Research Article

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Effect of Nanoclay and Surface Treatment on Mechanical Properties of Fish Tail Palm Empty Fruit Bunch Fiber Reinforced Epoxy Composites

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ABSTRACT

The addition of nanofiller into the fiber-reinforced composites aims to enhance adhesion between the fiber surface and the polymer matrix and improve strength. This paper examines the mechanical properties and morphological characteristics of fishtail palm empty fruit bunch (FTPEFB) fiber to evaluate its suitability as a reinforcing material in epoxy composites. The objective of this study was to evaluate the effects of Nanoclay reinforcement and surface treatment of FTPEFB fiber on the performance of fiber-reinforced composites. The FTPEFB fibers were prepared in three forms: untreated, alkali-treated, and benzoyl chloride-treated. Composites were prepared using these fibers with different Nanoclay levels: 0, 2, 4, and 6 wt. % through the hand layup method. Various mechanical characteristics were assessed, including tensile strength, flexural properties, impact resistance, and hardness of the surface. Furthermore, morphology was examined using Scanning electron microscopy to investigate how chemical processing affected the treated and untreated fibers. For alkali-treated FTPEFB fiber-reinforced epoxy composites, the best overall characteristics were achieved with 6 wt. % Nanoclay content, yielding a tensile strength of 77.52 MPa, a tensile modulus of 5.24 GPa, and a flexural strength of 142.8 MPa; the impact strength increased by 23.51%, and the hardness improved by 10.76% when compared to composites without Nanoclay fillers.

Keywords: Surface treatment; Composite; Mechanical properties; Natural fiber; Nanoclay.

1. INTRODUCTION

The demand for advanced materials with improved properties is continuously on the rise, driven by the need to meet evolving requirements and replace existing materials. With the increasing focus on environmental awareness and ecological issues, there has been a significant increase in attention towards using natural fiber-reinforced polymer composites as alternatives to synthetic fibers. Cellulose fibers embedded in polymer matrices have been a prominent research focus for decades. Researchers have explored these material combinations to find suitable replacements for synthetic fibers (Biagiotti *et al*. 2004; Sathishkumar *et al*. 2012). The primary advantages of using cellulose fibers in natural fiber composites include low energy consumption, cost-effective production, biodegradability, abundant availability, high specific strength, and low density (Belouadah *et al*. 2015). Various natural fibers derived from plants such as banana, flax, hemp, okra, sisal, jute, and coir are commonly utilized as reinforcements in polymer-based matrices (Jankauskiene *et al*. 2015; Kestur G. *et al*. 2013; Li *et al*. 2007; Mwaikambo and Ansell 2002). The benefits of natural fibers are diverse, including their biodegradability, sustainability, abundant supply, non-abrasive properties, low cost, and lightweight (Kabir *et al*. 2012). Natural fiber used as reinforcement offer numerous benefits but also come with certain drawbacks. At elevated temperatures, natural fibers are not stable. Their ability to absorb moisture limits their suitability for applications in moist environments (Morvan 1990). This moisture absorption also causes dimensional instability in fiber composites (Thygesen *et al*. 2007). These issues stem from the inherent structure of natural fibers. Cellulose, hemicellulose, pectin, and lignin are the primary components of natural fibers. Due to their abundant hydroxyl groups, these components exhibit a hydrophilic nature, which gives natural fibers a strong affinity for moisture (Mishra *et al*. 2003).

Earlier studies have explored the creation of polymer composites using plant-based fibers as reinforcements. These investigations have demonstrated that it is indeed possible to develop high-performing materials by incorporating environment-friendly reinforcements (Bhagat *et al*. 2014; Mishra and Biswas, 2013; Sapuan *et al*. 2006; Symington *et al*. 2009). Ratna Prasad and Mohana Rao (2011) investigated the mechanical behavior of various natural fiber-reinforced

polyester composites. According to their study, the tensile modulus of the Jowar composite showed notable improvements. Specifically, at a fiber volume fraction of 0.40, the tensile modulus of the Jowar composite exceeded that of bamboo and sisal composites by 11% and 45%, respectively. The effect of fiber content on the mechanical properties of the short basalt fiber reinforced composites examined by (Valadez-Gonzalez *et al*. 1999).

Amuthakkannan *et al*. (2013) revealed that composites with 10 mm fiber length exhibit better properties than other lengths. However, for the composites to function effectively as structural members, these natural fiber-reinforced composites must exhibit high strength under tensile and flexural loading. To enhance the functionality of natural fiber polymer composites, various chemical treatments can be used to improve the compatibility between the fibers and the matrix (Valadez-Gonzalez *et al*. 1999). Alkaline treatment, commonly referred to as mercerization, is a widely employed chemical treatment method used to modify natural fibers for the fabrication of polymer composites. This process effectively modifies the fiber structure by disrupting hydrogen bonds within the network, leading to increased surface roughness. Alkaline treatment effectively removes lignin, wax, and oil deposits that typically coat the external surface of the fiber cell wall (Pickering *et al*. 2016).

In the last decades, numerous chemical treatments have been applied to natural fibers to investigate their effects on the mechanical properties of composites. These studies have revealed new opportunities for further enhancing the performance of natural fiber-reinforced composites. The influence of NaOH concentration and the microstructure of coir fiber-reinforced polymer composites was investigated by (Yan *et al*. 2016), and they observed that coir fibers treated with 5 wt. % NaOH enhanced the flexural strength and tensile strength by 16.7% and 17.8%, respectively. The enhancement in the mechanical characteristics of hemp fiber-reinforced PLA (polylactic acid) bio-composites was reported by (Sawpan *et al*. 2011). At 35 wt. % fiber loading, the alkali-treated (5% NaOH concentration) long-aligned hemp fibers exhibited a maximum impact energy of 7.4 kJ/m² and a tensile strength of 85.4 MPa.

The effect of different chemical treatments on the surface of hemp fibers has affected the mechanical characteristics of the resulting composites (Sankara *et al*. 2019). Their research revealed that composites made from fibers treated with a 4 wt. % NaOH solution demonstrated the best mechanical properties among the various treatments tested.

Conversely, subsequent research revealed that incorporating nanoparticles into matrix composites could address specific limitations in their properties (Sankara *et al*. 2019). The incorporation of Nanoclay into natural fiber composites offers a solution to this shortcoming. By adding Nanoclay, the composite's characteristics are enhanced through improved interfacial adhesion between the matrix and reinforcing components. Moreover, Nanoclay contributes to both the elasticity and stiffness of natural fibers in a single process (Rajeshkumar *et al*. 2021). The impact of Nanoclays as additives in wood-based composite materials was investigated by (Ashori and Nourbakhsh, 2009). The findings revealed that increasing the Nanoclay content from 2 to 6% by weight resulted in improvements to both the flexural modulus and strength of the composite materials. The fiber from the fish tail palm empty fruit bunch (FTPEFB) represents a sustainable resource with minimal production expenses, owing to its widespread availability and abundance, especially in India. As a member of the Areca family, it is distinguished by its unique gray trunk, which can grow to heights of about 15–20 m. This study aims to evaluate the influence of Nanoclay fillers and various surface treatments on the mechanical properties of FTPEFB fiber-reinforced epoxy composites. Specifically, the research analyzes the impact on tensile strength, tensile modulus, impact strength, and hardness. This data is essential for the effective design of composites utilizing this versatile fiber.

2. MATERIALS AND METHODS

2.1 Materials

The raw fish tail palm empty fruit bunches (FTPEFB) were collected from local sources and soaked in water for about 20 h. This soaking process was used to loosen the fibrous material from the stem, making extraction easier and helping to remove dust that could contaminate the fibers due to their exposure to the natural environment. The retting process is carried out to extract FTPEFB fiber material from the stem. The extracted fibers were dried up under the sun for one week, followed by oven drying at 70 °C for 12 h, and then cut to the preferred length. The extraction process of fish tail palm empty fruit bunch fiber is shown in Fig. 1. The epoxy (LY 556) and hardener (HY 951) mixture, supplied by Sree Industrial Composite Products, Hyderabad, India, is used as a matrix to bond with the fibers. The Nanoclay was purchased as reinforcement materials from a fiber source in Chennai, India.



(a) (b) (c)

Fig. 1: Extraction process of fish tail palm empty fruit bunch fiber

2.2 Fiber Surface Treatment

The effectiveness of surface treatments on FTPEFB fiber properties is typically influenced by the concentration and type of the chemical treatment. The present study employed sodium hydroxide and benzoyl chloride to modify the surface of FTPEFB fibers. The process began with an alkali treatment, where the fibers were immersed in a 5% NaOH solution at room temperature for 4 h. Afterward, the fibers were thoroughly rinsed with fresh water to eliminate any residual NaOH. Neutralization was then performed using diluted acetic acid, followed by additional rinsing with distilled water until a neutral pH of 7 was achieved. Finally, the fibers were air-dried at room temperature for 48 h and subsequently oven-dried at 100 °C for 6 h. For the benzoyl chloride treatment, the FTPEFB fibers were immersed in a 10% NaOH solution and then treated with benzoyl chloride under agitation. After 15 minutes, the mixture was filtered, and the fibers were thoroughly washed with water. To remove any residual benzoyl chloride, the fibers were soaked in ethanol for 1 hour, followed by a final rinse with water and drying in an oven at 80 °C for 24 h. The untreated and surface treated fish tail palm empty fruit bunch fibers is shown on Fig. 2.



(a) (b) (c)

Fig. 2: Fish tail palm empty fruit bunch fibers: (a) Untreated, (b) Alkali-treated and (c) Benzoyl-treated

2.3 Fabrication of Composite

The FTPEFB fibers were chopped to a length of 20 mm and subjected to various surface treatments as described earlier. A simple hand layup process was used for fabricating the randomly oriented FTPEFB fiber-reinforced epoxy composite. A wooden mold with dimensions of 250 mm × 250 mm × 4 mm was initially designed for this purpose. A mold-releasing plastic sheet was placed at the bottom of the mold, and semi-solid wax was applied to the plastic sheet to facilitate easy removal of the composite plate. The Nanoclay particles (0, 2, 4, and 6 wt. %) were used as filler material. These particles were incorporated into the epoxy resin before the reinforcement of FTPEFB fibers within the matrix. The matrix was prepared by mixing epoxy and hardener in a 10:1 weight ratio. This mixture was then evenly mixed with the FTPEFB fibers, followed by pressing and rolling with an iron implement to eliminate air bubbles. Pressure was then applied to extract any excess matrix material, and the composite was allowed to be cured at room temperature for 24 h. The same procedure was used repeatedly for all the cases - untreated, alkali-treated, and benzoyl chloride-treated composites. Table 1 shows a detailed composition of the fabricated composites.

Table 1. Composition of the fabricated composites

Sample	Chemical treatment	Nano silica (wt. %)	Total Reinforcement	Matrix (wt. %)
1	Untreated	0	20	80
2		2	22	78
3		4	24	76
4		6	26	74
5	Alkali-treated	0	20	80
6		2	22	78
7		4	24	76
8		6	26	74
9	Benzoyl chloride-treated	0	20	80
10		2	22	78
11		4	24	76
12		6	26	74

2.4 Mechanical Testing of Composite

The composite specimen tensile characteristics were evaluated according to ASTM D3039-76 standards. The samples were cut to 153 mm in length, 12.5 mm in width, and 4 mm in thickness. An electronic tensimeter was employed to perform the tests, with the crosshead moving at a rate of 5 mm/min. To ensure accuracy, three identical specimens were tested for each fiber weight percentage, and the mean value was documented. A three-point bending test was employed to assess flexural strength, following ASTM D790-07 standards. Specimens were prepared with dimensions of 125 mm in length, 12.7 mm in width, and 4 mm in thickness. The experiments were carried out using the same tensimeter, with a constant loading rate of 5 mm/min. The specimen's loading configuration for the flexural test is illustrated in Fig. 3.

Impact energy measurements of the composite specimens were taken using an Izod impact test, following the ASTM D256 procedure. The tests employed a low-velocity Izod impact tester provided by VEEKAY Test Lab, India. Test specimens were fabricated with measurements of 64 mm in length, 12.7 mm in width, and 4 mm in thickness. These samples were then positioned in the testing apparatus and subjected to pendulum impact until fracture or breakage occurred. To evaluate the surface hardness of the composite samples, a Vickers hardness tester was utilized following ASTM D785 test standards. For each type of composite, the value of the mechanical characteristic was determined as the arithmetic mean of at least four different specimens. Scanning electron microscopy (SEM) was employed to examine and compare the microstructural characteristics of the surface fibers before and after treatment using JEOL JSM-6480LV. The fiber surfaces were coated with gold to enhance their conductivity before SEM observation.

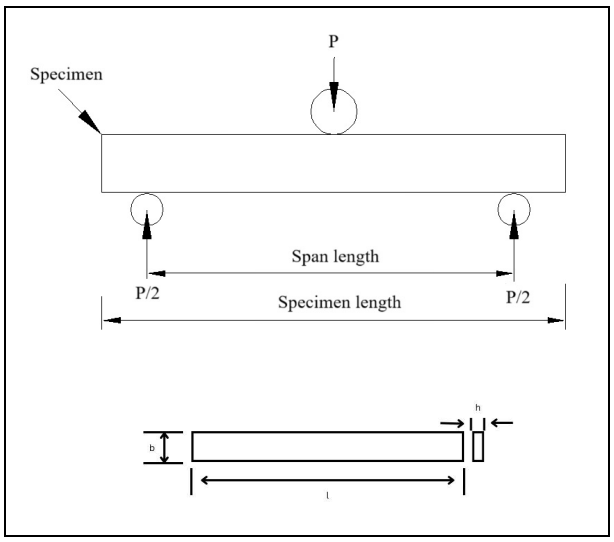


Fig. 3: Loading arrangement of the specimen under Flexural test

3. RESULT AND DISCUSSION

3.1 Tensile Properties of Composites

The effect of fiber surface treatment and Nanoclay content on the tensile properties of FTPEFB fiber-reinforced epoxy composites is illustrated in Figures 4 and 5. The findings suggest that as the percentage of Nanoclay increased, both the tensile strength and modulus of the treated and surface-treated composites improved. However, composites treated with alkaline solutions exhibited greater strength and modules compared to those treated with benzylation. The improvement in the tensile properties of surface-treated specimens was due to the removal of contaminations such as fats, lignin, and pectin from the fiber surface. The tensile characteristics of the composite specimens improved with Nanoclay percentage up to 4 wt. % and then decreased at 6 wt. % of filler content. When the Nanoclay content reaches 4 wt. %, the highest tensile strength values observed were 56.19 MPa for untreated composites, 77.52 MPa for composites subjected to alkaline treatment, and 68.27 MPa for those treated with benzyl. Compared to untreated composites at 4 wt.% of Nanoclay, the tensile strength of alkali-treated specimens improved by 37.96%, and benzyl-treated specimens improved by 21.49%. The tensile modulus of the pure epoxy was found to be 2.96 GPa. At 4 wt. % of Nanoclay, the maximum tensile modulus was found to be 4.31, 5.14, and 4.79 GPa for untreated, alkali- and benzyl-treated composites, respectively. The tensile modulus values were approximately 48.98% higher for untreated composites, 77.02% higher for alkali-treated composites, and 65.2% higher for benzyl-treated composites compared to the neat epoxy matrix. Earlier research on the influence of fiber chemical treatment on the mechanical behavior of composites has shown that treating jute (Saha *et al*. 2010), Agave (Mylsamy and Rajendran, 2011) and Bamboo (Lu *et al*. 2013) fibers with a 6% sodium hydroxide concentration improved the tensile properties of the composites.

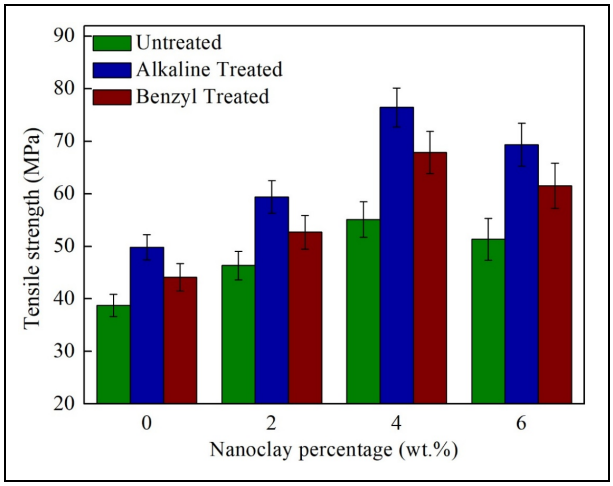


Fig. 4: Comparison of the Tensile strengths of Nanoclay filled composites

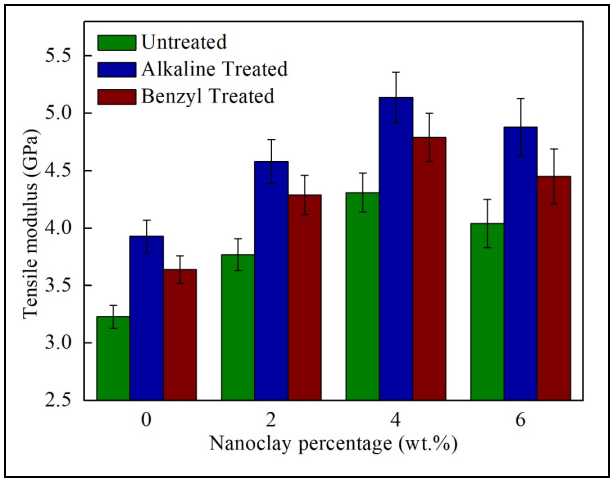


Fig. 5: Comparison of the Tensile moduli of Nanoclay filled composites

3.2 Flexural Strength of Composites

Bending tests subject composite samples to both normal and shear stresses. Fig. 6 illustrates how the flexural strength of FTPEFB fiber-reinforced composites varies based on untreated and surface-treated fibers. The incorporation of FTPEFB fibers into the epoxy increased flexural strength up to a Nanoclay level of 4 wt.%. However, exceeding this and reaching a 6 wt.% reinforcement led to a decrease in flexural strength (Devireddy and Biswas, 2017; Narayana *et al*. 2020). The 4 wt. % of Nanoclay-filled composites showed maximum flexural strength values of 108.4, 142.8, and 127.6 MPa for untreated, alkali- and benzyl-treated composites, respectively. A significant difference in the results was observed between the untreated composites and the treated composites. Compared to untreated composites,

the flexural strength increased by 31.73% and 17.71% with alkaline and benzyl treatments, respectively. This illustrates that alkaline treatment leads to a greater increase in flexural strength than benzyl treatment.

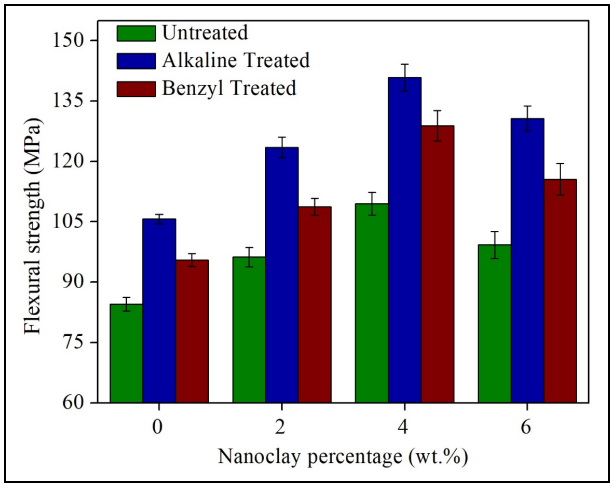


Fig. 6: Comparison of the Flexural strengths of Nanoclay filled composites

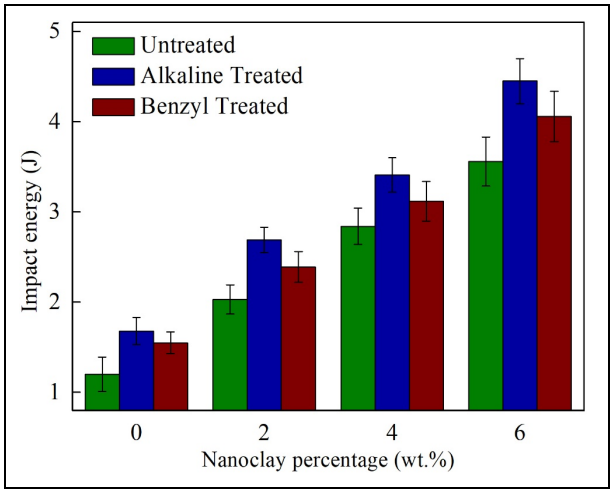


Fig. 7: Comparison of the Impact energies of Nanoclay filled composites

3.3 Impact Energy of Composites

Impact energy is a measure of a material's ability to resist sudden applied loads. It quantifies the amount of energy needed to fracture a given sample under impact conditions. The impact energy of a composite material is influenced by factors such as the interfacial bonding between components, fiber pullout, and the properties of both the matrix and the reinforcement. Fig. 7 illustrates the variation in impact energy of FTPEFB fiber-reinforced composites. Untreated FTPEFB fiber-reinforced composite specimens demonstrated lower energy impact compared to those with treated fibers. As the Nanoclay percentage increased, the impact energy of the fabricated composites also rose, reaching a maximum value at 6 wt.%. The maximum impact energy values recorded were 3.36 J for untreated composites, 4.15 J for alkaline-treated composites, and 3.86 J for benzyl-treated composites. Compared to untreated composites, the impact energy of alkaline-treated composites was higher by approximately 23.51%, while the impact energy of benzyl-treated composites was higher by about 14.88%. A similar trend was observed by Bachtiar *et al*. 2009 with sugar palm-reinforced epoxy composites, where alkali treatment with NaOH effectively modified the fiber surface, increasing the impact strength.

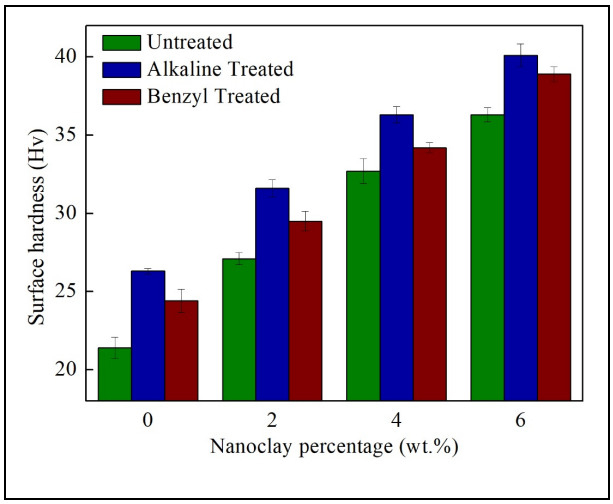


Fig. 8: Comparison of the hardness of Nanoclay filled composites

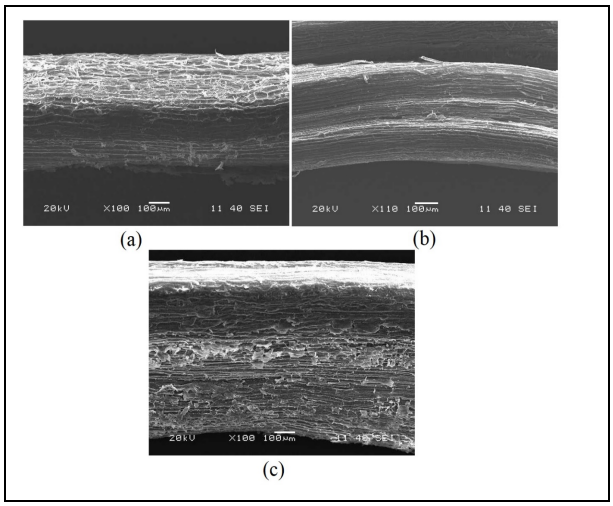


Fig. 9: SEM images of FTPEFB fibers: (a) Untreated (b) Alkali-treated and (c) Benzyl-treated

3.4 Surface Hardness of Composites

Fig. 8 shows the surface hardness values of the various FTPEFB fiber-reinforced composite specimens investigated in this study. The surface hardness of the fabricated composites gradually increased as the

Nanoclay filler percentage rose from 0 to 6 wt. % across all cases - untreated and treated composites. The maximum surface hardness was found to be 35.3, 39.1, and 37.9 HV for untreated, alkaline- and benzyl-treated composites, respectively. At 6 wt. % Nanoclay filler content, the composite treated with alkali exhibited a surface hardness value of 39.1 HV which was 10.72% higher than the untreated composite and 3.16% higher than the benzyl-treated composite. The increase in hardness was due to the better distribution of FTPEFB fibers within the epoxy, resulting in minimal voids and strong interfacial bonding between the epoxy and the reinforcement.

3.5 Morphological Analysis

In this study, morphological changes in fibers after treatment were examined. Fig. 9a shows the SEM micrograph of an untreated fiber, which contains impurities, fatty substances, wax, and globular protrusions. Significant differences were observed in the fiber morphologies after alkaline and benzyl treatments compared to the untreated fibers. Following alkaline treatment (Fig. 9b), a definitive change in the morphological structure was noted: the fibers appeared cleaner, and the roughness was significantly reduced as almost all impurities were removed from the fiber surface. Treatments with NaOH and benzyl effectively removed the surface wax and cuticle, resulting in a smoother fiber surface due to the interaction with sodium. The removal of binding materials led to fibrillation, which in turn caused the appearance of micropores in the treated fibers. The elimination of the superficial layer increased the contact area by exposing more fibrils. This process also reduced the fiber diameter and resulted in a rougher surface.

4. CONCLUSION

It was observed that the incorporation of Nanoclay and surface modification of FTPEFB fibers were essential to enhance the adhesion between the hydrophobic matrix and the hydrophilic fibers. The study revealed that incorporating 5% NaOH and 4 wt. % of Nanoclay substantially improved the tensile and flexural strength of the epoxy composite reinforced with FTPEFB fiber. This enhancement was demonstrated by the findings of the research. With the addition of 4 wt.% Nanoclay, specimens subjected to alkali treatment demonstrated a 37.96% increase in tensile strength and a 31.73% enhancement in flexural strength. The tensile modulus of the composite containing alkaline-treated fibers was approximately double that of the composite fabricated with untreated fibers. As a result, the epoxy composite containing Nanoclay filler exhibited enhanced stiffness. The findings indicated that a 4 wt. % Nanoclay content led to more significant enhancements in tensile properties and flexural strength compared to 6 wt. % Nanoclay. For the composites incorporating alkali-treated fibers and 6 wt. % Nanoclay, the impact strength increased by 23.51%, and the hardness improved by 10.76% when compared to composites without Nanoclay fillers. As evident from the SEM analysis of FTPEFB fiber surfaces, the application of chemical treatments removed impurities, including wax and fatty substances, leading to a decrease in the fibers' surface roughness.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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