

**EFFECT OF FIBER SURFACE MODIFICATION ON THE MECHANICAL
PROPERTIES OF RICE HUSK/GLASS FIBER REINFORCEMENT EPOXY RESIN
HYBRID COMPOSITE**

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ABSTRACT

Rice husk was manually removed. A part of rice husk fiber was soaked in alkaline 6 percent, potassium permanganate 0.125 percent, and benzoyl peroxide 6 percent, respectively. A portion of glass fiber was used for enhancing the mechanical properties of the hybrid composite. Epoxy resin and hardener were also used being 60% and 40% respectively in composition, to fabricate the composite, For this work the mechanical properties of this synthetic composite was calculated using ASTM specifications for tensile and flexural strengths in material manufacturing. The result show that the processed hybrid blend of benzoyl peroxide rice husk / glass fiber (RH / GFHC) has better mechanical properties than untreated, alkaline and potassium permanganate treated sugarcane fiber composite. Tensile and flexural strengths increased by 27% and 48% respectively, Study of the FT-IR revealed that chemical alteration was done by reducing the hemicellulose and lignin content utilizing rice fiber benzoyl therapy. In comparison, the synthetic composite displayed acid, alkali tolerance and also had lower water absorption. The composites of rice husk fiber composite/glass fiber hybrid have the properties that advise their relevance for use in the engineering industries.

Key words: Epoxy resin, Glass fibre, Hybrid composite, Rice Husk.

INTRODUCTION

Production of polymer composites in 19th century introduced a new era of study with a new option of using natural fibers as part of more diverse [1]. A composite material is a ' material structure ' consisting of a mixture of two or more micro or macro constituents, which vary in shape and chemical composition where the components are basically insoluble in each other [2-3]. Such interesting and beautiful structures were born out of advances in the field of materials science and technology. A composite material can provide superior and special mechanical and physical properties because it incorporates the most desirable properties of its constituents while at the same time eliminating their least desirable properties [4]. Ironically, all of these benefits make it possible for composite materials to be commonly used in the aerospace and automotive industries [5]. Recently there has been a growing interest in using natural fibers as composite reinforcements [6-8]. 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 [9]. Because of their strength and stiffness composites are important components, yet they can be very light in weight. Strength-to-weight ratios and weight-to-weight ratios can therefore be many times stronger than steel or aluminum. Combinations with unachievable properties can also be accomplished with plastics, ceramics, or polymers alone [10]. This research work dealt with the hybrid effect of composite made of rice husk / glass fibers manufactured using hand lay-up method, with the use of Epoxy resin and hardener being 60:40 in composition, respectively. In the case of synthetic polymers this is particularly common because of the desirable properties of the resultant products. Epoxy resin, for example, is one of the polymer matrices that undergo such modification due to its high strength, high stiffness, chemical consistency and low cost [11-12]. In this study, a composite material strengthened with epoxy resin will be made using natural fibers derived from rice husk and reinforced with glass fibre. The effect of alkali, potassium permanganate and benzoyl peroxide treatments of rice husk fiber on the mechanical structure and functional groups was studied using Fourier Transform Infrared Spectroscopy (FT-IR). Changes in the physico-mechanical properties of the fibre-reinforced material will be studied. The natural fiber was exposed to chemical testing before product processing in order to enhance its surface chemistry.

MATERIALS AND METHODS

The epoxy resin Bis (phenol-A-diglycidylether), crosslinker HY-951 and glass fiber were obtained from Nycil Nigeria Ltd. The resin was used as a matrix agent with a classification LM-556 and a density of $1.3 \pm 0.2 \text{ g / cm}^3$.

Fiber was refined from the rice husk using water retting technique. Because the rice husk was already small in size, it was submerged for seven days in water at room temperature. After that the water on the fiber had pectin, lignin and other impurities. For gummy materials it developed an unwanted odour. The fiber looked new and milky in a networked sheath. Wastewater was then washed multiple times with the tap water and allowed to dry under shade for three days [6, 13]. The dry fiber was sieved resulting in a fine powder, the ready substrate for treatment and composite manufacture.

Chemical Treatment of Rice Husk

Alkali Treatment of Rice Husk Fiber

Sodium hydroxide (NaOH) is one of the approaches used to enhance linking of the fibre-matrix interface. This procedure extracts wax, oil coating, tiny quantities of lignin and the silicon content of the rice husk fiber cell wall's exterior surface [1].

Potassium Permanganate KMnO_4 and Acetone Treatment

The treatment of rice husk with potassium permanganate and acetone improves compatibility, better fiber/matrix adhesion, higher fiber/matrix interaction and good wetting. [14].

Hydrogen Peroxide Treatment (H_2O_2)

Hydrogen Peroxide treatment utilized oxidative delignification to detach and solubilize the lignin and loosen the lignocellulose matrix. Temperature and concentration have a continuous effect on the percent lignin reduction during hydrogen peroxide pretreatment. [1].

Alkali Treatment Procedure

- NaOH solution was prepared using 6% sodium hydroxide pellet and 94ml of water.
- Rice husk fiber was dipped into the solution.
- Then it was kept in an oven for 3 hours at 95°C
- It was stirred after every 30 minutes while in the oven.

Potassium Permanganate and Acetone Treatment Procedure

- 0.125% of Potassium Permanganate $KMnO_4$ was dissolved into 90ml of water and 6% of acetone was added to the solution.
- 15g of alkali treated rice husk fiber was dipped inside the solution and kept for 30 minutes.
- After 30 minutes, the fiber was sieved and dried under shade for 7 days at room temperature.

Hydrogen Peroxide Treatment Procedure

- Hydrogen Peroxide at concentration of 20% was used to preheat 15g of rice husk fiber.
- Treatment was performed at 75°C with continuous stirring until the fiber was bleached.
- After that it was sieved and dried under shade for 24hrs.

Fabrication Tables and Preparation of Composite Specimen

Table 1: Composite Fabrication

S/N	Material	Fabrication
1.	Epoxy resin	60%
2.	Hardener	40%
3.	Fiber (Rice Husk)	1g
4.	Glass fiber	0.5g
5.	Releasing Agent	Blue Seal
6.	Curing Temp.	2hrs at room temp.

Preparation of Composite Specimen

For the preparation of this composite the hand lay-up method was used. Thus, the epoxy resin and hardener of (respectively 60:40) is cautiously combined with gentle stirring to eliminate air bubbles. 1 g of each treated and untreated rice husk fiber was used, and epoxy resin and hardener was measured, A mold release agent was added at the mold's inner surface for quick and easy removal of the composite sheets. A thin layer of the matrix mixture was sprayed after pressing the mold onto a glass tray. In the same process, 0.5 g of treated rice husk fiber and 0.5 g of glass fiber were also made. On the mixture, the required amount (1.0 g) of fibre was then spread. Then the residual mixture was poured into the pipe. Sonication mechanism was used to prevent air bubbles from developing. The mold was then permitted to cure for two hours at room temperature after which the samples were taken out of the mold and kept in for additional research.

Water Absorption Test

Analysis of water absorption is done in compliance with standard test procedure ASTM D 570-98 [15] for water absorption of plastics. The sample weights were collected and then immersed in normal water. The samples were taken out of the humid atmosphere after 24 h and all surface moisture was collected with the aid of a clean dry cloth. The composite samples were dried in an oven at 65°C, then measured before being immersed in distilled water at room temperature. The weight gain was measured for the absorption of liquids. The percentage weight gain of the samples was calculated at regular time intervals. The % weight loss/weight gained of the composite was evaluated using equation 1 [16].

$$\% \text{ weight gain/loss} = \frac{w_2 - w_1}{w_1} \times 100\% \quad \dots\dots\dots \text{Equation 1}$$

Where W_1 = Initial weight of the sample

W_2 = Final weight of the sample

Chemical Resistance Test

The composite chemical test was conducted utilizing acid and base (H_2SO_4 , HCl and KOH). The acid and base used were 10 percent in 90ml of water. According to ASTM specification D-570 [17]. The composite sample was measured using each component. The average value was assessed. The composite was pre-weighed (W_1) and submerged in the corresponding tubes at room temperature for 24 h. After the sample was cleaned with distilled water and dried by pressing with tissue paper side of the instrument and then re-weighed as (W_2). The sample weight gain / loss was determined from equation 1.

$$\% \text{ weight gain/loss} = \frac{w_2 - w_1}{w_1} \times 100\%$$

Where W_1 = Initial Weight of the sample; W_2 = final weight of the sample

Fourier Transform Infrared Spectrophotometric Analysis

The Fourier Transformed Infrared (Cary 630 FITR, Agilent Technologies, Japan) was used. Spectra of treated and untreated (rice husk fibre) and its hybrid were conducted within the range of 4,000-650 cm^{-1} .

Tensile Strength Test

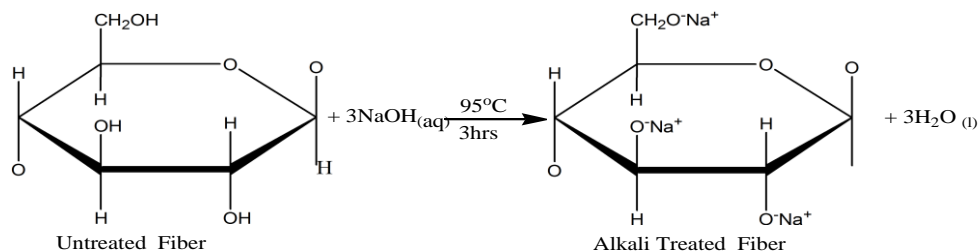
Tensile Resistance of a Dumbbell-shaped specimen of scale 150 mm x 27 mm x 4 mm was conducted in compliance with ASTM D 3039-00 [18] using a cross head speed of 10mm / min Testometric materials measuring machines.

Flexural Test

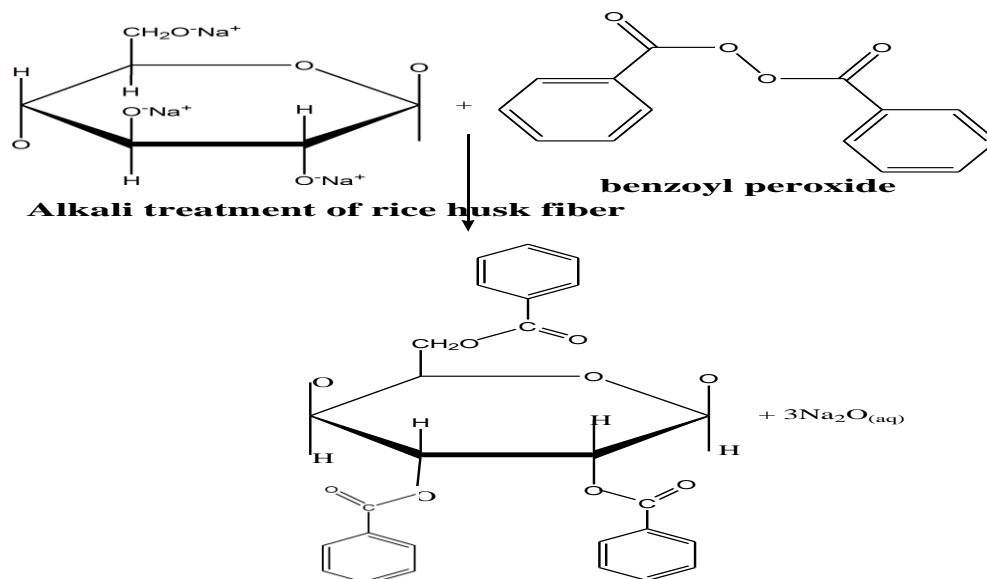
An Instron IX 4302 unit, using the 3-point bending feature according to ASTM D-790 [19], was also used for flexural checks. Across-head speed was used at 5 mm per minute. The test sample dimensions were typically 60x 6x 2 mm. The composite findings were collected from 3 research specimens. The value of the flexural force (π) was calculated in accordance with equation 2 $=3FL/(2dh^3)$.

RESULT AND DISCUSSION

The goal of the chemical fiber modification was to improve the fiber surface by exposing more sensitive groups. The smooth coupling between the fiber and the polymer resin was further facilitated. Consequently, the resulting substance would boost mechanical properties of rice husk fibre, this is expected to make dynamic groups on the surface of the fibre more readily available. As such, interfacial properties can be improved, although better performance would improve the mechanical properties of the natural fiber-reinforced polymer composite [16]. Adding aqueous NaOH into rice husk natural fiber stimulated alcoxide ionization in the hydroxyl group (Scheme 1). Later, fiber washing allowed Oxygen to react with H₂O to produce further hydroxyl groups on the fiber surface. This produced hydrogen bond and also allowed the attainment of a neutral pH [4, 6, 16]. The alkali treatment of the natural fibers of rice husk resulted in the development of cellulose-O⁻Na⁺ groups in the tissue. The H-bonding in the network structure is disrupted when new reactive H-bonds are established between the molecular and relaxed cellulose's normal crystalline structure.

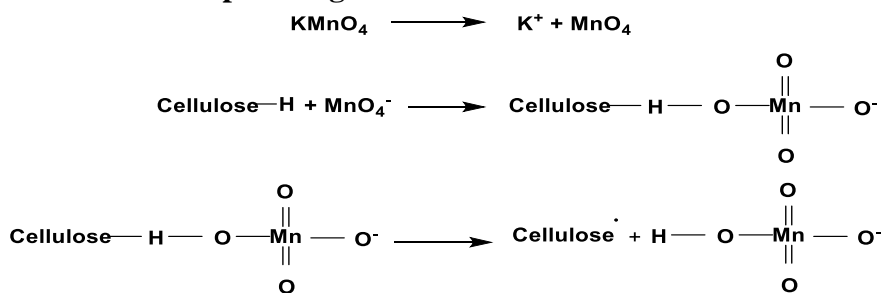


Scheme 1: Alkali treatment of Rice Husk Fibre

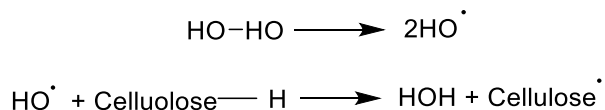


Scheme 2: Benzoylated rice husk fiber

Potassium permanganate Treatment on Ricehuskfibre



Scheme 3: Reaction of Ricehuskfibre with Potassium permanganate
Hydrogen Peroxide Treatment on Rice Husk Fibre



Scheme 4: Reaction of Rice Husk fiber with Hydrogen peroxide

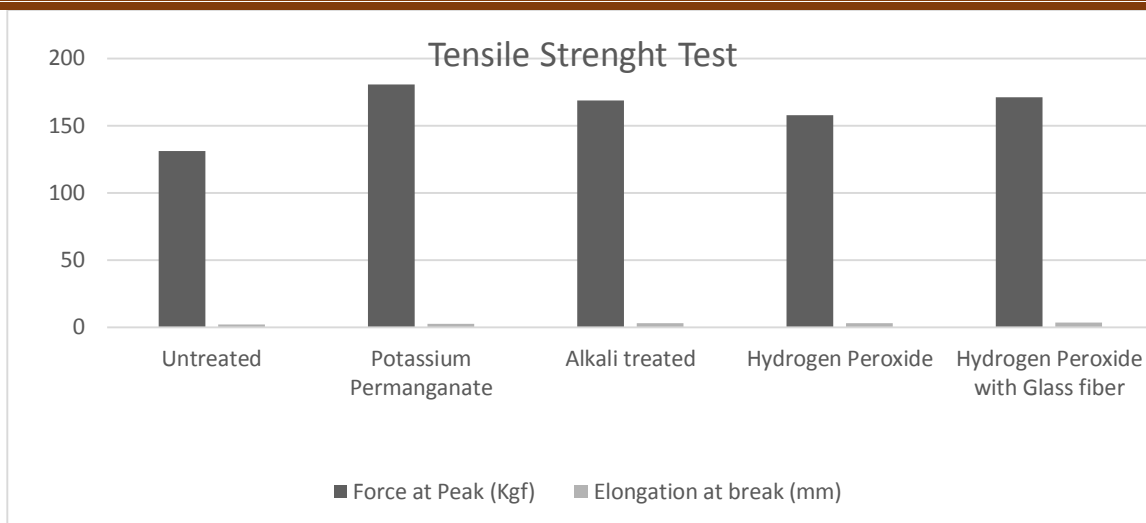


Figure 1: Tensile strength of the treated samples.

Tensile Properties

The result obtained as shown in the Figure 1, indicates that the treated fiber of Potassium Permanganate composite and hydrogen peroxide with glass fiber composite had the highest strength at 180.6kgF and 171.15kgF. This shows that Potassium Permanganate and hydrogen peroxide with glass fiber improved the tensile strength of the untreated fiber composite which recorded 131.1kgF. It shows Potassium Permanganate's ability to alter the fiber. Hydrogen peroxide treated fiber composite, fiber composite treated with hydrogen peroxide, and glass fiber treated composite peroxide reported values of 168.5kgF, 157.65kgF and 171.15kgF respectively[20-21].

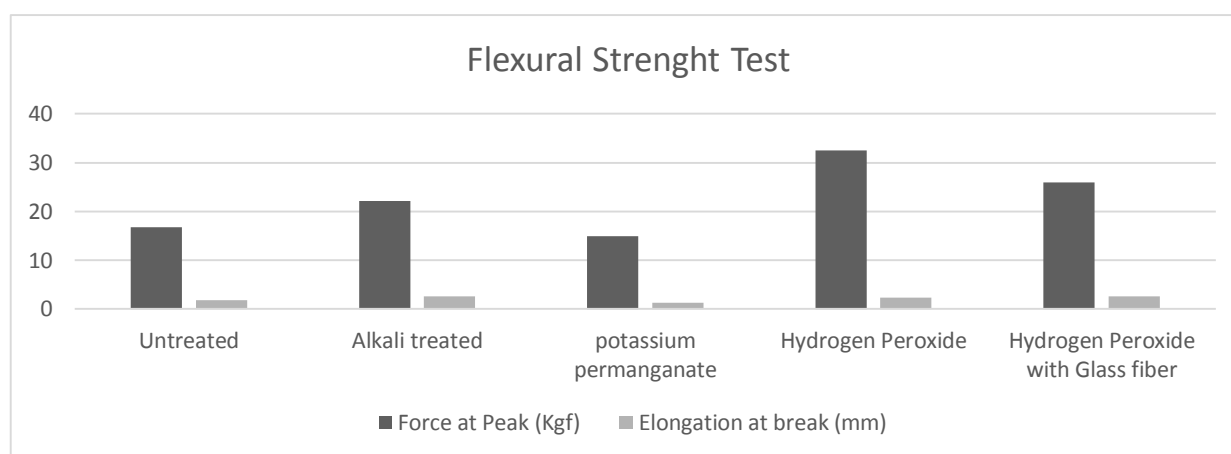


Figure 2: Flexural Strength of the samples

Flexural properties

The outcome of the flexural check (Fig. 2) indicates that hydrogen peroxide treated with 32.5kgF and 26.0 glass fiber composite Rice Husk and hydrogen peroxide treated with 32.5kgF and 26.0 respectively have a greater tendency to tolerate higher stress than other fiber composites treated just before yield stage. That is, it is more prone to endure any significant deformity, cracking or fracturing for a long time during use.

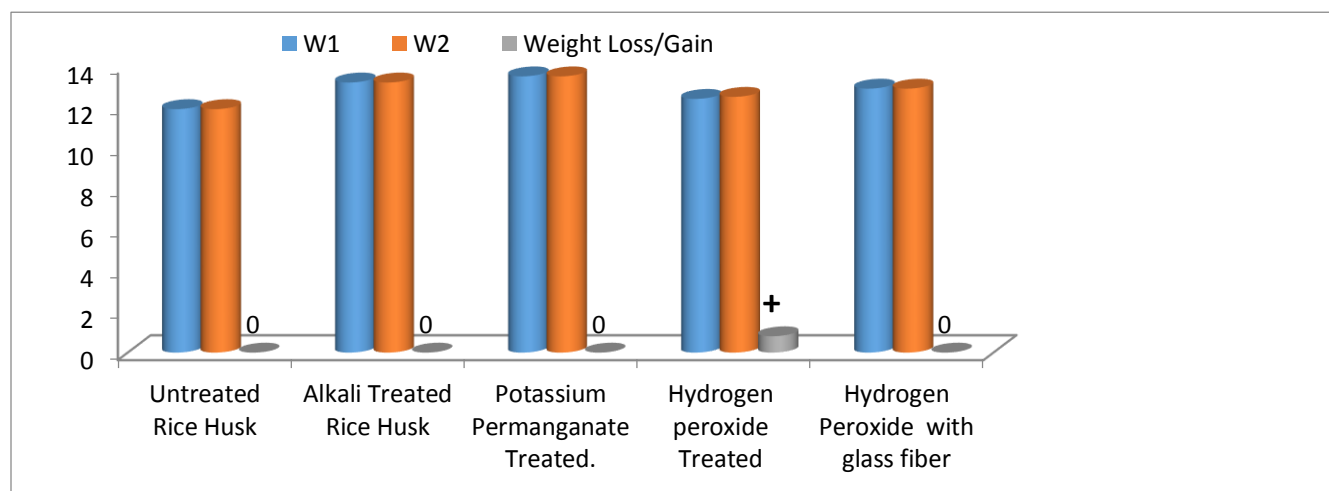


Figure 3: Result of water absorption test

The result at the Figure 3 indicates that treated hydrogen peroxide rice husk composite can retain more moisture relative to untreated and treated fibers such as potassium permanganate, glass fiber hydrogen peroxide and treated alkali. This is because the lipophilic material is removed from the fiber as such, this will consume more humidity. This is because treated hydrogen peroxide fiber and epoxy are not in the same phase (hydrophilic and hydrophobic), therefore there is incompatibility between them, resulting in the ability of the composite to absorb water.

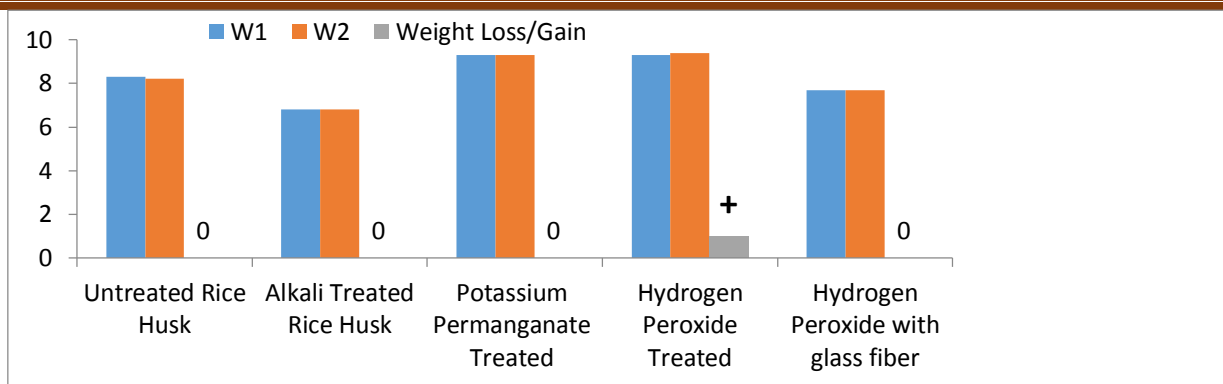


Figure 4: Result of chemical resistance test using hydrochloric acid for various samples of composite

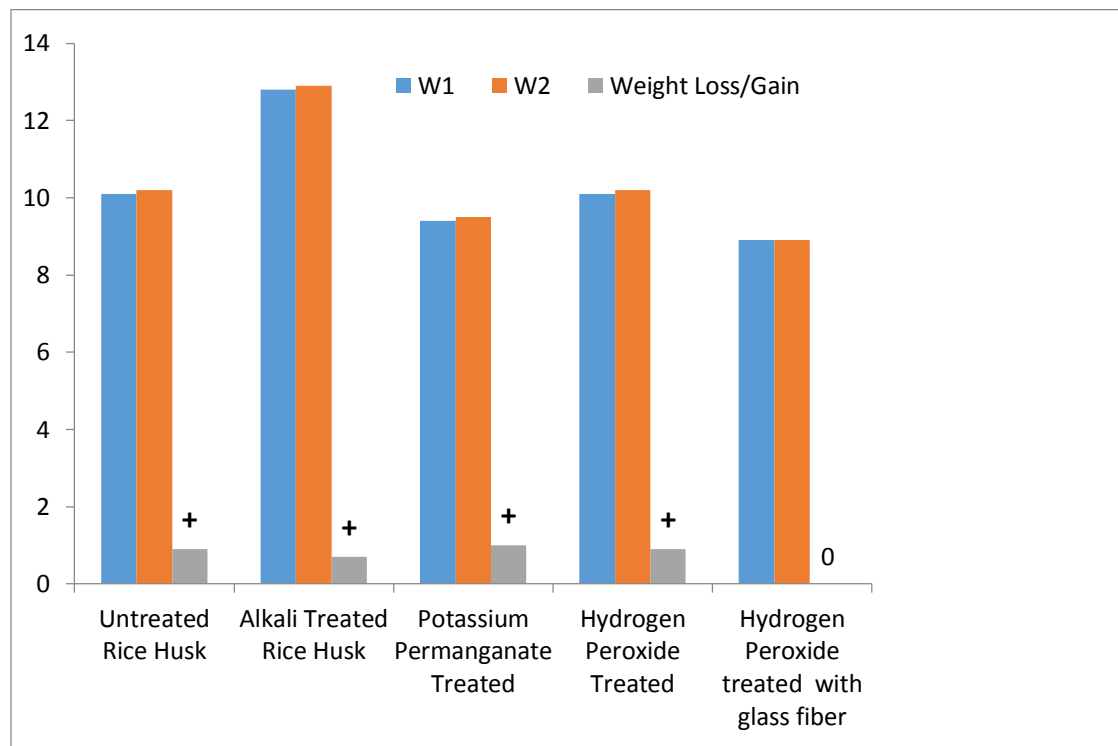


Figure. 5: Result of chemical resistance test using sulphuric acid for various samples of composite

The result in Fig. 4 indicates that hydrogen peroxide treated rice husk composite can react with hydrochloric acid whereas untreated rice husk composite, composite-treated alkali, composite-treated potassium permanganate and glass-fiber hydrogen peroxide may be prone to chemical

resistance due to the composite's hydrophobic existence. Although rice husk composite treated with hydrogen peroxide is hydrophilic, it could not absorb hydrochloric acid for this purpose.

The result for Figure 5 above indicates that the hydrogen peroxide treated with glass fiber reinforcement was able to withstand sulphuric acid. This is because the hydrogen peroxide fiber treated with glass fiber is hydrophobic, while the fiber hybrid treated with alkali, untreated, potassium permanganate and hydrogen peroxide was able to absorb sulphuric acid. This is because neither the fiber nor the epoxy was in the same phase.

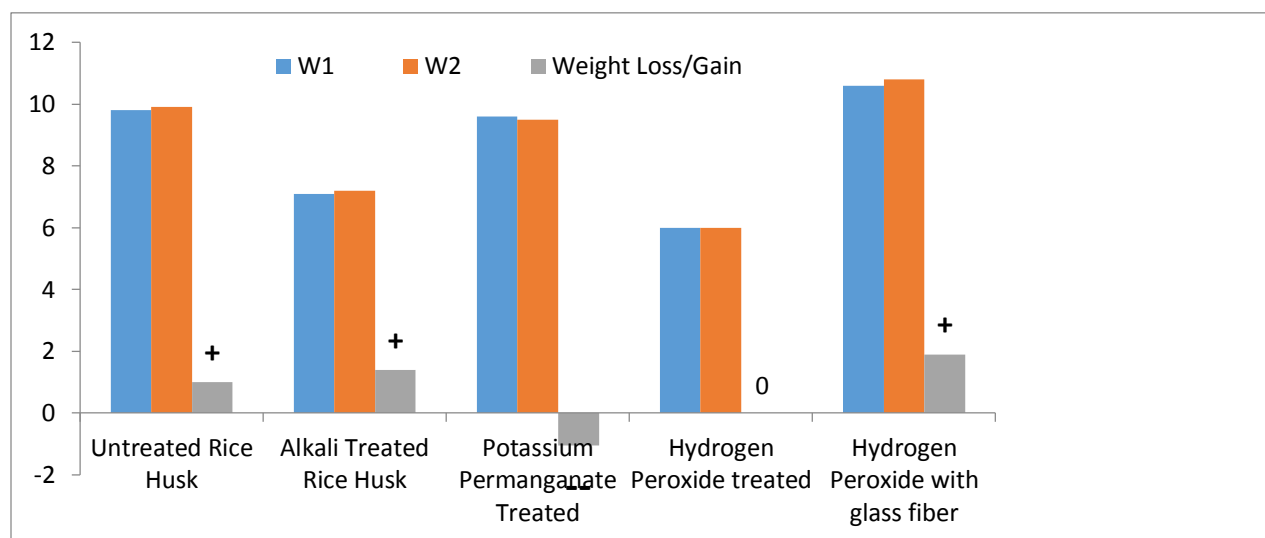


Figure 6: Result of chemical resistance test using potassium hydroxide for various samples of composite

The result obtained above is Fig. 6 It shows that composite-treated hydrogen peroxide cannot be absorbed into potassium hydroxide because it is hydrophobic while others such as (untreated composite, composite-treated alkali and glass-fibre-composite hydrogen peroxide could absorb potassium hydroxide. Figure 6 above shows untreated, alkali treated, potassium permanganate hydrogen peroxide treated and treated hydrogen peroxide with glass fiber tensile test results of composite material with different tests.

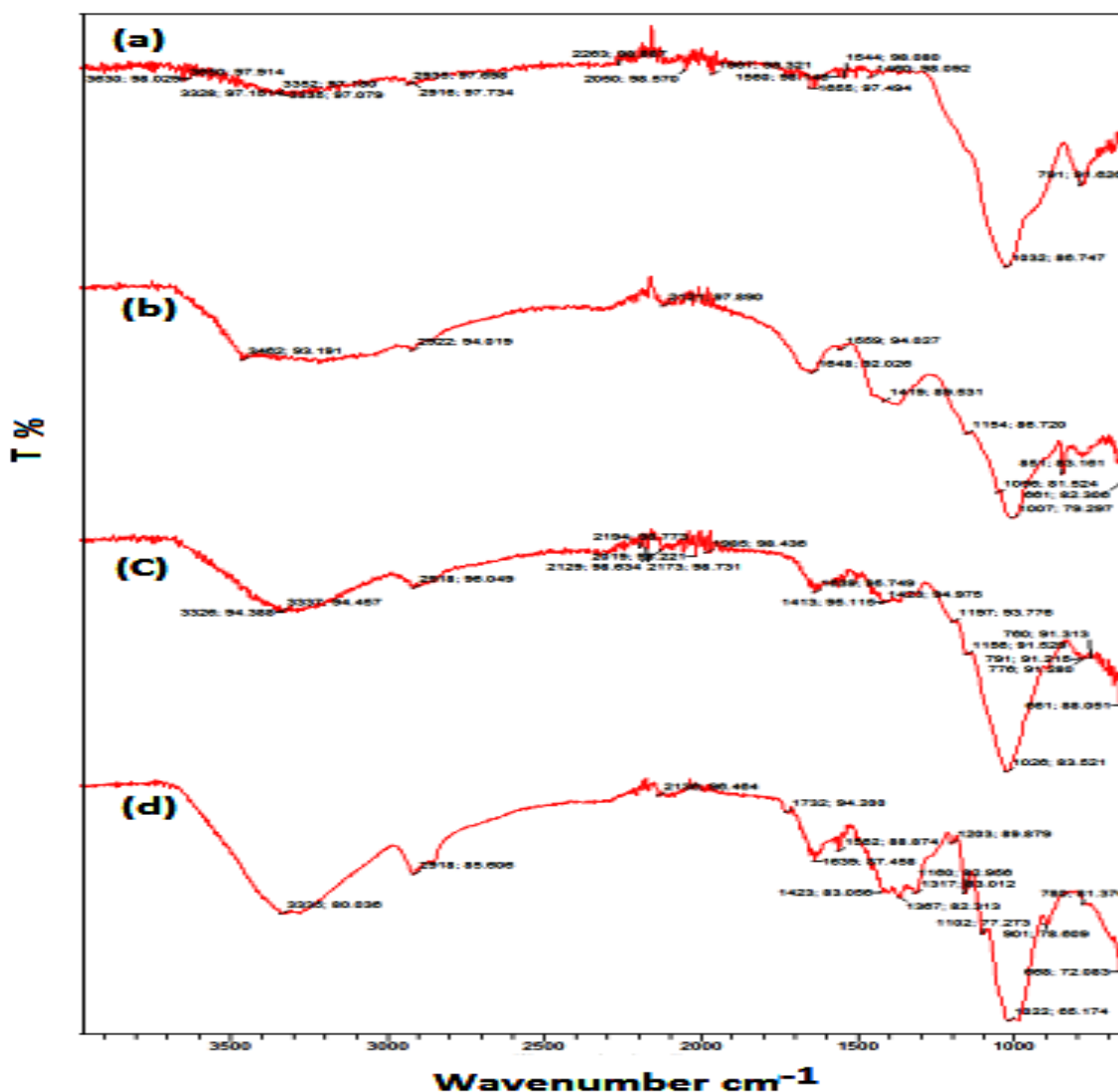


Figure7: FTIR for (a) untreated Rice Husk (b) alkali treated Rice Husk (c) hydrogen peroxide treated Rice Husk (d) potassium permanganate treated Rice husk

Table 2: FTIR spectra of untreated and treated Rice Husk fiber

Rice Husk fiber	Wave number cm^{-1}	Functional group assigned
Untreated fiber	3630 -3328	O-H primary alcohol
	2935 -2916	C-H Aliphatic
	1655	C=C Aromatic
	1032	C-O for ether
	791	C=C bending for alkene
Alkali treated	3462	O-H primary alcohol
	2922	C-H Aliphatic
	1640	C=C Aromatic
	1007	C-O for ether
	661	C=C bending for alkene

Hydrogen peroxide treated	3326	O-H primary alcohol
	2918	C-H Aliphatic
	1639	C=C Aromatic
	1026	C-O for ether
	661	C=C bending for alkene
potassium permanganate	3335	O-H primary alcohol
	2918	C-H Aliphatic
	1639	C=C Aromatic
	1022	C-O for ether
	668	C=C bending for alkene

The result in Table 2 showed the FTIR spectral peaks for both treated and untreated rice husk fibers. All the studied fibers have O-H stretching vibrations in the range of 3630-3326 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 1032-1007 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 7.

CONCLUSION

For use as reinforcement in natural fiber composite, potassium permanganate treated fiber composite seems promising, as it is apparent from the results obtained in the water absorption hydrochloric acid and tensile tests conducted. The test showed that potassium permanganate composite has strong indentation resistance and other significant deformations that can reveal the composite when usage. The tensile and flexural tests also suggest that bonding intensity is quite high compared to other handled and untreated composites, which is why the connectivity and stability of the fiber / matrix has increased.

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