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Effect of Surface Modified Montmorillonite Nanoclay on Tensile and Flexural Properties of Pineapple Leaf Fiber Reinforced Epoxy Composite

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Due to their biodegradability, affordability, low density, and numerous other benefits, natural fiber polymer composites are preferable to conventional GFRP in maritime applications. However, when exposed to moisture, their mechanical qualities deteriorate. A significant agricultural waste called pineapple leaf fiber (PALF) can be employed as reinforcement in epoxy matrices. Improved interfacial bonding between phases improves interfacial bonding and hence enhance mechanical and water absorption properties. Only evaluation of mechanical properties is undertaken in this paper. Nanoclay in 1.5 and 3 wt% was incorporated in epoxy resin via magnetic stirring and ultrasonication. PALF fibers were subjected to NaOH treatment and was analyzed using SEM and FTIR techniques. Hand layup and compression moulding were used to fabricate composites using a nanoclay-epoxy resin combination and chemically treated PALF (20 & 30 wt%). The combination of 30 wt% PALF and 1.5 wt% nanoclay results in the maximum mechanical properties, namely tensile and flexural properties. The results of SEM investigation of fractured specimens show that interfacial bonding in epoxy composites containing PALF is poor while that in epoxy composites containing PALF and 1.5 wt% nanoclay is excellent. Due to nanoclay agglomerations, bonding is inadequate at 3 wt% nanoclay, which lowers the mechanical properties.

Keywords: *Pineapple Leaf Fiber (PALF), natural fiber, nanoclay.*

1. Introduction

Despite the fact that traditional glass fiber reinforced polymer composites still dominate the recreational boating market, natural fiber composites have made progress in marine applications. This is due to the benefits that natural fibers offer, including biodegradability, lower end-of-life disposal costs, low density, and ease of supply[1-3]. However, they possess drawbacks too, such as inferior mechanical properties compared to synthetic fibers[4], high moisture absorption due to hydrophilic nature of natural fiber and weak compatibility with polymer matrices[5]. These characteristics result in inability to transfer stress from matrix to fiber, which along with swelling of natural fibers result in micro-cracking phenomenon. Such composites suffer greater mechanical degradation when they are exposed to severe environments like marine. The micro-cracks absorb more moisture which results in further moisture uptake[6], fiber swelling and eventually delamination of composite layers[7]. All these suggests that there is a need to impart hydrophobicity to natural fibers and improve the interfacial bonding or compatibility between fiber and polymer matrix. Among natural fibers, Pineapple Leaf Fiber (PALF) is a non-climacteric herbaceous perennial. It a tropical fruit cultivated widely after banana and citrus[8]. Once pineapple is harvested, the leaves are waste and have huge potential to be used as reinforcement material in composite due to its moderate mechanical property and other advantages that natural fibers possess in general[9]. literatures suggest that 7% NaOH chemical treatment enhances compatibility with the polymer matrix and leads to maximum improvement in tensile strength & thermal stability, reduced ash content and lowest moisture uptake[10]. Alternate way of enhancing interfacial bonding is by addition of nanoclay along with the natural fibers. Addition of nanoclay also leads to enhancement in fire resistance and optical behavior[5,11]. Literatures also suggest that it also leads to enhancement in mechanical properties[12], reduced saturation moisture uptake and diffusion coefficient[13] and negative effect of moisture absorption on mechanical properties. Researches have taken place on natural fiber nanoclay epoxy composites. Mylsamy et al.[14] researched the influence of nanoclay on mechanical properties of chemically treated cocinnia indica reinforced epoxy composites. Due to uniform dispersion and intercalation of nanoclay, properties enhance when 1-3 wt% organoclay is added. However, due to weaker interfacial bonding and agglomeration, the properties decrease at 4 wt% of organoclay. Bulut et al.[15] in an investigation on basalt fiber reinforced epoxy composites found that maximum enhancement in tensile, flexural and impact strength is for 2, 1.5 & 0.5 wt% of organoclay respectively. Mohan et al. researched the influence of nanoclay on water barrier property of sisal fiber epoxy composite. With 1-5 wt% nanoclay, there was no clear trend in variation of diffusivity. However, equilibrium water uptake reduced with increase in nanoclay content[16]. Ramakrishnan et al. enhanced mechanical properties and water absorption parameters of jute

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fiber epoxy composite by adding nanoclay in 1 to 7 wt%[17]. Alamri et al. found out that flexural properties of recycled cellulose fiber epoxy composites increase with increase in nanoclay content. With addition of nanoclay in 1, 3 and 5 wt%, saturation water uptake reduced whereas no clear trend in diffusivity was noticed. Water absorption resulted in decrease in flexural properties but surprisingly impact properties increased due to plasticization effect. Also, the impact of water absorption on flexural properties was less for composites with higher nanoclay content[18]. Literatures suggest that the there are no work conducted on pineapple leaf fiber nanoclay composites. Therefore, this paper harnesses the potential of a major agro-waste in India called as pineapple leaf fiber to be used as reinforcement for epoxy composites. Montmorillonite nanoclay a naturally occurring material is used as a barrier for the flow of moisture as well as to enhance the interfacial bonding. SEM analysis of treated and untreated PALF along with FTIR analysis is carried out. The alkali treated fibers are incorporated in epoxy matrix, tensile and flexural properties are evaluated. SEM analysis of fractured specimens are also carried out.

2. Experimental Procedure

2.1. Materials

Epoxy resin and curing agent used were Araldite LY556 and Aradur HY951. Technical specifications about the chemicals used is listed in Table 1. Pineapple leaf fiber (PALF) bi-directionally woven having 300 GSM was purchased from Go Green products, Chennai, Tamil Nadu. Surface modified nanoclay (modified by 15-35 wt. % octadecylamine, 0.5-5 wt. % aminopropyltriethoxysilane) manufactured by Sigma Aldrich was supplied by the local supplier. NaOH Pellets were also purchased locally.

2.2. Treatment of PALF

The treatment of the Pineapple Leaf Fibers (PALF) is essential to obtain enhancements in the natural fiber properties, thus improving the composite material made from those treated fibers. By using chemical treatment, the fiber-matrix adhesion will be enhanced by mechanical interlocking due to the increase in fiber surface adhesion. Based on the literature review done for this research, the optimum treatment would be to subject the PALF to alkaline treatment using NaOH pellets. The fibers are to be immersed in a NaOH solution having a 7% concentration[10].

The bidirectional woven fiber mats are immersed in the solution for 1 hour and then washed in water to remove the remaining NaOH. The mats are dried in hot air oven for 12 hours at a temperature setting of 70 - 80°C. The dried mats are used to produce laminates of different configurations of PALF reinforced composite.

Table 1. Specifications of resin-curing agent matrix.

Property	Unit	Araldite LY556	Aradur HY951
Viscosity at 25°C	mPa.S	10000-12000	10-20
Density at 25°C	gm/cc	1.15-1.20	0.97-0.99
Flash point	°C	>200	>180

2.3. Epoxy resin-nanoclay mixture preparation

To prepare the Epoxy Resin – Nano-Clay mixture, initially the required amount of nano-clay is mixed into an adequate amount of acetone (based on the percentage of nano-clay used for a particular composition in the final composite material). The initial mixture is mixed at room temperature for 30 minutes using a Magnetic Stirrer. After this, the required amount of epoxy resin is mixed into the initial mixture and subsequently stirred by hand. This mixture is then sonicated using an ultrasonicator. The sonicated mixture is then stirred at 60°C using a magnetic stirrer to remove the acetone present in the mixture. The mixture is then placed in a vacuum oven to make sure there is no presence of acetone in the mixture.

2.4. Fabrication of Composites

The composite is prepared using the hand-layup method. It is a simple, low-cost method of manufacturing composite material. The wet laminate is then compression molded to a pressure of 15 kg/cm^2 at room temperature for 10 minutes to ensure that the fibers and resin have no air gaps in between and that the bonding is done well[19]. This was followed by curing at room temperature for 48 hours. The PALF fiber, Epoxy resin, and Nano-Clay weight percentages are varied to produce 6 different configurations as shown in the Table 2.

2.5. Characterization

2.5.1. Fourier Transform Infrared Spectroscopy (FTIR)

Initially, the PALFs are to be analyzed before preparing the composite, using FTIR spectroscopy, to understand the various components present in the fiber, such as cellulose, lignin, wax, hemicellulose, etc., and to identify the fingerprint and functional groups present in the materials. This form of analysis is useful in identifying the chemical bonds present in the material. Using FTIR spectroscopy, the untreated and alkali treated PALF samples are analyzed and the different chemical bonds are compared to understand the difference between the two types of PALF samples.

o Fourier Transform Infrared Spectroscopy (FTIR): Shimadzu IRsprit. Sample size 1cm × 1 cm

2.5.2. SEM-EDX Analysis

SEM analysis is used to observe the microfibril structure of both the untreated and alkali-treated fiber. This helps in identifying the change in the fibers after the alkali treatment. The analysis of fractured specimens is also to be conducted on the composite samples after the tensile test to analyze the bonding between the fiber, matrix, and nano-clay.

The two different PALF specimen types are chemically analyzed using EDX, or Energy-Dispersive X-ray Spectroscopy. In this examination, the samples are struck by an electron beam from the EDX apparatus, which will excite the impinging electrons to a free electron state or an unoccupied level of greater energy. These excitations cause photons to be released that are unique to each element present in the fiber samples, providing us with information about the elements contained in the sample and their corresponding percentages.

2.5.3. Tensile and Flexural Testing

The purpose of these tests, as their name implies, is to determine the tensile and flexural characteristics of the created composite samples. For the tensile test and the flexural test, the samples are prepared in accordance with ASTM D3039 and ASTM D790, respectively. Tensile test was carried out in BiSS 50 kN servo hydraulic machine at cross head displacement rate of 2 mm/min. 3 point bending or flexural tests were carried out in Instron 3366-10 kN universal testing machine. Samples are cut from the several manufactured PALF reinforced composite configurations. It is feasible to determine the impact of PALF and nano-clay content on the mechanical properties of these composites by conducting these experiments on the various configurations of PALF reinforced composites.

3. Results and Discussion

3.1. FTIR Analysis

Figure 1's FTIR spectroscopy findings for untreated and alkali treated PALF demonstrate a quantifiable distinction between the two types of PALF samples. Since PALF is a natural fiber, its lignocellulosic substance is made up mostly of cellulose, hemicellulose, lignin, wax, and other residual components[20].

The spectra show that the untreated sample's absorption peaks at 3361.14 cm^{-1}, which is consistent with the O-H stretching of -cellulose. The peaks at 2927.62 cm^{-1} in the untreated sample are the C-H bonding that the lignocellulosic material of the PALF has created[21]. The hemicellulose's C=O carbonyl group and the ether group's C-O stretch are shown by the tiny peaks at wavelengths of 1649.91 cm^{-1} and 1244.91 cm^{-1}, respectively. Only the untreated sample contains these peaks. The large peak at 1036.72 cm^{-1} is due to the cellulose's polysaccharide's C-O/C-C bond stretching[22,23].

The transmittance has generally risen for the alkali-treated sample. At 3324.06 cm^{-1}, the peak has significantly decreased, which can only be explained by the O-H group being eliminated because of the alkali treatment. Similarly reduced peak heights can also be observed at 1609.98 cm^{-1} and 2920.49 cm^{-1} wavelengths. It is possible to see an increase in transmittance at 1244.91 cm^{-1}. At 1031.01 cm^{-1}, a sharp increase in transmittance can also be seen. These differences in transmittance between the two types of samples demonstrate that the alkali treatment of the fibre sample removed lignin, hemicellulose, wax, and other trace components[24]. A comparison of the FTIR results for the two types of PALF samples is shown in Table 3.

3.2. SEM analysis of PALF

The SEM images of the untreated and alkali treated PALF are shown in Figure 2. When comparing 2(a) and 2(b), the sample's microfibrils are evident in the shape of grooves in Figure 2b, but the untreated sample in Figure 2a has a smoother waxy surface. Similar investigations have employed SEM to examine the surface morphology of untreated pineapple fiber and discovered that it has a very

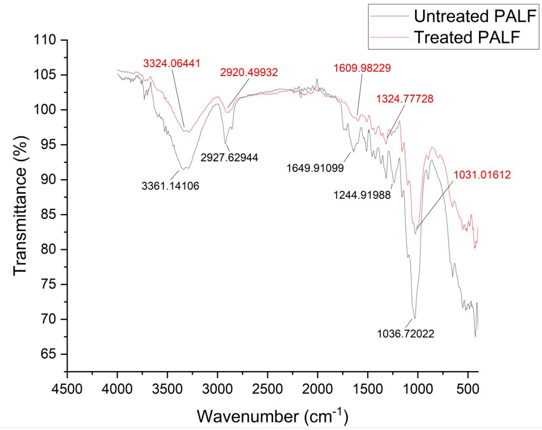

Figure 1. FTIR spectra of treated and untreated PALF fiber.

Table 2. Configurations of PALF reinforced composite.

Code	P20NC0	P20NC1.5	P20NC3	P30NC0	P30NC1.5	P30NC3
PALF (wt%)	20	20	20	30	30	30
Nanoclay (wt%)	0	1.5	3	0	1.5	3
Epoxy (wt%)	80	78.5	77	70	68.5	67

Table 3. Wavenumber and transmittance of different peaks corresponding to specific chemical bonds.

Sl No.	Untreated Sample		Treated Sample		Chemical Bonds
	Wavenumber (cm^{-1})	Transmittance (%)	Wavenumber (cm^{-1})	Transmittance (%)	
1.	3361.14	91.71	3324.06	96.98	O-H stretching of α-cellulose
2.	2927.62	95.46	2920.49	99.85	C-H bonding (lignocellulosic material)
3.	1649.91	94.34	1609.98	98.69	C=O carbonyl group of hemicellulose (Untreated only)
4.	1244.91	89.95	1244.91	96.95	C-O stretch of ether group (Untreated only)
5.	1036.72	70.45	1031.01	82.56	C-O/C-C bond of polysaccharides present in cellulose

smooth surface free of pores or pits, indicating that wax, lignin, and other fatty acids are present there. The PALF sample gets rougher and has better cohesiveness with the reinforcement once these components are removed[25].The morphology observed in Figure 2b agrees with the findings of Jain et al., who found that the alkaline treatment removes lignin and other outer waxy material, leaving a rough surface suitable for polymeric matrix[20].

Figure 3a and 3b are EDX spectrum of untreated and treated PALF respectively. The elements and its weight percentage obtained from the analysis are given in Table 4. It is observed that carbon (c), nitrogen (N) and oxygen (O) are the major elements. There are other trace elements such as Na, Mg, Si, Co, etc. In the treated fiber, the weight percentage of nitrogen elements has reduced[26].

3.3. Tensile and Flexural tests

Composites containing combination of alkali treated PALF and nanoclay weight percentages in different configurations were tested for tensile and flexural properties. The stress-strain curves from tensile tests and force-displacement curves from flexural tests are plotted in Figure 4 and 5 respectively. The average values of tensile strength, tensile modulus, flexural strength, flexural modulus and percentage elongation at break along with their standard deviation are plotted in Figures 6, 7, 8, 9 & 10 respectively.

Figures 6 and 7 show that increasing the amount of fibre in a PALF epoxy composite material from 20 to 30 weight percent significantly improves its tensile strength and modulus respectively. This is because there is a greater transmission of applied load from the epoxy matrix to the fibre reinforcement when more fibres are added for a certain volume of composite material[27]. According to the rule of mixtures theory of composite materials, the tensile strength and modulus will also increase when fibre weight or volume

Table 4. Elemental composition of untreated and treated PALF from EDX analysis.

Untreated fiber		Treated fiber	
Element	Weight %	Element	Weight %
C	36.27	C	49.42
N	22.72	N	3.84
O	38.30	O	44.97
Na	0.29	Na	0.14
Mg	0.07	Mg	0.05
Si	1.76	Si	0.57
Ca	0.15	Ca	0.33
Co	0.45	Co	0.12
-	-	K	0.02
-	-	Fe	0.10
-	-	Cu	0.42

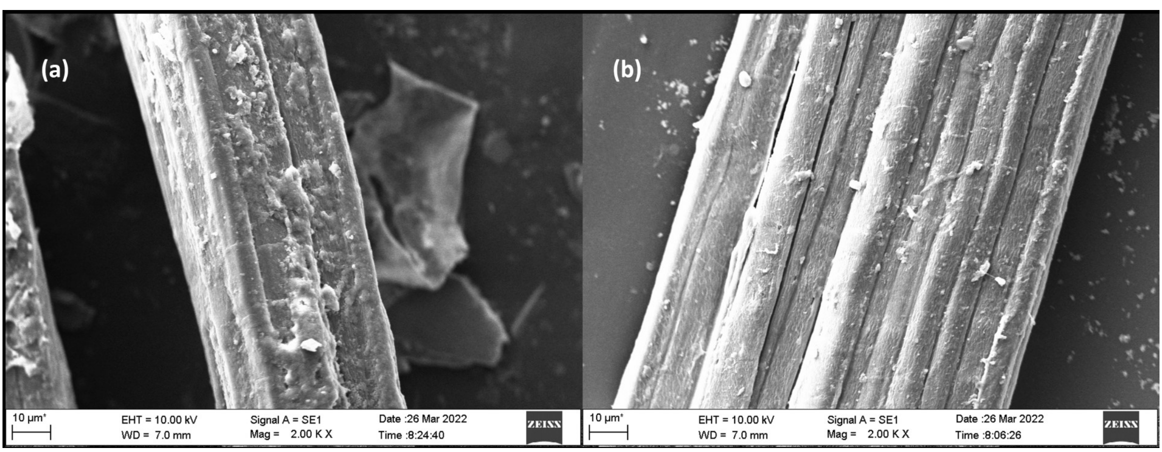

Figure 2. SEM Image of (a) untreated PALF (b) alkali treated PALF.

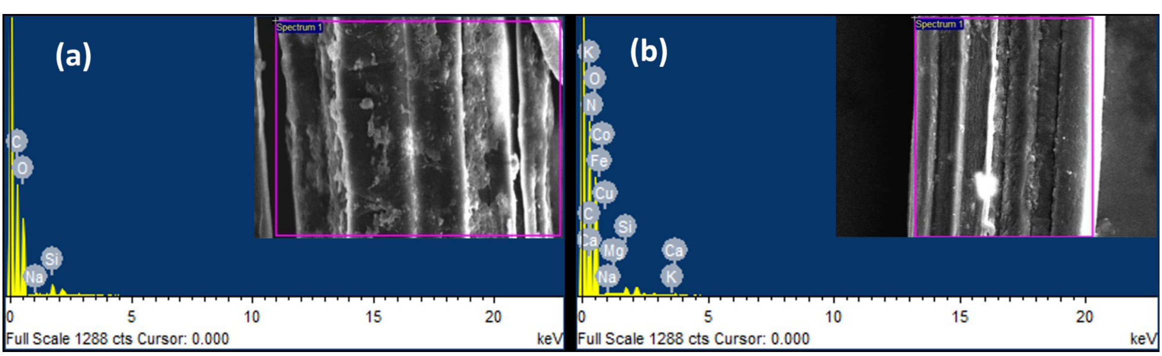

Figure 3. EDX images of (a) untreated and (b) treated PALF.

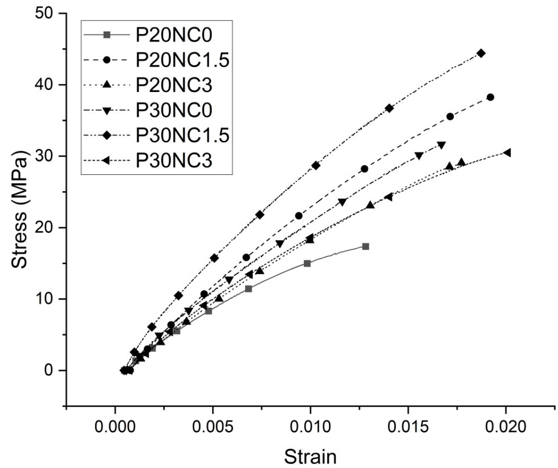

Figure 4. Stress-strain curve from tensile tests for epoxy PALF composites with and without nanoclay.

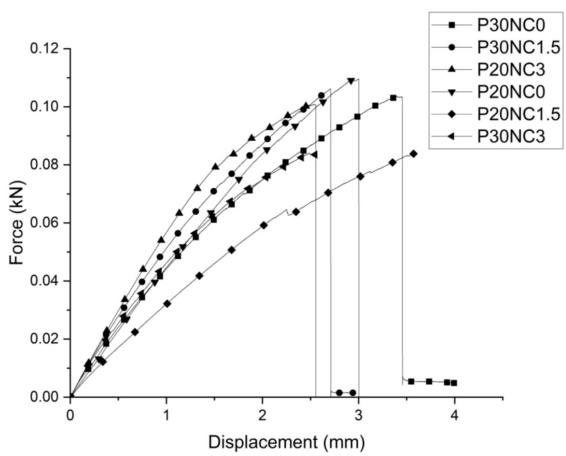

Figure 5. Force - displacement curves from flexural test for epoxy PALF composites with and without nanoclay.

percentage increases. In their study of the effects of PALF on polypropylene composites, Feng et al. found that increasing the amount of fibre from 20 to 30 weight percent led to a 55% improvement[28]. Similarly, Motaleb et al. obtained 34% improvement when PALF content was increased from 30 to 40 wt%[29]. When 1.5 wt% nanoclay is introduced to a PALF epoxy composite that contains 20 wt% PALF fibre, the tensile strength and modulus significantly increase. Similar to this, the tensile strength and modulus of a PALF epoxy composite that contains 30 weight percent of PALF fibres rise by 33% and 21%, respectively. Tensile characteristics generally improve when nanoclay is added in 1.5 wt%, but they degrade when nanoclay is raised from 1.5 to 3 wt%. Tensile capabilities improve when nanoclay is evenly spread in an epoxy matrix due to the matrix's strengthening properties and improved interfacial bonding. However, greater loadings of nanoclay tend to cause them to aggregate, which results in poor load transfer and consequently inferior tensile characteristics. Ramakrishnan et al. observed maximum strength at 5 wt%

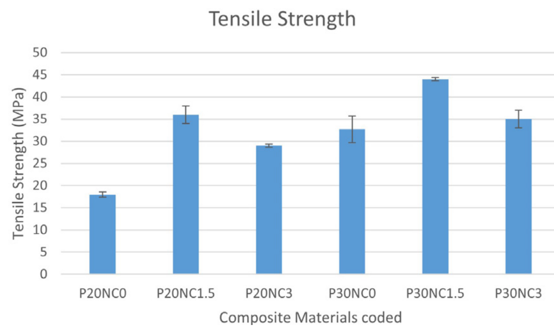

Figure 6. Tensile strength of alkali treated PALF nanoclay epoxy composites (P stands for PALF weight and NC stands for nanoclay weight percentage).

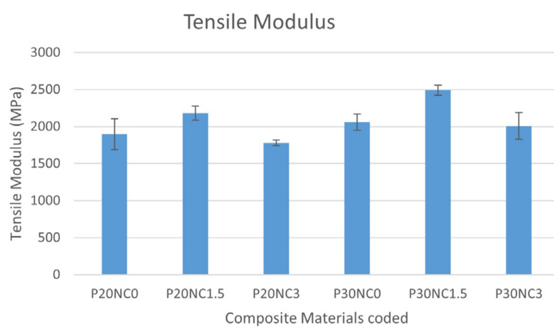

Figure 7. Tensile modulus of alkali treated PALF nanoclay epoxy composites.

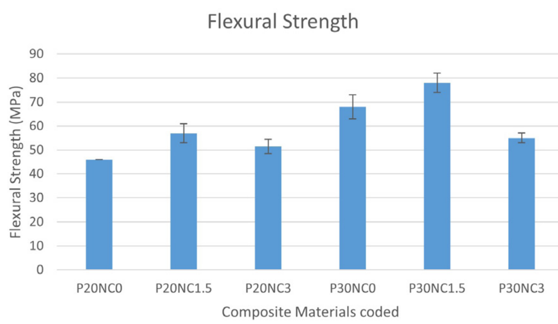

Figure 8. Flexural strength of alkali treated PALF nanoclay epoxy composites.

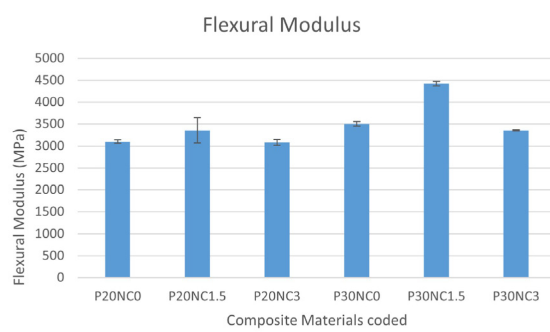

Figure 9. Flexural modulus of alkali treated PALF nanoclay epoxy composites.

addition of nanoclay and a dip at 7 wt% whereas Rao et al. observed maximum value at 4 wt% and reduction when nanoclay was increased to 5 wt%[17,30]. From Figure 8 & 9 it can also be observed that flexural strength and modulus also increase when fibers added increases from 20 to 30 wt%. When nanoclay added increases to 1.5 wt%, the flexural properties also enhance. However, at higher nanoclay loading, it decreases. From Figure 10 & Table 5, It is observed that, percentage elongation at break or fracture which represents ductility, enhances with increase in the amount of nanoclay in epoxy composites. When nanoclay is added to epoxy matrix, it causes plasticization of epoxy chain network, which leads to enhancement in ductility[31]. However, at nanoclay loading of 3 wt%, due to agglomeration, ductility reduces eventhough it is greater than that of PALF epoxy composites.

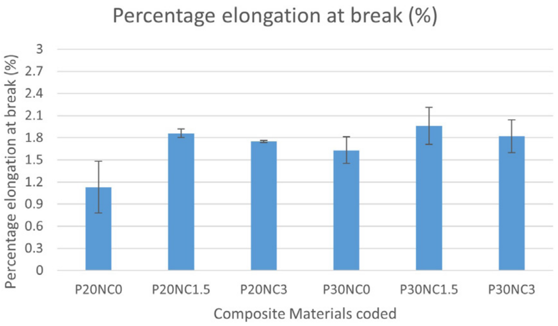

Figure 10. Percentage elongation at break (%) for different configurations of composite material.

3.4. SEM analysis of fractured composites

From Figure 11, it is observed that, composites that have 30 wt% of PALF (Figure 11b) have lesser spacing between fibers and have a denser structure compared to that with 20 wt% of PALF. Therefore, the tensile strength enhances because there is better transfer of applied load from epoxy matrix to PALF fiber all around.

Figure 12b shows the fiber pull-out in the P30NC0 composite after tensile failure. This signifies poor interfacial bonding between the matrix and reinforcement which is shown in Figure 12a. However, it can be seen in Figure 13b that fiber breakage has occurred in the P30NC1.5 composite. Fiber breakage occurs because the interfacial bonding is good because of addition of nanoclay and hence the energy supplied in applied load is easily transferred to fiber due to which it breaks only when applied load exceeds fiber ultimate tensile strength. Therefore when nanoclay is incorporated tensile strength enhances[14].

Figure 14a depicts how PALF is bonded with the epoxy matrix and 14(b) shows matrix rich zone. From 14(a), it can be noted that in some areas there is poor interfacial bonding and some other area it is exactly opposite. This is because when nanoclay content is increased from 1.5 to 3 wt%, nanoclay is not intercalated or exfoliated, they are rather in the form of nanoclay agglomerations. Due to agglomerations, load transferred is not effectively transferred to fiber, hence the decrease in tensile strength and modulus[17].

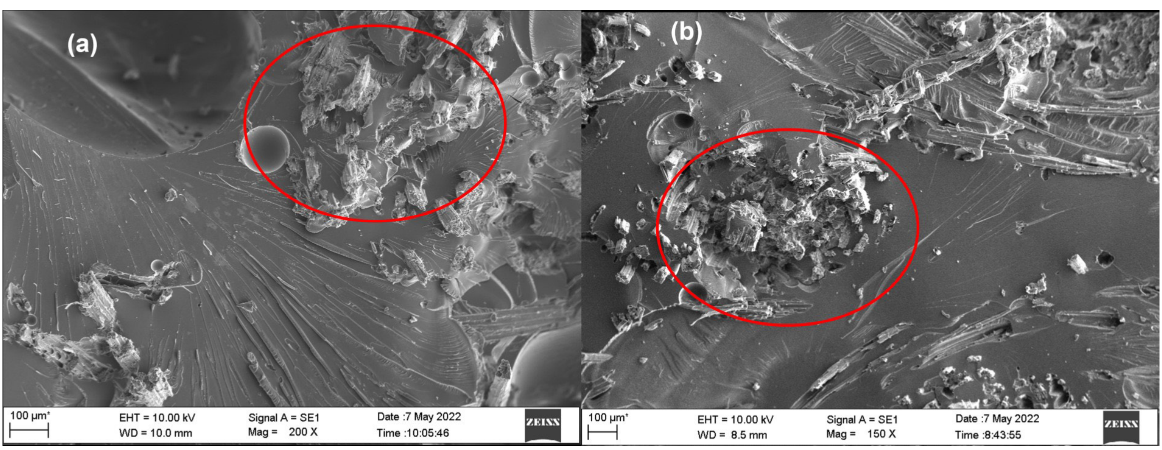

Figure 11. SEM images of (a) P20NC0 and (b) P30NC0.

Table 5. Tensile properties of composites.

Code	Tensile Strength (MPa)	Standard Deviation	Tensile Modulus (MPa)	Standard Deviation	Percentage elongation at break (%)	Standard deviation
P20NC0	18	0.6	1897	209	1.13	0.35
P20NC1.5	36	2	2179	95	1.86	0.06
P20NC3	29	0.4	1779	37	1.75	0.015
P30NC0	33	3	2058	109	1.63	0.18
P30NC1.5	44	0.4	2490	69	1.96	0.25
P30NC3	35	2	2007	181	1.82	0.22

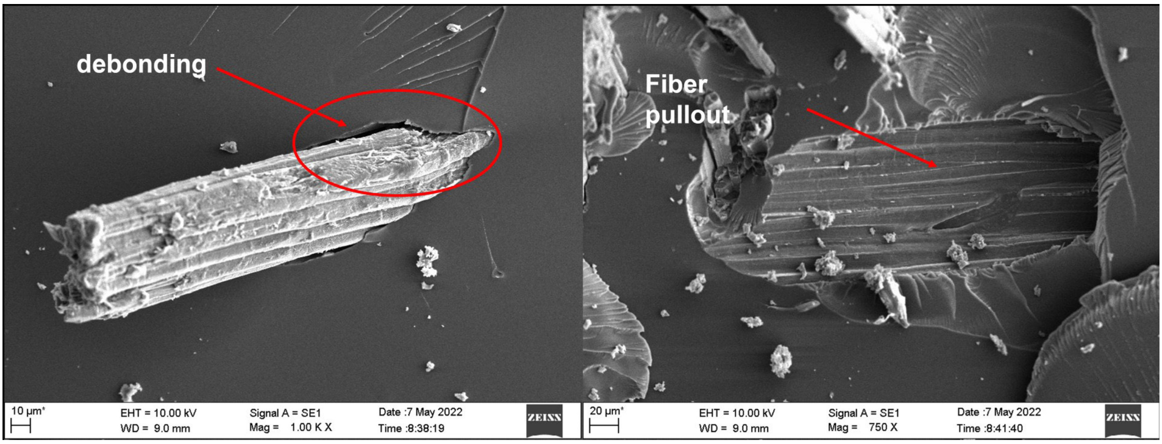

Figure 12. (a) SEM image of P30NC0 showing debonding at fiber matrix interface and (b) showing fiber pull-out.

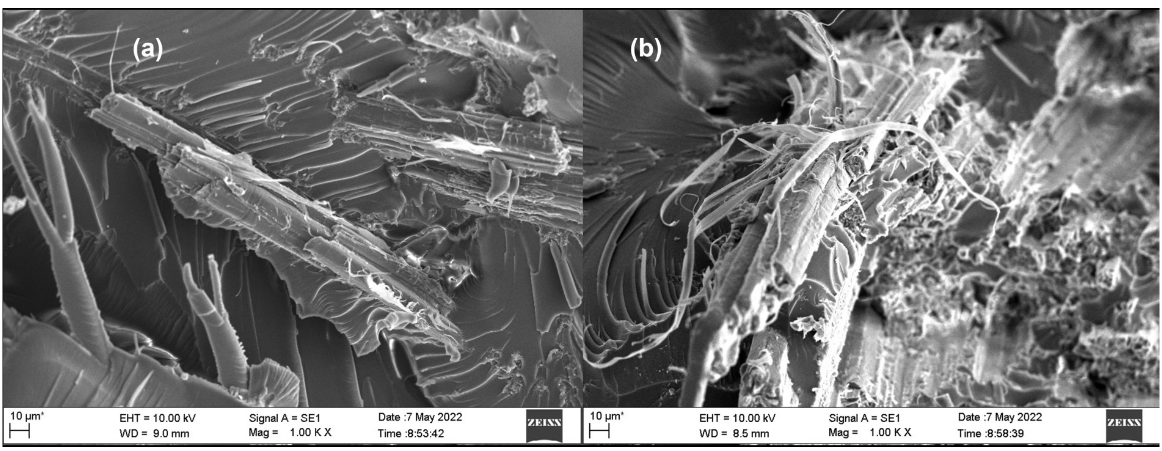

Figure 13. SEM images of P30NC1.5, (a)showing strong interfacial bonding, (b)showing fiber breakage or fracture.

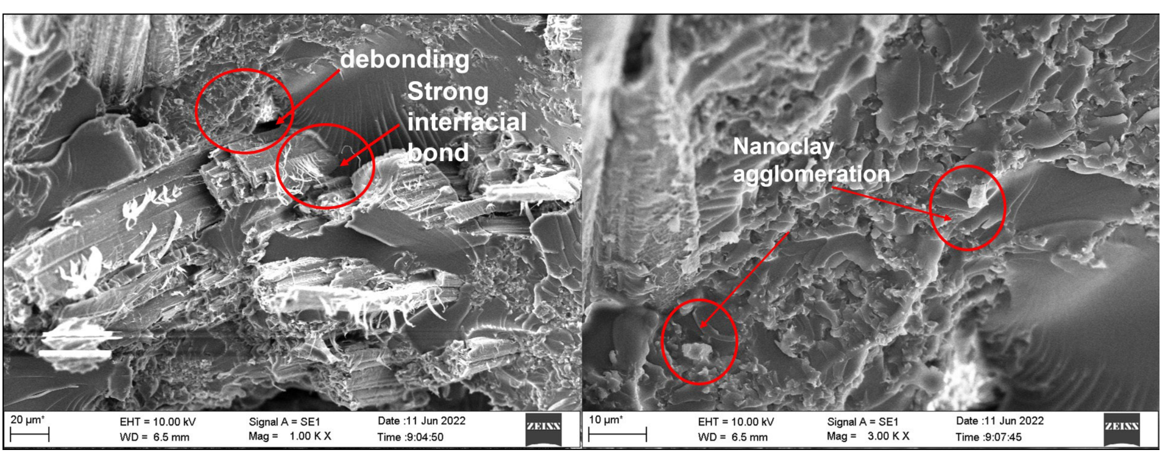

Figure 14. SEM images of P30NC3, (a) showing debonding in one area and strong interfacial bond in another area, (b)Matrix area showing nanoclay agglomeration.

4. Conclusion

The effect of alkali treatment on PALF and the effect of surface modified montmorillonite on alkali treated PALF reinforced epoxy composite were analyzed and following conclusions were drawn:

- Differences in Infrared transmittance between untreated and treated PALF, demonstrate that the alkali treatment removed lignin, hemicellulose, wax, and other trace components as it was evident from FTIR analysis. Surface morphological analysis of treated PALF, shows that the surface becomes rougher by removal of lignin and other waxy material.

- Tensile strength, tensile modulus, percentage elongation at break, flexural strength, and flexural modulus enhances when PALF content increases from 20 to 30 wt%. When montmorillonite is added till 1.5 wt%, the properties enhance, however, when it is increased to 3 wt%, the properties reduce.
- The SEM analysis of a fractured specimen assisted in determining the causes of the behaviour that was noticed after the addition of fibres and nanoclay. It was concluded that the addition of 3 wt% montmorillonite causes agglomerations that weaken interfacial bonding, which can reduce tensile and flexural characteristics.

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