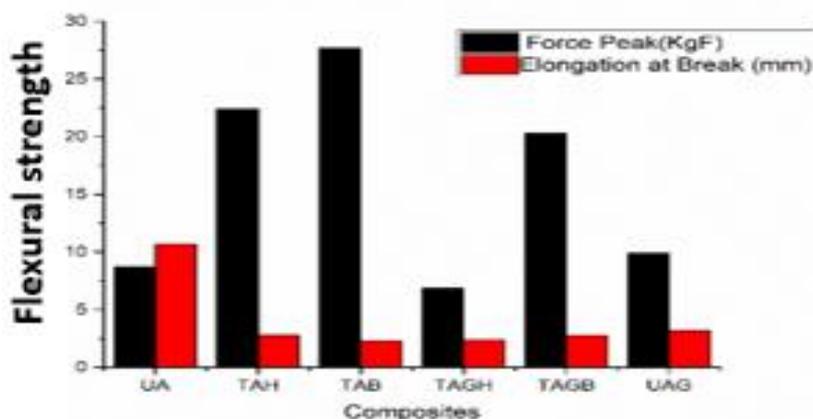


Figure 2: Flexural strength of fabricated composite

Table 1: FTIR Peaks Assignment

S/No	Blend composites	Spectral bands (cm ⁻¹)	Spectral bands found	Tentative bond.
1	TABP	3200-3400 2800-2860 1640-1810	3341, 3319 2853 1657	OH Alcohol (stretch) C-H Alkane (stretch) C=O Ketone (stretch)
2	TAHP	3200- 3400 2800-2860	3350, 3385 2851	OH Alcohol (stretch) C-H Alkane (stretch)
3	TANH	3200- 3400 1000-1300	3321, 3330 1160	OH Alcohol (stretch) C-O Carbonyl (stretch)
4	UAF	3200-3400 2800-2900	3332 2883	OH Alcohol (stretch) C-H Alkane (stretch)

The data from the FTIR experimental analysis given in Table 1 shows the presence of chemical components and chemical bonds in the natural fibers. Molecular orientation is one of the essential parameters, affecting the physical properties of the molecular systems. FTIR is commonly used to determine the composition of natural fibers. FTIR is also used to study the chemical treatment of

natural fibers. The spectra data provides information useful to interpret the effect of chemical treatment on natural fibers (Dadrasi *et al.*, 2019). FTIR analysis was carried on the composites and the degraded blends. The FTIR results confirmed the degradability of the polymer (cellulose) by showing chemical changes in terms of appearance and shortening of peaks. (Abubakar *et al.*, 2014).

Table 2: Moisture Absorption Test

S/no	Composite	Initial weight (g)	Final weight (g)	% change in weight (gain)
1	UA	14.36	14.61	1.74
2	TAH	15.98	16.11	0.81
3	TAB	16.32	16.42	0.6
4	TAGH	19.02	19.7	0.26
5	TAGB	19.1	19.07	0.15
6	UAG	15.17	15.16	0.59

The natural fiber-reinforced composite materials are hygroscopic in nature, thus the composites are considerably affected by the water. The natural fibers absorb water and bulge. Thus the fiber resin interface is damaged, lead to a loss of the

mechanical strength. The water is absorbed by the natural fiber thus this leading to breaking of natural fiber into fibrils. Thus, the fiber strength decreased considerably. However, upon modification of the fiber through composite formation, its resistant to water is enhanced, as shown in Table 2.

Table 3: Chemical Resistance

Acids (10%)		Sulphuric Acid (H ₂ SO ₄)					
S/No	Composite in (g)	Initial weight	Final weight	% change in weight gain	Initial weight	Final weight	% change in weight
1	UA	18.35	18.38	0.16	18.45	18.51	0.16
2	TAH	17.13	17.16	0.17	17.11	17.13	0.11
3	TAB	17.07	17.09	0.11	16.75	16.79	0.23
4	TAGH	15.13	15.18	0.25	14.98	15.12	0.93
5	TAGB	20.21	20.29	0.38	19.99	20.10	0.50
6	UAG	16.56	16.57	0.06	16.28	16.38	0.61

The chemical resistance result in Table 3 show that the treated glass fiber composite had higher resistance to the chemicals treatment. The reinforcing element of the natural fiber is cellulose being contained in a matrix of hemicelluloses and lignin. The failure of the fiber takes place when the matrix cohesion is lost, with the reinforcing fibrils and existing hydrogen bonding in the cellulose also affected. Hence, the lesser the cellulose content, the lower the tensile strength (Tumas, 2003).

CONCLUSIONS

This work shows that successful fabrication of an *A. nilotica* fiber reinforced epoxy composites can be undertaken by a simple hand lay-up technique. The medication of the surface of the material is important in order to decrease the hydrophilicity of *A. nilotica* fibers and to improve the contact force with the lignin matrix, which is hydrophobic in nature. The alkalization, benzolization, and peroxide surface treatments used in this work, modifying the cellulose component of the fiber, have had effects on the mechanical properties of the resulting composites. Those were successfully analyzed using FTIR where the modification was evident. The results indicate that

tensile strength and flexural strength for the treated fiber materials can be determined. The results show that the treated blend composites had a high force peak of 241.7 Kg f with a corresponding elongation at break value of 9.39mm.

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