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Hygral Analysis and Mechanical Strength Evaluation of TiO$_2$ Nanoparticle-enhanced Natural Fiber Reinforced Epoxy Composites

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ABSTRACT

The primary focus of this investigation is the hydraulic, mechanical and boundary stability of continuous nanocomposites with epoxy and natural fiber integrated with TiO2 nanoparticles. Continuous natural fiber nanoparticles that had been alkali-treated and those that had not were produced by cutting the natural fiber culm. The nanofibers' tensile properties, density, and equivalent diameter were measured by experimental evaluations. Composites with a volume proportion of 42% fiber were made using the Resin transfer technique (RTM) and reinforced with epoxy (NNF/EP). While it was noted that natural fiber's power was diminished when treated with an alkaline solution, epoxy nanocomposites made from alkali-treated natural fiber showed better tensile strength than those made from untreated natural fiber. The research also looked at how the tensile strengths of the epoxy and natural nanocomposites were affected by the size of the natural fibers. As the diameter of the natural fiber reduced, the findings showed that the composite's tensile strength and Young's modulus also fell. The mechanical characteristics were negatively affected by moisture; NNF/EP laminates showed a significant vulnerability to moisture absorption in hydrothermal aging experiments. This work has future scope so it may be patented in the future.

Keywords: Natural nanofiber; Alkali treatment; TiO$_2$; Bamboo fiber; Mechanical properties.

1. INTRODUCTION

The most used composite material is fiber-reinforced polymer (FRP). The literature claims that the 1940s saw the industry start producing glass fiber-reinforced composites in large quantities. Developing carbon fiber-reinforced composites will be crucial to the aerospace and defense industries. Environmental consciousness has grown in recent years, and at the same time, the supply of fossil fuels is steadily running out (Bronzino, 2000). There is an issue with environmental contamination because synthetic fibers that are used frequently, including carbon and glass fiber, do not degrade quickly in the natural surroundings (Malathi *et al.* 2023). As a result, there has been much concentration on creating green composites and biodegradable materials. The number of natural fibers used in composites in the 2010s was roughly 315,000 tons or 13.5% of the entire reinforcing substance used; by 2020s, it is predicted to rise to around 830,000 tons (Yan *et al.* 2014; Hobson and Carus, 2011). Bananas, sisal, cotton, natural fiber, and wood are the commonly used plant fibers (Jordan and Chester, 2017; Sood *et al.* 2018; Alomayri and Low, 2013; Zakikhani *et al.* 2014; Migneault *et al.* 2015). The benefits of these plant fibers include low density, strong mechanical and thermal insulation, affordability, sustainability and biodegradability (Van Voorn *et al.* 2001; Liu *et al.* 2012; Wollerdorfer and Bader, 1998). Natural fiber has outstanding mechanical qualities and can grow up to a few centimeters every day (Okubo *et al.* 2009). There are several ways to remove natural fiber, including saturation, water vapor detonation, alkali presentation and baking (Rao and Rao, 2007; Okubo *et al.* 2004; Kim *et al.* 2013; Biswas *et al.* 2013). All extraction techniques will directly impact the strength and quality of the fibers. A natural honeycomb fiber-reinforced composite material is a natural fiber.

Cells of the parenchyma surround the vascular bundles. Vessels, phloem, and many fibers make up the vascular bundle. The lignified wall of parenchyma cells is made up of lignin, cellulose and hemicellulose. Fiber and parenchyma make up about 40% and 50% of the entire natural fiber stem, respectively (Chen *et al.* 2018). The vascular bundle's cell structure of fiber features a tiny cavity in the center, which is encircled by a numerous-layered structure made of an internal primary wall and an external secondary wall.

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These cell walls are made of pentose sugars, polysaccharides, cellulose, and hemicellulose. Outside of the primary wall, there is an additional intermediary sheet between the fibers. Between vascular bundles, there is an intermediary sheet outside the main wall. With a level of almost 90%, lignin is the primary component of this layer (Banik *et al*. 2017). Zou *et al.* (2009) (Hao *et al.* 2018) investigated the unit composition and mechanical characteristics of natural fibers by using SEM, AFM and Nano-depression. The research revealed that natural fibers are roughly cobblestone-like in form and range in size from 21 to 198 nm. The hardness and elastic modulus measured for natural fiber with a microscopic-grain structure are 0.440.09 GPa and 10.41.8 GPa, respectively. In the plant cell wall, cellulose, lignin and hemicellulose account for roughly 95% (Hao et al. 2018). Despite having the ability to bind fibers together, lignin is a complicated substance that has firm connection cohesion with polymer resin (Hemnath *et al.* 2021; Kalia *et al.* 2009; Anbunathan *et al.* 2019). The experimental findings demonstrated that successful treatment of the surface (Sukmawan *et al*. 2016; Zhang *et al.* 2012) produced an interface that was superior to those acquired without the alkaline conduct (Muniappan *et al.* 2020; Khan *et al.* 2017; Sabari *et al.* 2024; Ramu *et al.* 2022). Composites are frequently revealed to be wet while being used. In this work, the mechanical, hydraulic, and contact strengths of stable natural fiber-reinforced epoxy laminates were examined. Resin transfer molding (RTM) was used to create composites with natural fiber-reinforced epoxy (NF/EP). In addition, the hydrothermal aging test was carried out and the magnitude response of natural fiber on the tensile characteristics of NF/Epoxy laminates was investigated (Sabari and Muniappan, 2024; *Dhakal et al.* 2007; Alamri and Low, 2012).

2. EXPERIMENT ANALYSIS

2.1 Production of Continual Natural Nanofiber

Fig. 1: Bamboo reinforced with TiO_2 Nano fillers and polyester matrix

The bamboo fibers (BFs) used in this research were obtained with great assistance from a natural fiber business firm, based at Vellore, Tamil Nadu, India (Danková *et al.* 2019). Utmost care was exercised in the preparation of the bamboo fibers to ensure very high quality. A gentle wash was given using clean water to remove any dirt. Then, the fibers were allowed to air-dry for two days while being exposed to sunlight (Robles *et al.* 2015). Sodium hydroxide (NaOH) solution was used to soak the fibers for four hours after they were dried. The treatment improved the surface characteristics of the fiber and helped to eliminate any remaining contaminants; then, to remove any remaining NaOH solution, the fibers were washed in clean water and then heated at 75 °C to make a structured fabric.

The alignment and arrangement of the fibers were ensured throughout the heating process, which contributed to the composite materials' overall strength and integrity. A matrix made of epoxy and titanium oxide (TiO_2) was used for this experiment. Naga Pharmaceutical Manufacturing, Chennai, Tamil Nadu, India, supplied the components. The matrix and the TiO_2 fillers greatly enhanced the structural support and improved the mechanical characteristics of the composite material. Research shows that the composite material was developed by integrating natural bamboo fibers with a TiO_2-epoxy matrix; Fig. 1 shows the composition of the reinforcing fibers, fillers and matrix used in the study. Reinforcing composite buildings using bamboo fibers is a promising idea because of their unique qualities. Fibers made of bamboo, which include cellulose in a range of 68.42 to 71.25%, have Young's modulus of 35 to 45 GPa and tensile strength of 615 to 862 MPa.

In comparison to other natural fibers, bamboo is very lightweight due to its low density of 1.41 g/cm^3. Their overall structural stability is enhanced by the presence of hemicellulose, which ranges from 25.10 to 32.47%, and lignin, which accounts for 17.25% of their composition. Despite being quite strong, bamboo fibers only extend around 1.3% to 1.7%. Composites made of bamboo fibers and epoxy resin, which have a lower tensile strength (between 29.5 and 31.25 MPa) but a higher Young's modulus (3.1 GPa), have improved mechanical properties and are thus useful in many contexts where strength, durability and light weight are of paramount importance.

2.2 Alkaline Treatment

Natural fiber was given an alkaline treatment by boiling it in a sodium hydroxide solution with an absorption of 0.1 N for 12 h at 100 °C. Then, distilled water was used to wash alkali-treated natural nanofiber (ALK-NNF), which was then put in an oven to dry out for eight hours at 80 °Celsius. To determine the comparable density and diameter of ALK-NNF, the mean volume of one natural fiber was also determined.

2.3 Synthesis of TiO_2 Nanoparticles

Nanoparticles of titanium dioxide are created using the Sol-gel method that has been tweaked from its initial use in molecularly imprinted titanium synthesis. The procedure begins with a 40 ml solution of 100% ethanol and 5 ml of deionized water being added to a 100 ml round-bottom flask with a septum, condenser and a magnetic bar. Before adding $TiCl_4$ (0.6 ml, 0.05 M) *via* the septum, the solution is slowly heated. The mixture is stirred continuously at 60 °C for 1 h. Then, the diluted NH_4OH solution (0.05 M) was gradually injected *via* the septum until the pH reaches 9. Then, the mixture was allowed to remain at 60 °C with constant magnetic stirring throughout the night, followed by vacuum heating at 100 °C to obtain white TiO_2 powder. Thermal and chemical treatments were used on this white powder to make it easier to synthesize crystalline or functional TiO_2 nanoparticles. The synthesis of TiO_2 nanoparticles using the Sol-gel technique is schematically illustrated in Fig. 2.

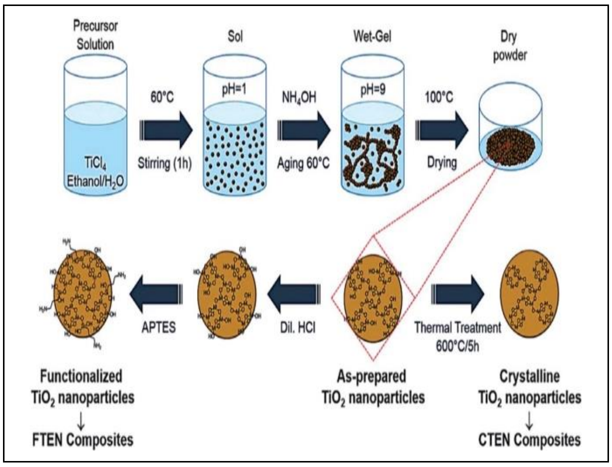

Fig. 2: Schematic illustration of the synthesis of TiO_2

2.3 NNF/EP Composites

Resin transfer molding was used to create epoxy composites reinforced with natural fiber. Before RTM, the resin was implanted in a degasification apparatus to remove blend air for 6 h. Though the cavity was seated on a heated press, the pressure cylinder was linked to the controlled high-pressure nitrogen to drive the resin into the chamber. Following the injection procedure, the curing process took place at 140 °C for two h. The fiber volume fraction of the final NNF/EP and ALK-NNF/EP nanocomposites was around 42%. Since the fibers were manually positioned, they were not exactly aligned, and those material-like natural were therefore regarded as unidirectional quasi-composites. The rule-of-mixture formula could be used to calculate the composite's theoretical density.

2.4 Nanofiber Testing for Tensile Analysis

The natural fiber and alk-natural fiber were adhered to a piece of paper with cohesion before the tensile test, as illustrated in Fig. 3. Fig. 4 shows the longitudinal strength of laminates of the micromechanical model (Llopiz-Guerra *et al.* 2024). Then, samples were dried for five hours at 80 °C to fix the epoxy cohesive and dry the fiber. A micro-stretcher was then used to determine the tensile strength of each fiber.

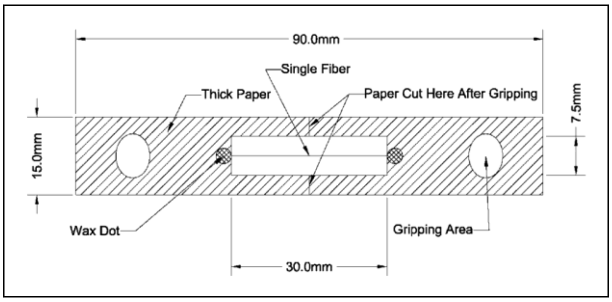

Fig. 3: Single fiber for tensile specimen

2.5 Experimental Test of NNF/EP Composites (Tensile and Bending)

A UTM apparatus with a 250 kN magnitude was used to conduct tensile and bending tests on composite materials. Flexural strength was evaluated using three-point bending tests with a 100 mm bending span. For the tensile or bending test, the NNF/EP composite was sliced into a clear pattern with 160121.84 or 160101.84 mm, respectively. To protect the specimen from the clamp, end tabs are added to both ends of tensile test specimens. To remove moisture before testing, each specimen was heated to 80 °C in the oven for 8 h. Bending and tensile tests were performed on laminates. The long-fiber direction of Young's modulus can be accurately determined for unidirectional composites using the mixture rule, which is expressed by following the equation (Alomayri and Low, 2013).

$$F_c = F_f \, V_f + F_m \, V_m \qquad (1)$$

where, V_f is the fiber fraction volume, V_m is the matrix fraction volume, and F_c and F_f are the Young's moduli of the laminate and the fiber.

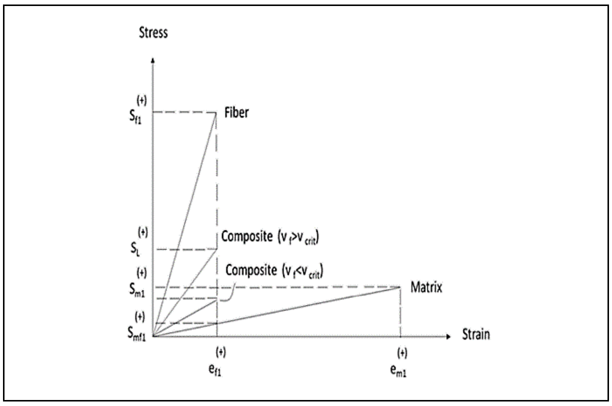

Fig. 4: The longitudinal strength of laminates of the micromechanical model

If the fiber failure strain is lesser than matrix failure strain, it is possible to estimate the lengthwise tensile strength of the prolonged fiber described by Kelly and Davies (Sabari *et al.* 2024) for the investigation of composite strength.

2.6 I_{ss} (Shear Stress) Measurement

The interfacing shear tension between natural fiber and resin is made of epoxy, as shown in Fig. 5 (a); the natural fiber is first inserted into the uncured resin to the proper depth. Fig. 5 (b) depicts a homemade pullout test stand utilized for the measurement. The sample was a wheel handle at a speed of around 0.15 mm per min. The reading of the loading force during the tugging was made at the same time using a force sensor. The lone fiber was removed from the resin after it had the pullout load, and the epoxy resin was recorded (Greszczuk, 1969).

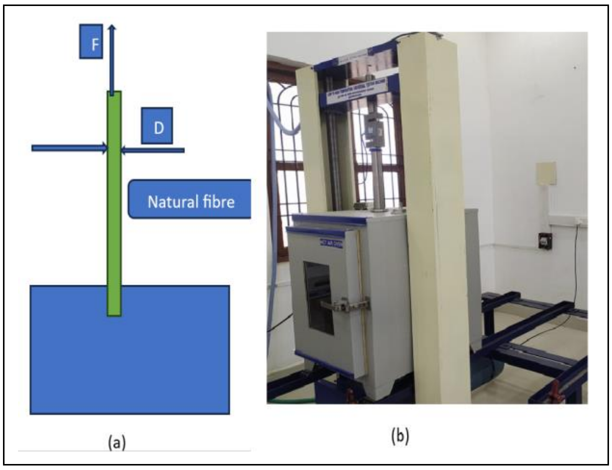

Fig. 5: Single fiber sample setup pullout stand

2.7 Influence of the NNF Diameter on Strength of ALKALINE-NNF Laminates

This investigation was aimed to determine how the diameter of the natural fiber affected the strength of ALKALINE-NNF laminates. Sliced multiple sectors of natural fiber, each with a dimension of 165 mm and a cross-sectional area of roughly 0.8 mm x 0.6 mm, 0.6 mm x 0.7 mm, and 0.5 mm x 0.5 mm, appropriately. The natural fiber was given an alkali treatment. The mean volume of ALK-NF was studied to determine the comparable diameter. Then, RTM was used to create samples of ALK-NNF/EP composite. The fiber volume ratio for every specimen is 42%. The specimens were then put through tensile testing to determine strength.

2.8 Hygrothermal Methods for Aging Investigations

In this experiment, the mechanical properties of NF composites and their ability to absorb moisture were examined. First, samples of the ALK-NNF/EP composite with a volume of fiber percentage of around 42% were made using RTM. Each specimen was dehydrated in an oven to remove any remaining dampness. The composite samples were then submerged at 100 °C for 1, 2, 3 and 4 h. The moisture content, W_c, of the composite was determined using the following equation:

$$W_c = (W_1 - W_0)/W_0 \qquad (2)$$

After hygrothermal aging, the specimens were put through three-point bending and tensile tests to examine how the mechanical characteristics of composites changed in hot and humid environments (Suresha and Ramesha, 2019).

Table 1: Natural fiber characteristics of ALK-NNF with TiO_2

Composites	Density (g/cm^3)	Identical dia. (µm)	Tensile strength measured in (MPa)	Break measured in elongation (%)	Young's modulus (GPa)
NF	0.90	640.0	715.0	2.0	45.0
ALK-NF	1.03	580.0	475.0	2.0	35.0

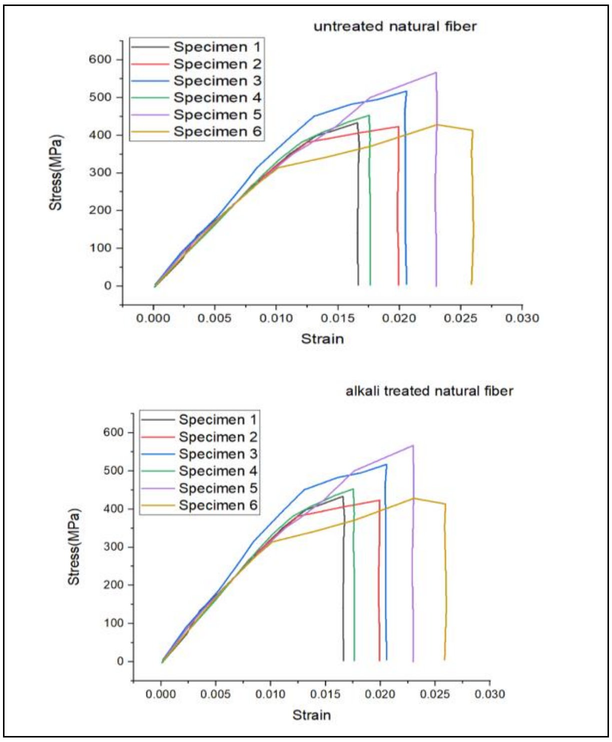

Fig. 6 (a, b): Stress-strain curves for untreated and alkali-treated nanofibers

3. RESULTS AND DISCUSSION

3.1 The Basic Characteristics of the Natural Fiber

Based on the test results, Table 1 lists the fundamental properties of natural fiber. After alkaline treatment, the average BF density increased from 0.9 to 1.2 g/cm^3, whereas the mean corresponding diameter dropped from 643.1 to 583.8 mm. The typical stress and strain curves, based on the tensile tests, of a standalone natural fiber are shown in Fig. 6. Table 1 includes a list of the outcomes as well. The average tensile strength of natural fiber treated with alkali reduced from 715.5 MPa to 472.04 MPa, while Young's modulus decreased from 42.34 to 32.3 GPa. After the alkaline treatment, natural fibers' mechanical characteristics significantly decreased. The ALK-NNF composite, when compared to NF/EP composite, had higher tensile strength because the interfacial shear strength is better.

Coupled-beam Focused Ion Beam microscopic images of the lamina structures of NF and ALK-NNF are shown in Fig. 7 (a and b); the untreated natural fiber shows that the NF maintains the complete vascular collection and the fibers within the vascular collection also maintain the entire polygonal configuration. The ALK-BF structure is depicted in Fig. 7 (c), where the alkali treatment eliminated the arteries, phloem, and parenchyma cells. Fig. 7 (d) demonstrated (in a higher magnification) how alkali caused the lignin between the fibers to disintegrate, leaving gaps between the fibers.

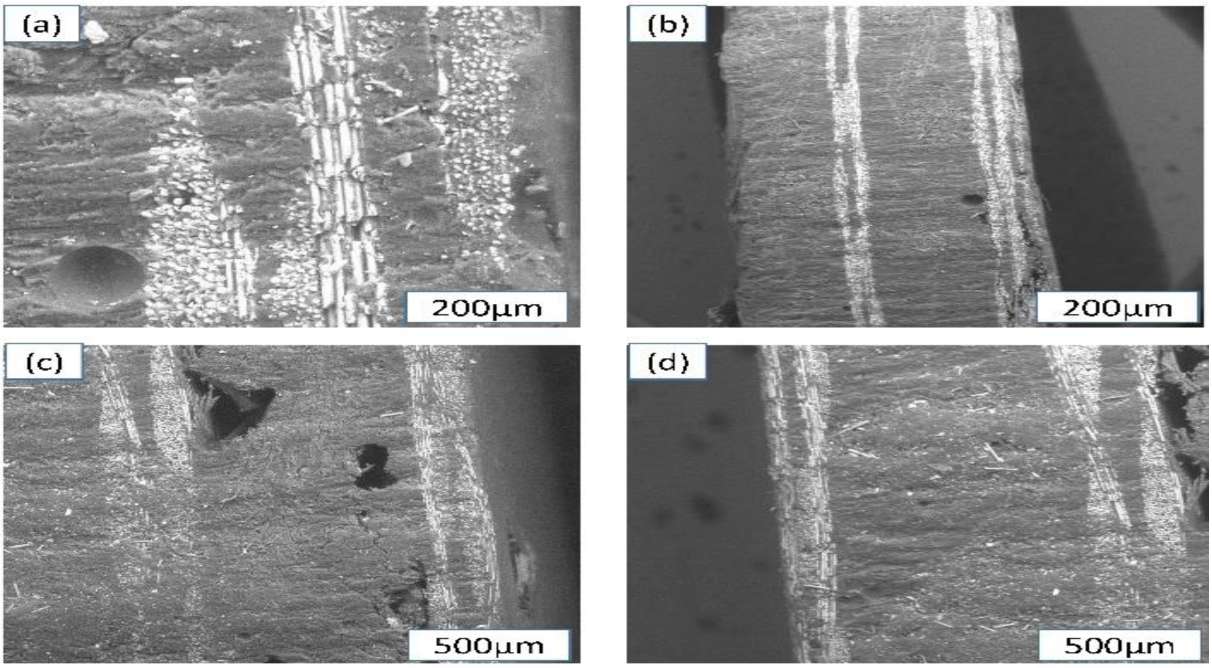

Fig. 7 (a, b, c and d): SEM image of NF and ALK-NNF and coupled-beam focused Ion Beam microscope image

This explains why alkali treatment causes natural fibers to lose some of their tensile strength. Table 2 lists the densities of composites made with natural fibers. Table 2 also provided theoretical estimates of the composite (ALK-NNF/EP) density using the rule of mixture. Theoretical calculations can produce predictions that are reasonable when compared to measured results.

Table 2: Natural fiber densities

Composites	Fiber volume fraction (%)	Theoretical density (g/cm^3)	Density (g/cm^3)
NF/NP Composites	40	1.05	1.10
ALK-NF/EP Composites	40	1.20	1.18

Due to the higher density of the BF following alkaline treatment, compared to NNF/EP composites, ALK-NNF/EP composites have a higher density. Furthermore, the density variation in NF/Epoxy is greater than that of ALK-NF/EP composites because of the high lignin, pectin, and impurity content of the BF. Fig. 8 shows photos of the completed extended unidirectional natural fiber composites for NF/EP.

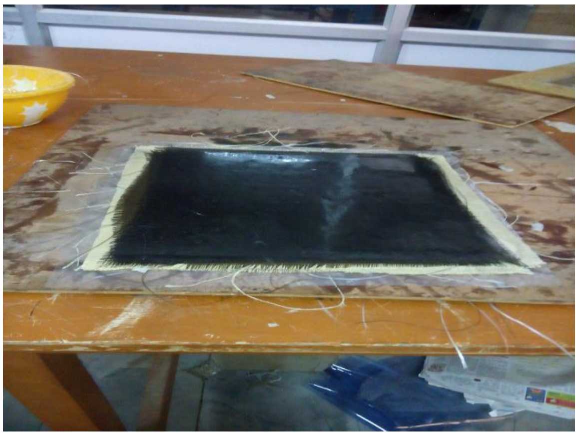

Fig. 8: Completed extended unidirectional TiO_2 with natural fiber composites for NNF/EP

3.2 NNF/EP Composites (3-point Bending Tests)

The modulus and tensile strength of epoxy, NF laminates, and natural composite are displayed in Fig. 9. In the fiber direction, the tensile strength of the AK-NNF laminates increased to 222.7 MPa after BF was alkali-treated, and the young's modulus was raised to 14.1 GPa. The alkali-treated BF components showed improvements in tensile strength and young's modulus by almost 30% and 40%, respectively.

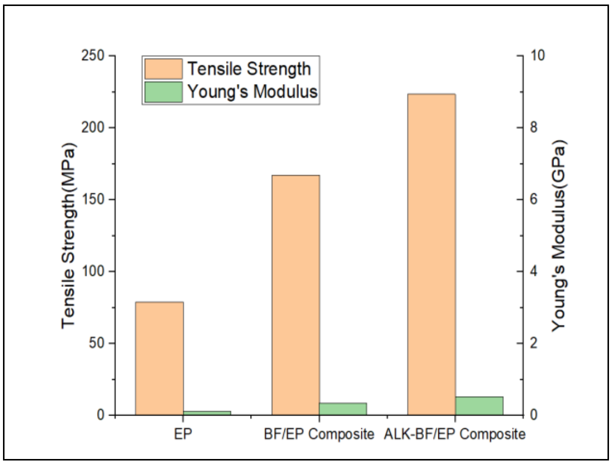

Fig. 9: Tensile strength and young's modulus of both EP and NF

According to the results of a single natural fiber test, ALK-BF's modulus and tensile strength are lower than NF. In contrast, ALK-BF composites are stronger and have a higher modulus than NF/EP composites. All strength and modulus of materials are determined by equations 1 and 2. Theoretical calculations are also possible in the longitudinal direction. Table 3 contains the results (both theoretical and experimental). It was discovered that the experimental values of the natural fiber laminates were more in line with the theoretical values. The experimental values for the NF/EP composite were far below the theoretical expectations, yielding diverse results. It was thought that the epoxy and untreated BF had low contact adhesion. The source of equations 1 and 2 pre-supposed a flawless interfacial between the reinforcement and matrix.

Table 3. Variations (theoretical and experimental)

S. No.	NF/EP COMPOSITES		ALK-NF/EP COMPOSITES	
	Tensile strength (MPa)	Young's modulus (GPa)	Tensile strength (MPa)	Young's modulus (GPa)
Theoretical	320.0	22.0	230.0	16.0
Experimental	170.0	11.0	255.0	15.0

In this case, it is not possible to apply Equations 1 and 2 to the NF/EP composites. This explains the error in the modulus and tensile strength predictions for NNF/EP composites. The specimen's surface fracture after the tensile test is shown in Fig. 10, which was carried out to examine interfacial cohesiveness between wind-natural fiber and adhesive. For NF/EP composites, a fiber pullback conduct is observed over the outward fracture, showing poor interfacial cohesion between the BF and epoxy. Since there was no fiber pullout on the fracture surface of ALK-NF/EP composites, strong interfacial cohesion was inferred. This could account for the improved mechanical qualities of ALK-NF/EP composites.

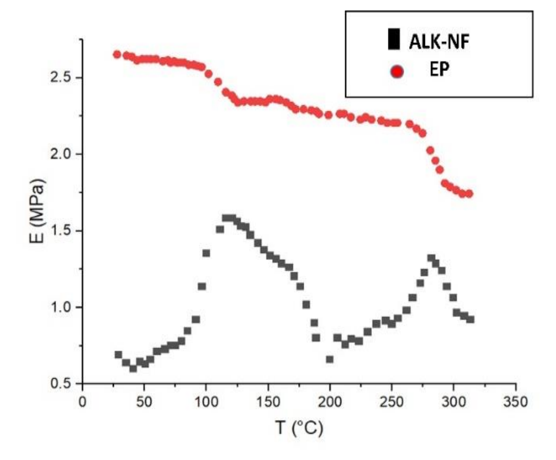

Fig. 10: Interfacial cohesiveness between BF and epoxy for NF/EP composites with temperature

3.3 Fiber Pullout Test

To validate the findings reached from the earlier conversation about the interfacial cohesion between BF and epoxy, a direct fiber pullout test was carried out. Compared to BF that has not been treated, ALK-BF has a stronger interfacial shear strength with epoxy. The intersection cohesion between the laminates and adhesive wax was superior, which led to a higher magnifying effect in composites, as confirmed by the data mentioned above.

3.4 Influence of the Natural Fiber Diameter

Three batches of ALK-BF were produced in examination under identical alkaline circumstances, each with dimensions of 160 mm and a mean identical diameter of 720 m, 580.8 m, and 340 m, individually. After that, RTM also produced ALK-NNF/EP composite specimens. The modulus and tensile strength of ALK-NF/EP composites made with natural fibers of various diameters are displayed in Table 4. The modulus and tensile strength for fibers with diameters of 719.33 m and 583.80 m have somewhat increased to tiny fiber diameter. However, the rising tendency is not as clear because of the high variation. Fig. 9 illustrates the dew osmosis of ALK-NF/EP with respect to temperature and mechanical strength, which are related to the average measured tensile and expansion rates.

Fig. 11 depicts the dew osmosis laminates adhesives for various immersion times in boiling water. For neat epoxy, there is no noticeable moisture absorption.

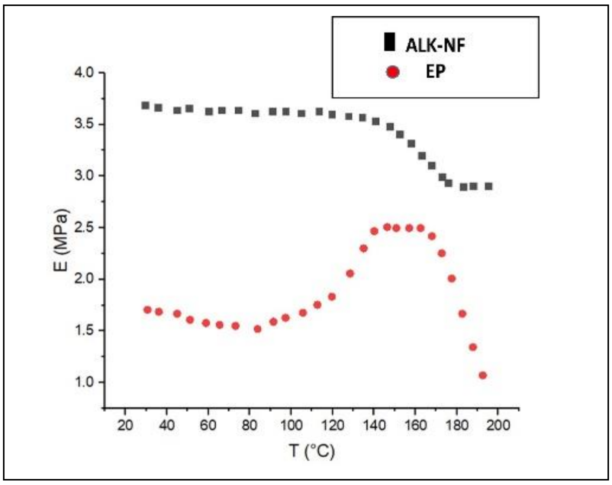

Fig. 11: Precipitation Osmosis of ALK-NF/EP composites and neat epoxy for various immersion times

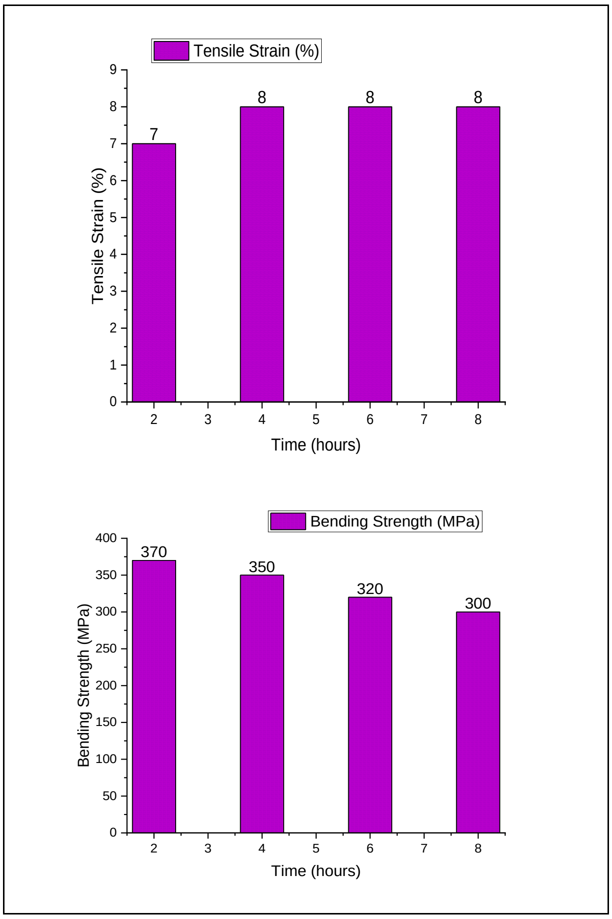

Fig. 12: (a) Tensile test and (b) Bending test

Table 4. Images variations of all results

Average comparable dia. (μm)	Measured tensile strength (MPa)	Measured Young's modulus (GPa)	Expansion (%)
735.00	220.0	11.95	1.86
593.00	218.0	13.25	2.03
412.00	285.0	17.43	2.28

However, the moisture absorption tendency is evident when ALK-BF is added to the composites. With the length of time, the ALK-NF/EP composite is immersed in boiling water, the dew substance quickly rises. The ALK-NF/EP composite's moisture content approaches saturation at about 19% after roughly 4 h. For all laminates after hygrothermal aging, the three-point bending and tensile tests were also carried out. Results from tensile and bending tests are shown in Fig. 12 (a and b). The mechanical strength and all moduli of laminates have been discovered to decrease as moisture content rises. A strength loss of 25% is possible during hygrothermal aging for one hour. As the dew content reaches saturation, the composites might only hold 50% of their initial strength. According to the findings, hygrothermal aging negatively affects the mechanical qualities of natural fiber composites.

4. CONCLUSION

The following inferences were made based on the study:

- The study demonstrates that alkaline-treated natural fibers (ALK-BF) exhibit higher density due to the removal of lignin, hemicellulose, fructose, and contaminants. However, this treatment also results in a reduction in modulus and tensile strength of the fibers. Among various extraction methods, mechanical extraction of bamboo fibers (BF) yields fibers with superior strength.
- When used in composites, continuous BF reinforcement in epoxy resin significantly enhances the reinforcing effect compared to short BF. ALK-NNF/EP composites exhibit higher modulus and tensile strength than untreated NF/EP composites. This trend is also observed in flexural strength, indicating that alkali-treated fibers improve composite performance. Comparisons with literature data show that untreated natural fibers used by Jain achieved similar strength results to this study, while Kushwaha's bi-directional NF/EP composites had lower strength than one-way composites. Banga's studies highlighted decreased strength in short natural laminates.
- Despite a suboptimal interfacial relationship between epoxy resin and BF observed via visual microscopy and pullout tests, alkali treatment can enhance interface cohesion. The study

concludes that reducing the diameter of natural fibers, while maintaining their length, improves the Young's modulus and tensile strength of fiber laminates. However, natural fiber composites are prone to deterioration due to their sensitivity to perspiration and the detrimental effects of hygrothermal aging on their mechanical properties.

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CONFLICT OF INTEREST

The authors declared no conflict of interest in this manuscript regarding publication.

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