



Mechanical, thermal and morphological analysis of hybrid natural and glass fiber-reinforced hybrid resin nanocomposites

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Abstract

In this report, natural fiber mats (cotton, bamboo, and jute) and artificial fiber mat (glass) reinforced with hybrid resin matrix (Lansea Coromandelica plant gum and epoxy) and bentonite nanoclay to form hybrid composites are fabricated by hand-lay-up compression technique and then analyzed. The structural morphology of tensile fracture in hybrid composites is investigated using the equipment of scanning electron microscope (SEM). Additionally, hybrid resin and hybrid composites are also analyzed by using differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA). The structural effect of nanoclay (2%, 4%, 6%) mixing in hybrid resin (HR) matrix reinforcement in cotton (C/G/C)/bamboo (B/G/B)/jute (J/G/J) fiber mats for finding the tensile properties, flexural properties, impact strength, hardness value, thermal degradation temperature, and water absorption property is investigated in this work. The findings of the abovementioned tests show that the treated C/G/C fiber mats with 4% vol fraction nanoclay mixed in HR matrix resin have superior mechanical properties of the tensile strength (87 MPa), tensile modulus (3701 MPa), flexural strength (274.6 MPa), flexural modulus (4713 MPa), impact strength (5.4 J), and hardness (35.1 MPa) when compared to other hybrid composites, and HR and the results of other tests are narrated completely in this paper.

Keywords Natural fibers mats (cotton/bamboo/jute) · Sodium hydroxide treatment · Hybrid resin · Water absorption

1 Introduction

In numerous applications, composite materials are growingly being employed to replace conventional materials. Because of the rapid growth of technology in industries, keeping track of the advancement of creative material designs for the replacement of existing materials is vital [1]. As a result of their low weight and high strength as well as their cost-effectiveness, polymer composites are commonly recognized as the preferable alternative materials [2,

3]. Recently, the growing technical field is paving the way for the utilization of environmental resources, particularly those generated from plants, while keeping environmental concerns and renewability in mind [4]. Natural fiber composites have recently grown in popularity as a result of their environmental benefits. Natural fiber composites have several advantages over synthetic fiber composites including minimal cost, biocompatible, low density, insulating, excellent physical properties, excellent heat properties, and reduced processing energy consumption [5]. Natural fibers are sustainable and renewable resources that are increasingly being used as clays in polymer or biopolymer composites to replace artificial materials [6]. Natural fibers are largely made up of cellulose, hemicellulose, and lignin with minimum amounts of lipids, waxes, and inorganic elements [7]. The correct bonding agents, chemical procedures, filler particle addition, layer thickness, and structural reinforcement are all used to improve the performance features of bast fiber (plant fiber) [8].

In the past few years, several studies have been conducted to construct composites with natural fibers as reinforcement and thermoplastic/thermosetting resins as matrices.

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Attempts to replace synthetic resin and synthetic fiber with biomaterials have been made in recent years [9]. Polymers are generally hydrophobic whereas natural fibers are hydrophilic. Many polymer composites are being developed by material scientists using readily available natural fibers. To produce the composites with better mechanical qualities and environmental performances, natural fibers must be chemically modified with appropriate additives or sprayed with appropriate resins [10]. Acetylation, alkali, acylation, silane, and benzylation are some of the chemical treatments that have recently been applied. The alkaline treatment is the least expensive and the most widely used treatment of all the procedures. It causes the hydrogen bonds in fiber bundles to break down into tiny fibers, reducing hydrophilic clusters in the matrix/fiber interaction [11].

Cotton (generic name: *Gossypium*) is a natural fiber from the Hibisceae (Malvaceae) family that is made up of pure cellulose with a high molecular weight, high strength, and great absorbency. Cotton fibers differ significantly in gross morphology such as convolutions, cell wall thickness, and cross-sectional form as regards fine structure such as fibrillar orientation, reversals, and microfibril packing density from variety to variety and species to species [12].

Using the rag-tearing procedure, cotton fibers were recovered from textile waste. The thermoset composites were made by utilizing a compression molding process with an epoxy resin matrix and four distinct reinforcements as cotton fiber's volume percent values of 0.1, 0.2, 0.3, and 0.4. DMA and TGA tests were used to evaluate the produced composites. With an increment in fiber vol %, the mechanical properties and storage modulus of the composites were improved, but the damping factor was decreased [13].

Researchers are interested in the cotton fiber-reinforced polymer composite (CFRPC) because of its easy uses and substantially improved mechanical properties. The CFRPC is widely utilized in architectural applications such as roof ornamentation, interior panels for automobiles, and exterior bodies of home appliances [14]. The separation of lignin and hemicelluloses from cotton fiber-reinforced in polyester matrix resin composites was improved when the fiber composite material was treated with NaOH, increasing the mechanical properties of the fiber composites [15].

Bamboo is a varied genus of evergreen flowering plants that belong to the Bambusoideae subfamily of the Poaceae grass family. It is completely sustainable, eco-friendly, non-toxic, low-cost, non-abrasive, and disposable. The cellulose fibers embedded in lignin are aligned throughout the length of the bamboo, which is another important factor in employing as a reinforcing material in polymer composites [16].

Bamboo fibers are gaining popularity because of their morphological properties and cellular structure, which enabled them to provide maximum tensile strength, maximum flexural strength, and higher specific strength than the other

natural fibers such as sisal, straw, banana, and coir [17]. Bamboo fibers were treated for 12 h with sodium hydroxide solutions of 2 wt. percent, 6 wt. percent, and 10 wt. percent, respectively. The manufactured bamboo composites were evaluated using mechanical properties, SEM, FTIR technique, and TGA testing. Bamboo fibers (6 wt. percent) had better mechanical strength and thermal strength than untreated fibers. SEM and FTIR study of bamboo fibers, after alkali treatment, revealed that non-structural elements such as lignin, pectin, and hemicellulose were partially eliminated with alkali treatment [18].

Jute (generic name: *Corchorus capsularis*) is a natural fiber from the Malvaceae family. The major elements of this lignocellulosic fiber are α -cellulose, hemicelluloses, and lignin. Jute is a lignocellulosic fiber that is half-textile and half-wood. Jute is categorized as a bast fiber (fiber collected from bast or skin of the plant) [19]. The mechanical properties of jute composites including various thermosets, thermoplastic polymers, bio-based resins, and jute hybrid composites were investigated. Long-staple length, maximum tensile strength, nonabrasive nature, low price, and ease of fabrication are a few advantages of jute fiber. Jute has been widely utilized to make ropes, bags, packaging, carpet backings, decorating fabrics, geotextiles, etc. [20]. The mechanical properties of jute composites including various thermosets, thermoplastic polymers, bio-based resins, and jute hybrid composites were investigated. The physical properties, mechanical properties, and impact of chemical treatment of jute fiber-reinforced epoxy matrix were discussed. The findings implied that chemically treating jute fibers promoted interfacial adhesion to the epoxy matrix which enhanced the tensile properties of composites. The fiber–matrix contact adhesion was increased in SEM pictures of the broken surface of treated epoxy/jute composites [21].

The effect of different nanoclay in the mechanical and thermal properties of natural fiber-reinforced thermoset and thermoplastic composites (hybrid bamboo and kenaf fiber) is investigated. Nanoclay-based hybrid composites particularly organically modified MMT displayed excellent mechanical properties, dimensional stability against water absorption, and thermal expansion due to the high interfacial adhesion between the nanoclay OMMT and the polymer matrix [22]. The effect of barium sulfate (BaSO_4) on the mechanical properties of natural hybrid-reinforced polymeric HNRP (aloe vera/flax/barium sulfate) composites was investigated. The tensile and hardness properties of HNRP were improved, whereas the flexural strength was decreased due to the presence of BaSO_4 particles. Due to its lower water solubility, the addition of BaSO_4 reduced the water absorption capabilities of the HNRP composites. The addition of BaSO_4 particles increased the storage modulus and reduced the damping factor [23].

From the above research works, it is inferred that alkali treatment has improved the performance of hybrid composites. Moreover, the combination of treated natural fiber mats and treated synthetic fiber mat-reinforced hybrid resin nanoclay composites was not studied yet. Consequently, the research gap is identified and hence the fabrication of the new hybrid composites in this work is done using treated hybrid fiber mats (cotton, bamboo, jute, and glass), hybrid resin (*Lannea Coromandelica* plant gum and epoxy), and nanoclay filler. The novelty of this work is the fabrication of new hybrid composites (combination of hybrid resin, hybrid fibers, and nanoclay filler) and then analyzing their thermal property of them by using differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA) technique.

The present research work's objectives are as follows:

1. Sodium hydroxide untreated and treated cotton/glass/cotton (C/G/C) fiber mats, bamboo/glass/bamboo (B/G/B) fiber mats, and jute/glass/jute (J/G/J) fiber mats with volume fraction (2%, 4%, 6%) of bentonite nanoclay (NC) mixing in every composition in HR matrix resin composites and hybrid resin (HR) specimen are fabricated by hand-lay-up compression technique.
2. The tensile properties, hardness value, flexural properties, and impact strength of hybrid composites and HR are examined.
3. Differential scanning calorimeter (DSC) test is conducted to determine the heat stability and degradation temperature of hybrid composites and HR.
4. Thermogravimetric analysis (TGA) is used to determine the thermal stability for hybrid resin and hybrid composites.
5. The property of resistant water is examined by a water absorption test.
6. Structural morphology of tensile fracture in hybrid composites is studied by scanning electron microscope.

2 Materials and methods

2.1 Materials

The epoxy resin (LY556) and hardener (HY951) are purchased from M/s Sakthi fiberglass, Chennai. Bentonite nanoclay (pH value 8.5–10) is purchased from Emerck (P) Ltd., Mumbai, India. Glass fiber/cotton fiber/jute fiber/bamboo fiber of density 450 GSM (woven roving fiber mat) is obtained from Fiber Region (P) Ltd., Chennai. *Lannea Coromandelica* having a pH value of gum in the range of 3.3–3.5 is procured from M/s Evergreen Fibers & Chemicals (P) Ltd., Chennai, India. The above materials are used to fabricate hybrid composites.

2.2 NaOH (alkaline) treatment

The glass fiber mats are cleaned by the way of pressurized water for a minimum of 1 h to remove the fine particles, residues, and organic materials from the mats. Then, the glass fiber mats are dried for 2 days under sunlight and hot air oven at 95 °C for 24 h to remove moisture content from them, and then, the glass fiber mats are immersed in distilled water at room temperature and dried in an oven (with air circulated) at 95 °C for 24 h. Afterwards, the glass fiber mats are immersed in 4 wt. percent of sodium hydroxide solution (relative to weight percent of glass fiber mat) for 1 day and then the treated glass fiber mats are washed thoroughly with distilled water to remove the excess of NaOH. Afterwards, glass fiber mats are dried at room temperature for 1 day, and then, the treated glass fiber mats are further dried in an oven at 95 °C for 24 h. Likewise, the identical method is adopted for natural fiber (cotton/bamboo/jute) mats, but the treating temperature is maintained at 40 °C. The alkaline treatment is the least expensive and the most widely used treatment. It leads to the breakdown of the hydrogen bonds in fiber bundles into small fibers and reduces the clusters of hydrophilic, polymer insufficiency, and poor matrix/fiber interaction [19, 24].

2.3 Preparation of HR

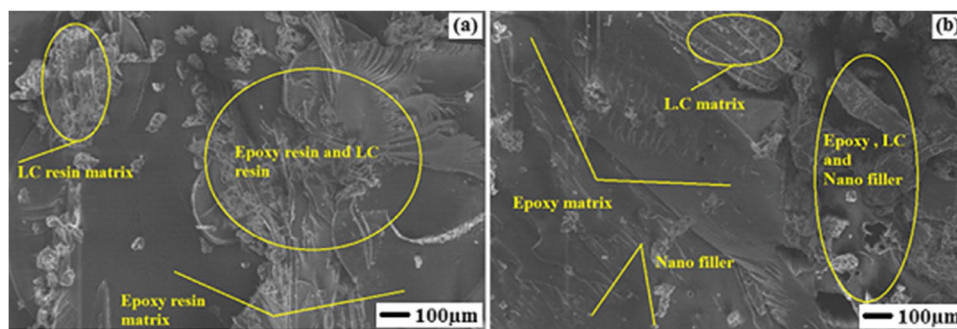
The *Lannea coromandelica* (plant LC) gum is dried at room temperature for 10 days before being dried at 40° centigrade in the oven for 1 h. To make fine powder, the coarse gum is blended for 10 min with a mechanical dynamic mixer (SunkaierSK090). HR is created by mixing LC fine powder (40%) with epoxy resin matrix (60%) using the mechanical mixer technique [25].

Hybrid resin is made up of the fine structure of LC matrix powder in the pure epoxy resin matrix as seen in Fig. 1(a). In hybrid resin, the clay is well intercalated and exfoliated with clay tactoids as shown in Fig. 1(b). Nanoclay filler is intercalated and exfoliated as well as confirming clay platelet dispersion in hybrid resin. Clay tactoids give a bright view on the possibilities of increasing clay dispersion in resin in order to increase the mechanical strength [3].

2.4 Preparation of laminates for hybrid composites and HLCE resin

Laminates (300 × 300 × 3.2 mm³) dimension with three layers (each layer having the weight of 15 g) of C/G/C fiber mats are placed in order. HR (300 ml) resin (optimum contents) is combined with 2% of volume fraction (weight of 10 g) of bentonite nano clay (NC) powder and hardener (HY951) in a 10:1 ratio by high shear mixing for half an hour at an optimal speed of (1000–1050) RPM and then

Fig. 1 SEM images of **a** hybrid resin and **b** hybrid resin with nanoclay filler



coated well in the fiber mats while fabrication of laminates. During the fabrication of laminates, the surrounding temperature must be at room temperature to avoid the air from trapping into the specimen. The roller is used on the laminates to remove the trapped air bubbles during the hand lay-up technique and to keep the laminate's thickness below certain limits to minimize exothermal heat generation. The required amount of resin clay mixture is poured into the mold and then a stiff plate (for better pressure distribution) with a weight of 500 N is kept on the mold to maintain pressure (5 bar) which compresses the laminates. As indicated in Table 1, other hybrid composites are made in a similar manner by utilizing the compression hand lay-up technique.

2.5 Composite characterizations

The fabricated hybrid composite's specimens are conducted through a number of tests to determine the mechanical properties including the tensile test, impact test, hardness test, and flexural test. Using five specimens of each hybrid composite, the average values of the results obtained are calculated for every test category. The short explanations for the several testing methods and devices used in this research are given below.

2.5.1 Tensile testing

HR and hybrid composites' specimens are tested for tensile properties by using Tinius Olsen universal testing machine (UTM) as per standard ASTM D638 as depicted in Figs. 2 and 3.

2.5.2 Flexural testing

HR and hybrid composites' specimens are tested for flexural properties by using Tinius Olsen universal testing machine (UTM) as per standard ASTM D790. Specimen dimensions and specimen photos are illustrated in Figs. 4 and 5.

2.5.3 Impact test

The unnotched specimens of HR and hybrid composites are tested for impact strength as per ASTM standard D4812 using Izod Testing Machine (Make: EIE Instruments PVT LD). Specimen dimensions and specimen photos are given in Figs. 6 and 7.

2.5.4 Hardness test

The hybrid composites and hybrid resin specimens are tested for hardness using universal hardness tester—Wilson (UH4250) (Make: BUEHLER PVT LD) using indenter of carbide balls of 2.5 mm in accordance with the standard ASTM E 10.

2.6 Water absorption test

Water absorption in percentage is determined in accordance with standard (ASTM D570) for specimens of hybrid composites. The dimension used for the test specimen is $25.4 \times 7.62 \times 3.2 \text{ mm}^3$. The equation for calculating the percentage (%) of water absorption is as follows:

$$\% \text{ Water absorption} = \left(\frac{(W_f - W_i)}{W_i} \right) \times 100$$

where W_i denotes the sample's initial weight (gm) prior to immersion and W_f denotes the sample's final weight (gm) after water immersion.

2.7 Fractography study

Hybrid composite specimens are applied with gold coating and then kept inside a device of sputter coating for the time range of 19–21 min. Hybrid composite specimens are tested to determine the microstructure for analyzing the mechanical properties and structural morphology by utilizing—Apreo S scanning electron microscope (FESEM: Model Quattro S) having magnification range ($650\times$) and pressure range (25 Pa).

Table 1 Details of composition of untreated and treated C/G/C, B/G/B, and J/G/J fiber mat-reinforced hybrid resin (HR) nanocomposites

Sample specification		Hybrid resin matrix (HR) 300 ml	Reinforcement (woven roving mat (WRM)) 2vol%–10 g 4vol%–20 g 6vol%–30 g
Hybrid resin (HR)	HR	HR	–
Cotton fiber (C)	UTC1	HR	UT C/G/C WRM + 2 vol% NC
	UTC2	HR	UT C/G/C WRM + 4 vol% NC
	UTC3	HR	UT C/G/C WRM + 6 vol% NC
	TC4	HR	T C/G/C WRM + 2 vol% NC
	TC5	HR	T C/G/C WRM + 4 vol% NC
	TC6	HR	T C/G/C WRM + 6 vol% NC
Bamboo fiber (B)	UTB1	HR	UT B/G/B WRM + 2 vol% NC
	UTB2	HR	UT B/G/B WRM + 4 vol% NC
	UTB3	HR	UT B/G/B WRM + 6 vol% NC
	TB4	HR	T B/G/B WRM + 2 vol% NC
	TB5	HR	T B/G/B WRM + 4 vol% NC
	TB6	HR	T B/G/B WRM + 6 vol% NC
Jute fiber (J)	UTJ1	HR	UT J/G/J WRM + 2 vol% NC
	UTJ2	HR	UT J/G/J WRM + 4 vol% NC
	UTJ3	HR	UT J/G/J WRM + 6 vol% NC
	TJ4	HR	TJ/G/J WRM + 2 vol% NC
	TJ5	HR	TJ/G/J WRM + 4 vol% NC
	TJ6	HR	T J/G/J WRM + 6 vol% NC

Fig. 2 Dimension of specimen used for tensile test (ASTM D638)

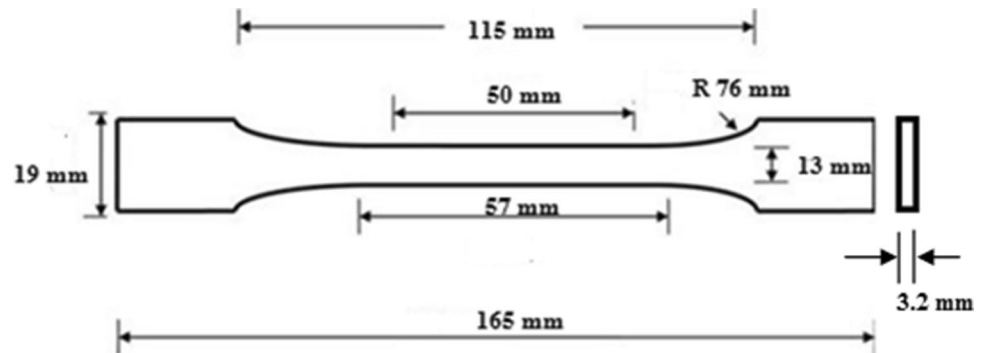


Fig. 3 Photographic image of tensile test of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites

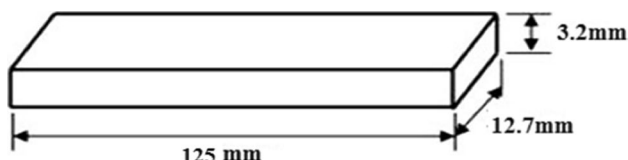
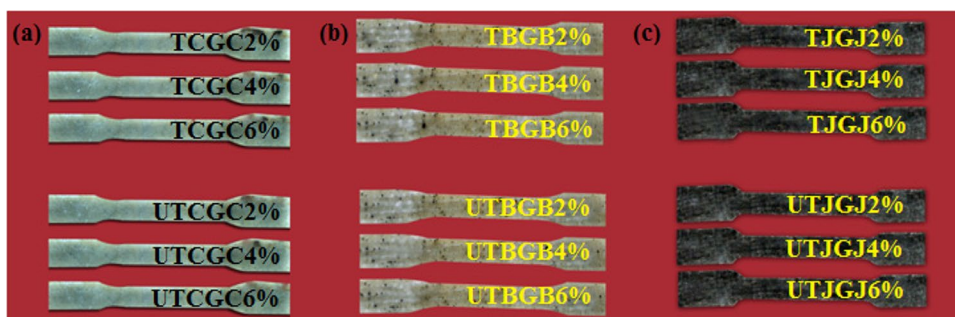
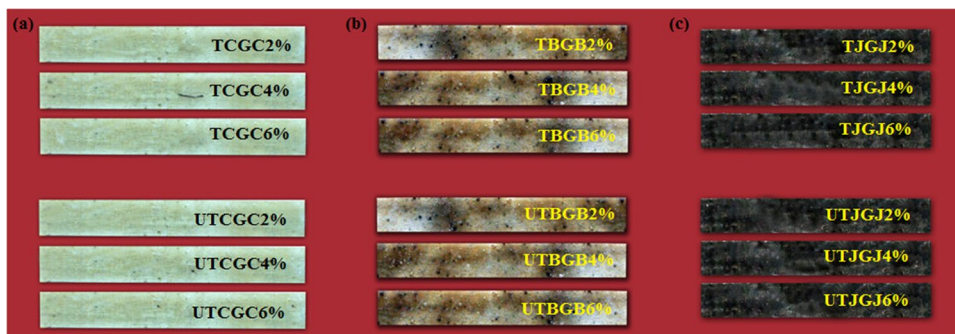


Fig. 4 Dimensions of specimen used for flexural strength (ASTM D790). W width of the specimen = 12.7 mm, L length of the specimen = 125 mm, and T thickness of the specimen = 3.2 mm

Fig. 5 Photographic image of flexural test of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites. W width of the specimen = 12.7 mm, L length of the specimen = 125 mm, and T thickness of the specimen = 3.2 mm



2.8 Differential scanning calorimetry (DSC) test

Heat flow and thermal degradation of the hybrid composite specimens are determined by differential scanning calorimetry technique using Thermogravimetric analyzer (Model: NETZSCH STA 449F3) available at Annamalai University, Chidambaram. A weight of 10 g of each sample powder is taken in a platinum pan and heated up to a temperature of 600 °C at an increment of 20 °C/min under a nitrogen environment.

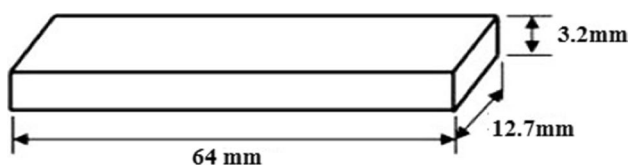


Fig. 6 Dimensions of specimen used for impact strength (ASTM D4812). W width of the specimen = 12.7 mm, L length of the specimen = 64 mm, and T thickness of the specimen = 3.2 mm

2.9 Thermogravimetric analysis (TGA) test

The thermal stability of the hybrid composite and HLCE specimen's powder is investigated by TG/DTA 6200 SEIKO TGA analyzer in the range of temperature 0–600 °C with a minimum increment of 25 °C/min. The weight loss % of hybrid composites and HR specimens is determined by TGA test.

3 Results and discussion

3.1 Tensile properties

The tensile strength of HR is 15.7 MPa. Figures 8 and 9 show the effect of adding bentonite nano clay (NC) in HR matrix reinforced in fiber mats. The tensile strength of hybrid composites having treated C/G/C fiber mats 4% nanoclay reinforced in HR matrix has improved up to 87 MPa. The values of tensile strength observed for treated cotton fiber mats (C/G/C) 4% nanoclay-reinforced HR composites are greater than 20.6% in comparison with treated bamboo fiber mats (B/G/B) 4% nanoclay-reinforced HR composites and 28.5% in comparison with treated jute fiber mats (J/G/J) 4% nanoclay-reinforced HR composites. The tensile modulus of HR is 712 MPa. The treated C/G/C fiber mats 4% nanoclay feeding in HR composites have the maximum tensile modulus of 3701 MPa. The values of tensile modulus observed for treated cotton fiber mats (C/G/C) 4% nanoclay-reinforced HR

Fig. 7 Photographic image of impact test of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites. W width of the specimen = 12.7 mm, L length of the specimen = 64 mm, and T thickness of the specimen = 3.2 mm

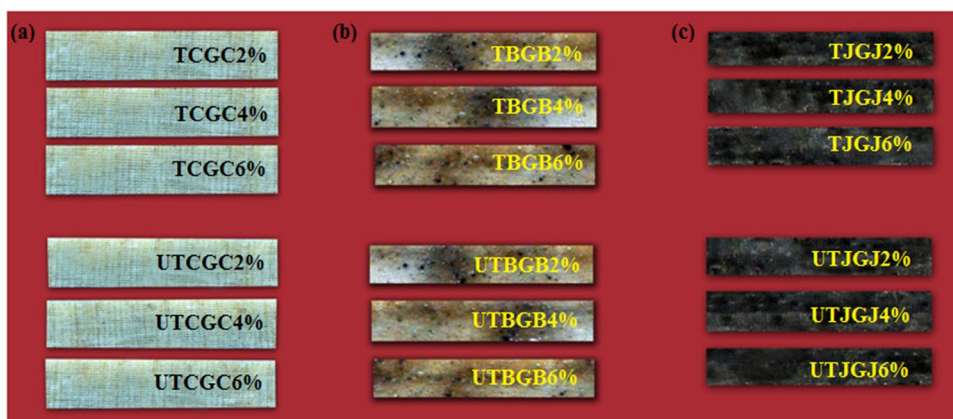


Fig. 8 Comparison of tensile strength of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites

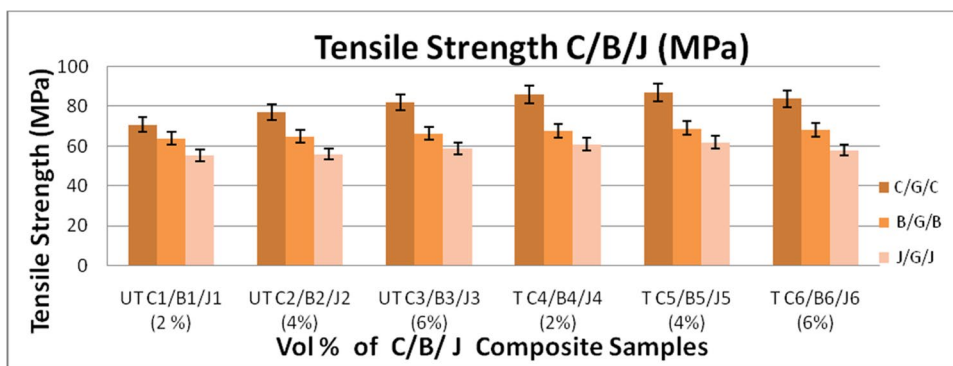
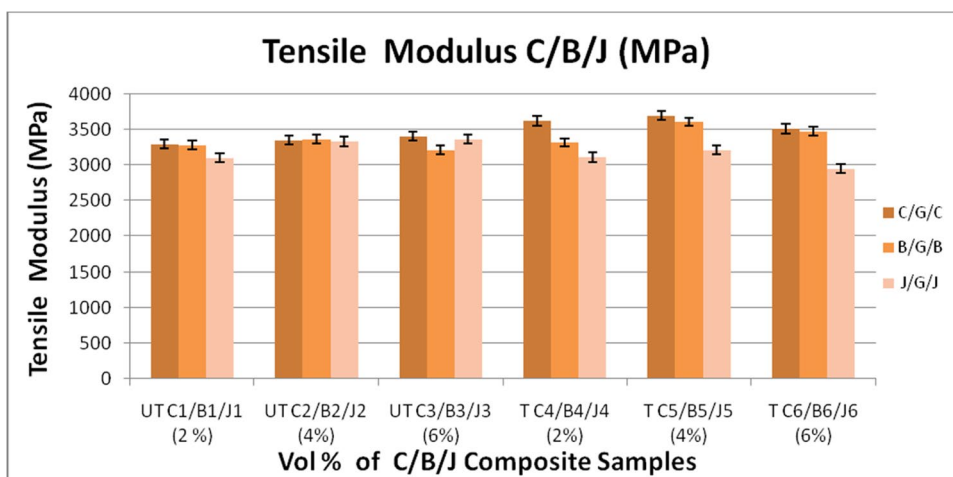


Fig. 9 Comparison of tensile modulus of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites



composites are more than 2.5% in comparison with treated bamboo fiber mats (B/G/B) 4% nanoclay-reinforced HR composites and 13.2% in comparison with treated jute fiber mats (J/G/J) 4% nanoclay-reinforced HR composites. In comparison with the other hybrid (B/G/B/J/G/J) fiber mats mixed with HR nanocomposites, it is concluded that the treated cotton fiber mats (C/G/C) nanoclay reinforced HR composites have gained the highest tensile properties. The stress–strain graph of treated cotton fiber mats (C/G/C) nanoclay (2%, 4%, and

6%)-reinforced HR composites also shows more load-bearing capacity as seen in Fig. 10. This is due to the reason of the cotton fiber cellular structure having strong, tough, wear resistance, and tear-resistant properties. The authors had stated that the cotton fiber was combined with artificial glass fiber and resin to form a good strength and durability property [13, 14]. Poor tensile strength parameters are caused by insufficient bonding between jute fiber and hybrid resin matrix as shown in the stress–strain graph (Fig. 10). When compared to other

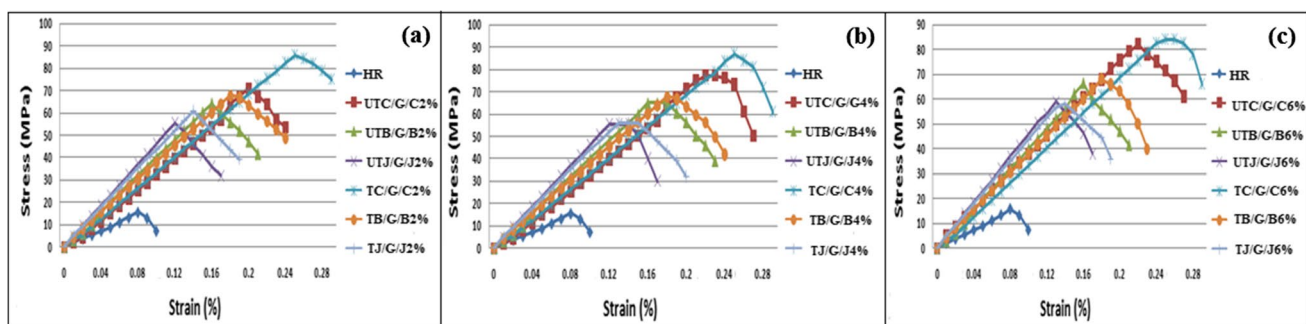


Fig. 10 Comparison of stress strain curves of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites

hybrid (B/G/B/J/G/J) fiber mats blended HR nanocomposites, the presence of nanoclay fillers in equal quantities was responsible for the modest increase in tensile strength [3, 20]. The tensile properties have increased linearly up to 4 vol % nanoclay, but then began to deteriorate, despite increasing the fiber reinforcement to 6 vol % nanoclay.

The results show evidence that higher mixing of nanoclay in resin has taken a difficult time to completely include in the fibers, leading to poor bonding and reduced mechanical strength. The cracked and compressed nanoclay dispersed in the HR matrix is also known to induce the various matrix material's strengthening mechanisms [3]. Crack stapling, rupture arresting, and other competitive advantages noticed in clay materials to enhance the strength of hybrid composites are mentioned by the researchers [26].

3.2 Fractography observation

SEM images (Figs. 11, 12, and 13) show the structural morphology of the cross-sectional area of hybrid composites. The tensile strength of composites is improved by treating C/G/C fiber mats with (2%, 4%, and 6%)

volume percentage nanoclay feeding in HR. Because the treated C/G/C fiber mats with (2%, 4%, and 6%) volume percentage nanoclay feeding in HR have no voids, are well trapped, and have good interfacial bonding. In comparison with other treated hybrid composites, the treated C/G/C fiber mats indicate the best bonding between cotton (C/G/C) fiber, hybrid resin, and nanoclay. The HR matrix has nanoclay distributed homogeneously in hybrid cotton (C/G/C) fiber mats as shown in Figs. 11(a), 12(a), and 13(a). The hybrid *Lannea Coromandelica* blender epoxy matrix features uniform dispersion of hybrid fiber and nanofiller. C/G/C fiber mats are commonly treated with the chemical sodium hydroxide solution to promote interfacial bonding between fiber, matrix, and filler resulting in increased tensile strength due to a better fiber-matrix well-built interface for the full-fiber surface area [14, 27]. The tensile strength of bamboo fiber mats with (2%, 4%, and 6%) volume fraction of nanoclay feeding in HR has decreased. The inclusion of nanoclay filler loadings causes void coalescence and fiber pull out between the nanoclay filler and hybrid resin matrix as shown in Figs. 11(b), 12(b), and 13(b), resulting in the reduction in

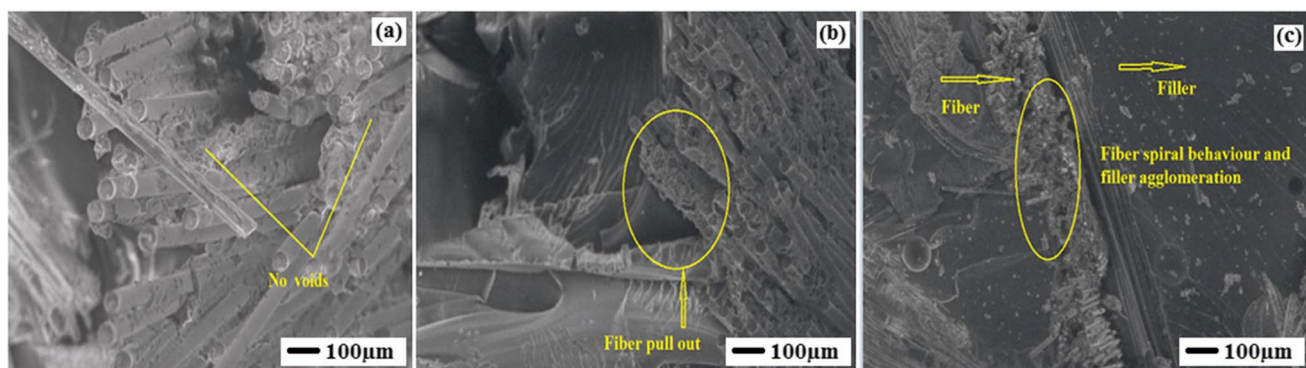


Fig. 11 SEM images of cross-sectional area of the treated of a C/G/C, b B/G/B, and c J/G/J fiber mat-reinforced HR nanocomposites (2%)

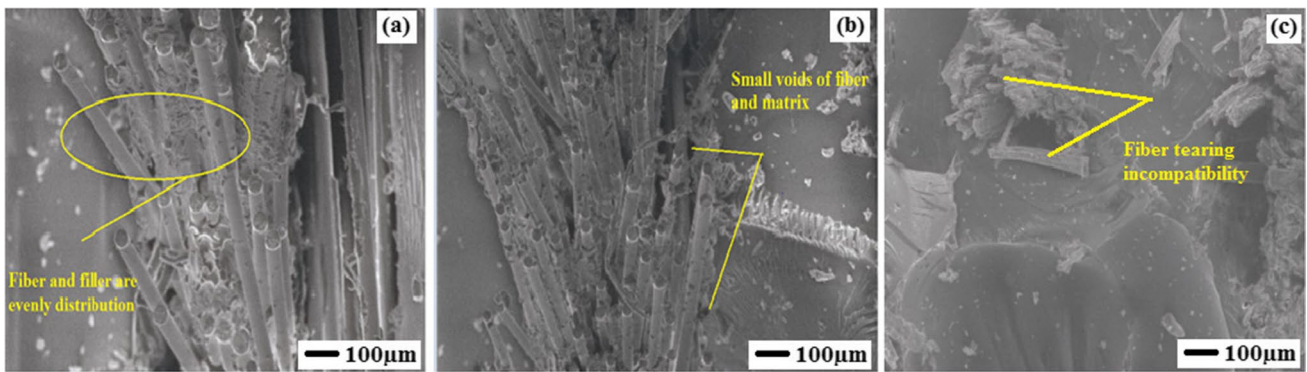


Fig. 12 SEM images of cross-sectional area of the treated of **a** C/G/C, **b** B/G/B, and **c** J/G/J fiber mat-reinforced HR nanocomposites (4%)

tensile strength of bamboo fiber mats. With the addition of bamboo fiber, tensile strength has a little bit improved. The strength of the composites is slightly improved due to fiber’s ability to withstand pressures transferred from the polymer elements [22]. The tensile strength of jute fiber mats with (2%, 4%, and 6%) volume fraction of nanoclay feeding in HR has highly decreased. SEM images of the treated J/G/J fiber mats with 2%, 4%, and 6% volume fraction of nanoclay reinforced hybrid resin reveal the fiber’s spiral behavior, fiber’s tearing incompatibility, agglomeration of filler, and uneven spreading of hybrid

resin matrix in composites as shown in Figs. 11(c), 12(c), and 13(c), leading to decreased tensile strength [20, 28].

SEM images of the surface area as depicted in Fig. 14 of treated J/G/J, B/G/B fiber mats with 4% volume fraction feeding in HR, reveal poor bonding of bamboo fiber/nanoclay and clustering and agglomeration of jute fiber/nanoclay, resulting in weak strength between (J/G/J, B/G/B) fiber mats, nanoclay, and hybrid resin matrix. The fiber pullout demonstrated the matrix’s poor interfacial interaction with the fiber. The energy dissipation during the frictional process mechanics was also detected [3, 26].

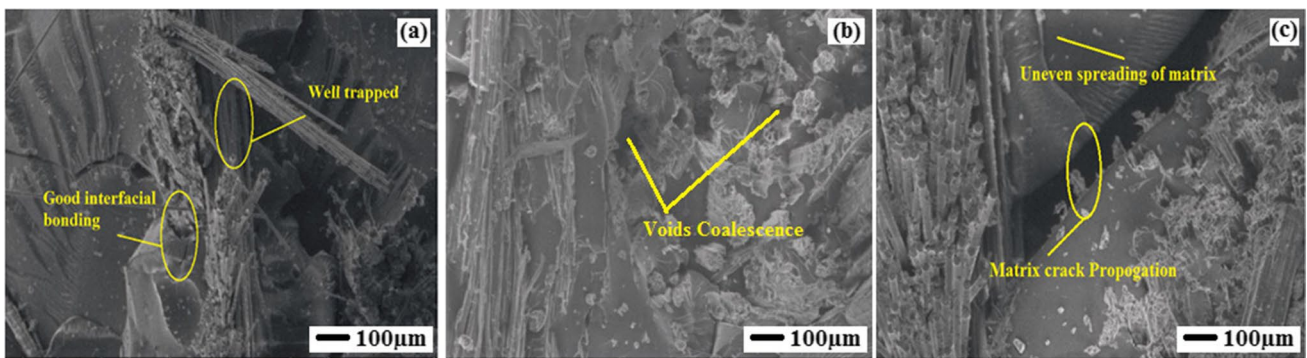
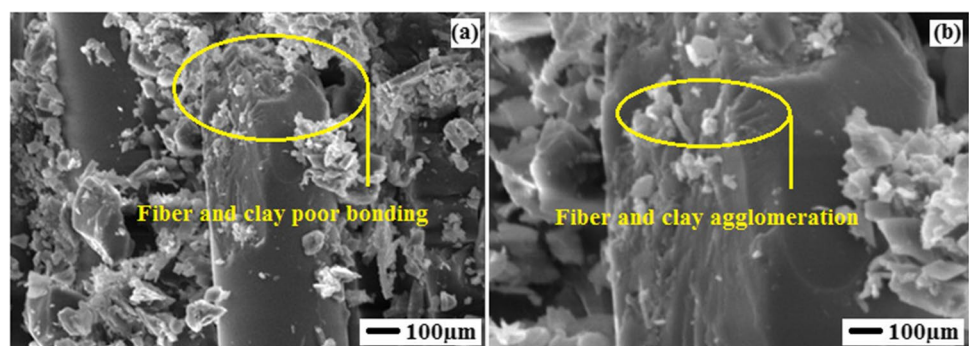


Fig. 13 SEM images of cross-sectional area of the treated of **a** C/G/C, **b** B/G/B, and **c** J/G/J fiber mat-reinforced HR nanocomposites (6%)

Fig. 14 SEM images of surface area of the treated of **a** B/G/B and **b** J/G/J fiber mat-reinforced HR nanocomposites (4%)



3.3 Flexural properties

HR has a flexural strength of 37.6 MPa. Figures 15 and 16 show the effects of adding bentonite nanoclay (NC) in HR matrix feeding in hybrid fiber composites. Flexural strength of treated C/G/C fiber mats with nanoclay (4%) feeding in the HR matrix has increased to 274.6 MPa. The values of flexural strength confirmed for treated cotton fiber mats (C/G/C) 4% nanoclay-reinforced HR composites are greater than 11.5% in comparison with treated bamboo fiber mats (B/G/B) 4% nanoclay-reinforced HR composites and 10.5% in comparison with treated jute fiber mats (J/G/J) 4% nanoclay-reinforced HR composites. HR has a flexural modulus of 855 MPa. The treated C/G/C fiber mats with nano clay (4%) feeding in the HR matrix have the maximum flexural modulus of 4713 MPa. The values of flexural modulus noticed for treated cotton fiber mats (C/G/C) 4% nanoclay-reinforced HR composites are more than 4.2% in comparison with treated bamboo fiber mats (B/G/B) 4% nanoclay-reinforced HR composites and 7.2% in comparison with treated jute fiber mats (J/G/J) 4% nanoclay-reinforced HR composites. In comparison with the other hybrid (B/G/B,

J/G/J) fiber mat-reinforced HR nanocomposites, treated C/G/C fiber mats 4% nanoclay mixed in HR composite has better flexural properties in a similar manner as the results obtained in tensile properties. The fiber alignment in the composite's outer layer surface had accounted for the composite's higher flexural strength readings as related to their tensile strength [29, 30].

3.4 Impact strength

Figure 17 shows the impact strength of hybrid composites. HR has 1 J impact strength. The higher impact strength of 4.6 J is found in untreated C/G/C fiber mat-reinforced HR with 6 vol % nanoclay composites. Likewise, the impact strength of treated C/G/C fiber mats mixed with 4 vol % nanoclay in HR matrix has been founded to be 5.4 J. Cotton (C/G/C) fiber mats mixed with HR nanocomposites have higher impact strength in compared with other hybrid (B/G/B/J/G/J) fiber mats feeding in HR nanocomposites. As per the results, the untreated and treated cotton fiber mats (C/G/C)-reinforced HR nanocomposites exhibit the highest impact strength compared to other fiber (J/G/J and B/G/B)

Fig. 15 Comparison of flexural strength of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites

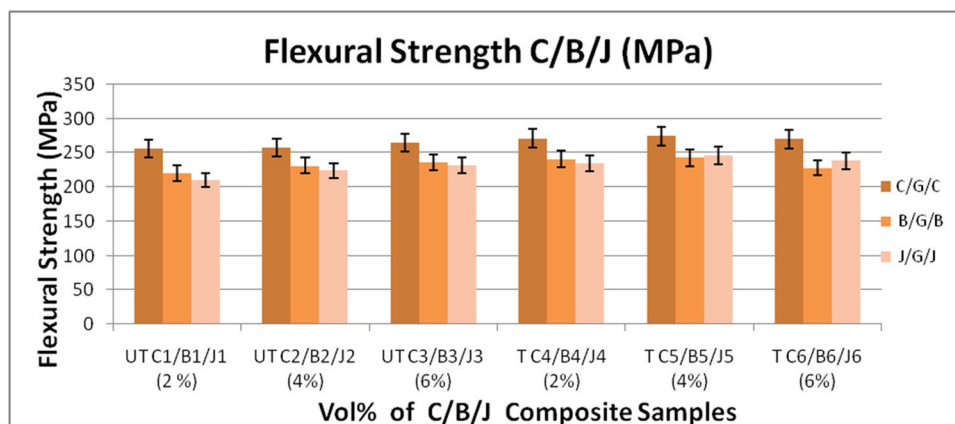


Fig. 16 Comparison of flexural modulus of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites

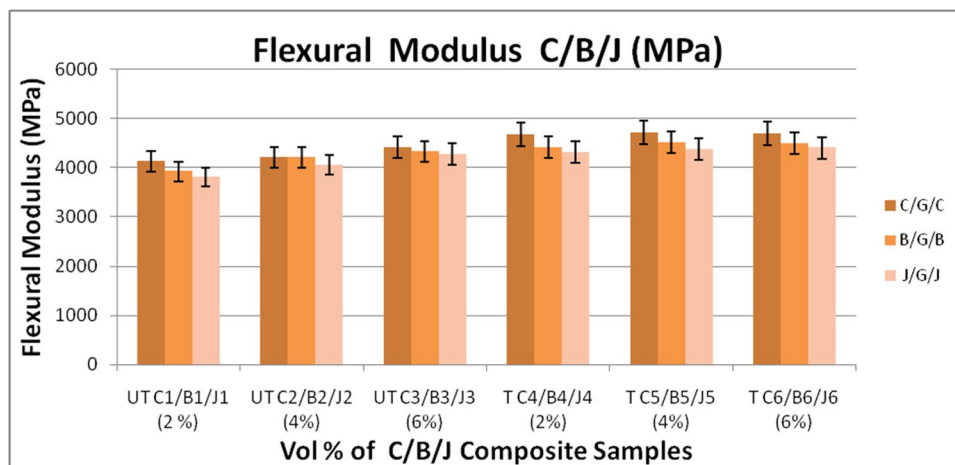
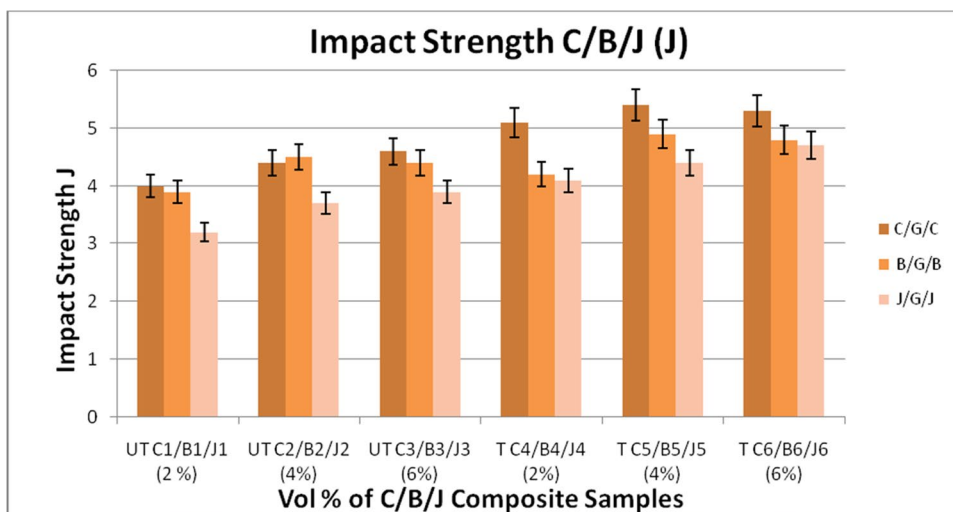


Fig. 17 Comparison of impact strength of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites



hybrid composites. The alkaline treatment changes the structure of cotton fibers by eliminating wax, lignin, and oil that act as a barrier between the fibers and the matrix increasing surface roughness and resulting in a better fiber-matrix interface [13].

Higher impact strength referred to the composite’s capacity to withstand energy [30]. This was attributed to a tight interfacial bond among the fiber, resin, and clay. The inclusion of nanoclay in C/G/C fiber mats/HR had functioned as fracture cover, enabling the fracture to expand in a more complex pattern and increasing the uptake of energy [3, 26].

3.5 Hardness

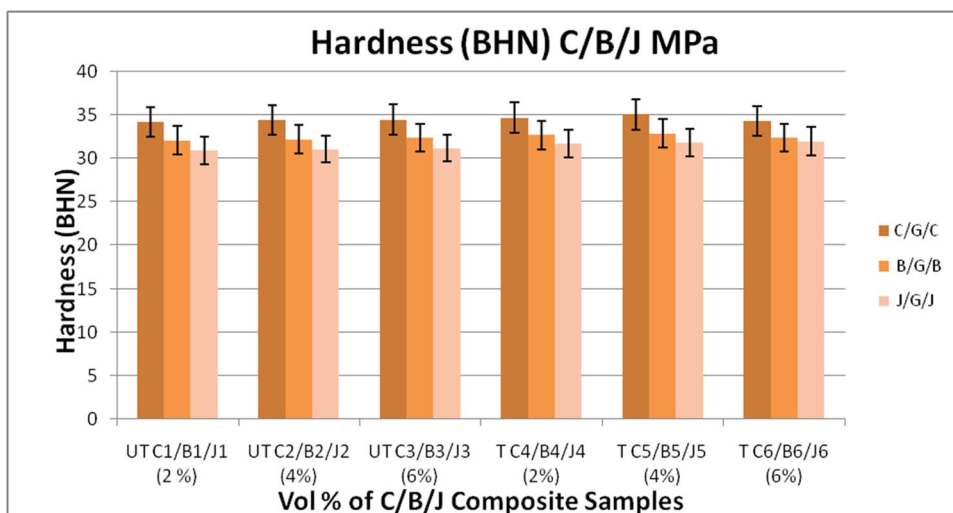
Figure 18 depicts the result of the hardness values. HR has a hardness value of 8.9 MPa. The hardness of treated C/G/C fiber mats with 4% nanoclay feeding in HR composites has reached the maximum value of 35.1 MPa. Hardness values for treated cotton fiber mats (C/G/C) 4% nanoclay feeding

in HR composites are greater than 6% in comparison with treated bamboo fiber mats (B/G/B) 4% nanoclay feeding in HR composites and 9.5% greater in comparison with treated jute fiber mats (J/G/J) 4% nanoclay feeding in HR composites. The greatest value is obtained due to the increase in the percentage of hard and brittle in treated C/G/C fiber mats 4% nanoclay feeding in HR composites, as revealed by the fiber, matrix, and clay compositions and also suggests that fiber with a wide surface area [26].

3.6 Differential scanning calorimeter

The DSC curves help to present the endothermic and exothermic processes of hybrid resin and hybrid composites as shown in Figs. 19, 20, and 21. The endothermic peak value known as glass transition temperature (Tg) is obtained for hybrid resin at a temperature of 75 °C, and the endothermic peak of hybrid-treated composites is increased in the range of 75 to 90 °C leads to the dehydration process in the fiber [31].

Fig. 18 Comparison of hardness of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites



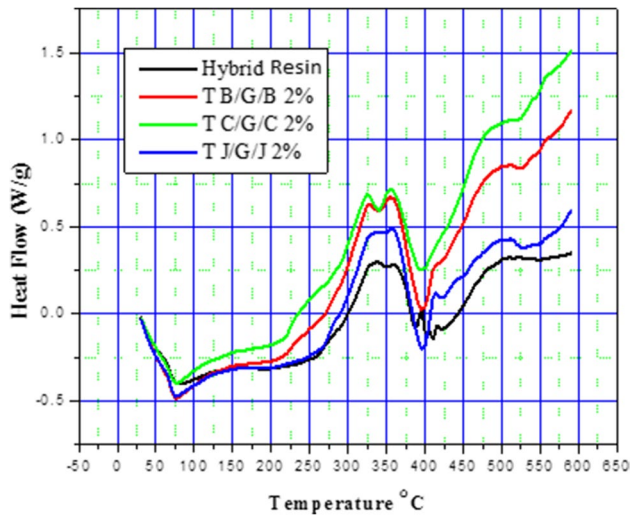


Fig. 19 DSC analysis of hybrid resin (HR) and treated C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR resin nanocomposites (2%)

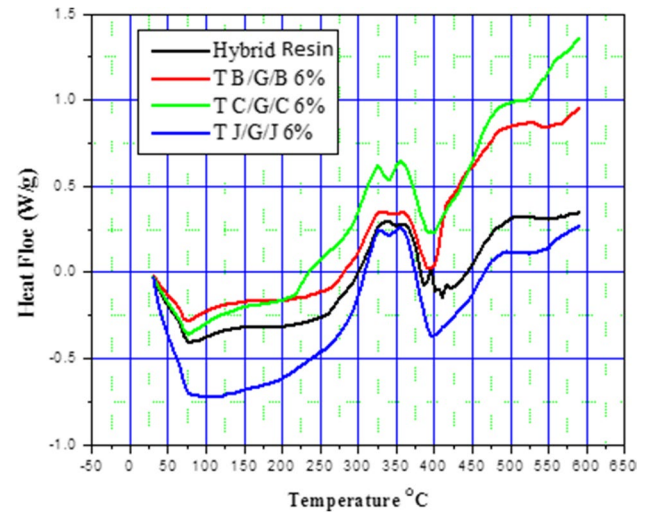


Fig. 21 DSC analysis of hybrid resin (HR) and treated C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR resin nanocomposites (6%)

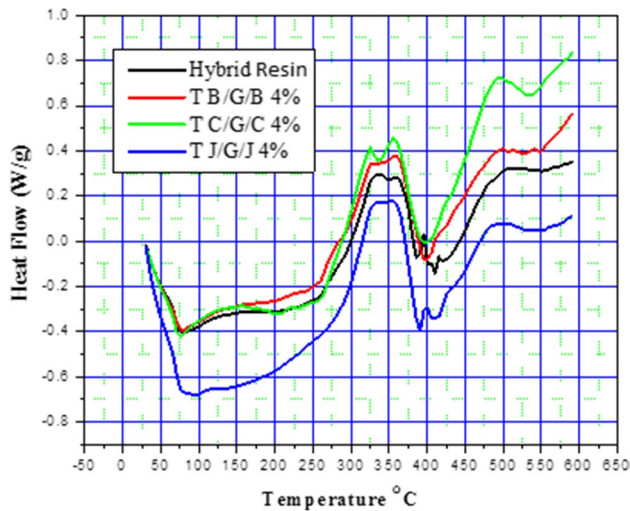


Fig. 20 DSC analysis of hybrid resin (HR) and treated C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR resin nanocomposites (4%)

In the range of 140 °C, the exothermic peak known as crystallization temperature (T_c) is obtained, which is attributed to the residual observed in DSC curves of the hybrid resin and also the exothermic peak of hybrid-treated composites is increased in the range of 140 to 150 °C. The melting point temperature of hybrid-treated composites is in the range of 170 to 205 °C, and the initial degradation temperature (T_d) is obtained for hybrid resin at a temperature of 312 °C and for hybrid composites is in the range of 318 to 360 °C, which is correlated to the degradation of cellulose in hybrid resin and hybrid-treated composites [32]. The next stage of degradation temperature is obtained for hybrid resin at a temperature of 375 °C, and hybrid-treated composites have the degradation temperature

in the range of 378 to 425 °C, respectively. The last stage of degradation temperature is obtained for hybrid resin at a temperature of 475 °C, and hybrid-treated composites have the maximum degradation temperature in the range of 480 to 580 °C and also indicate the maximum exothermic events that occur in the final decomposed product of the fibers, nanoclay, and the hybrid resin matrix. Due to the lack of hemicellulose in fibers, these results have represented the greater diffusion of nanoclay and resin inside the fibers treated with an alkali solution [33].

3.7 Thermogravimetric analysis

According to the findings as shown in Fig. 22, the treated hybrid CGC fiber reinforced with 2%, 4%, and 6% nanoclay (NC) feeding in every composition in *Lannea Coromandelica* blender epoxy composites has much greater thermal stability in comparison with hybrid resin and other treated hybrid composites. Heat causes evaporation of moisture in the specimen, and the beginning of the degradation in hemicellulose and cellulose has formed. All laminates have also indicated 10 to 20% weight loss percent between 310 and 359 °C. The vital elements of cellulose, hemicelluloses, and lignin are responsible for the severe weight loss percent between 360 and 499 °C [34]. The complete degradation of all combustible elements and the development of residue are observed in all specimens between 585 and 595 °C. When compared to other composites, the hybrid composite of treated C/G/C fiber mats feeding with 4 vol% nano clay in HR have the largest residual weight of 18.4% at 595 °C as shown in Table 2. This suggests that cotton fiber had greater

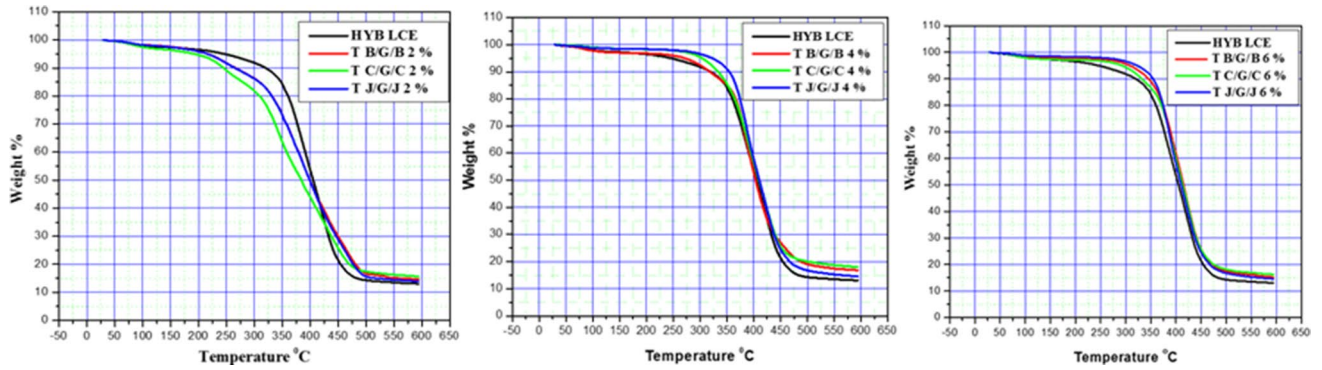


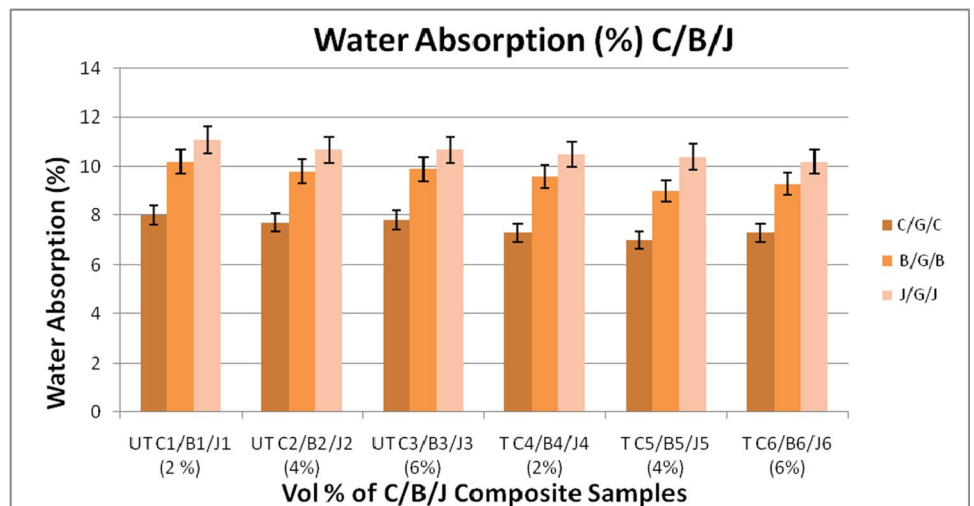
Fig. 22 TGA curves for C/G/C, B/G/B, and J/G/J with 2%, 4%, and 6% (NC) hybrid resin nanocomposites

Table 2 TGA values for treated C/G/C, B/G/B, and J/G/J with 2%, 4%, and 6% (NC) hybrid resin nanocomposites

Composition	Degradation temperature (°C) and Weight loss (%)				Residue weight % at 595 °C
	Initial degradation temperature IDT	Weight loss % at IDT	Final degradation temperature FDT	Weight loss % at FDT	
Hybrid resin (HR)	310	19.34	474	84.04	13.09
T C/G/C WRM+2 vol% NC	318	22.47	478	79.85	15.64
T B/G/B WRM+2 vol% NC	344	24.21	493	82.71	14.68
T J/G/J WRM+2 vol% NC	344	24.29	499	84.33	13.98
T C/G/C WRM+4 vol% NC	324	7.9	478	78.85	18.04
T B/G/B WRM+4 vol% NC	349	14.63	489	80.08	16.835
T J/G/J WRM+4 vol% NC	354	9.82	493	82.93	14.588
T C/G/C WRM+6 vol% NC	343	9.87	484	81.64	15.369
T B/G/B WRM+6 vol% NC	349	13.04	489	81.11	16.24
T J/G/J WRM+6 vol% NC	358	11.42	494	82.92	14.58

Bold indicate best material

Fig. 23 Comparison of water absorption of specimens (untreated and treated) of C/G/C, B/G/B, and J/G/J fiber mat-reinforced HR nanocomposites



experimental thermal stability than that of other hybrid polymer composites, documented in the literature [35].

3.8 Water absorption

Figure 23 depicts the water absorption in the percentage of hybrid composites. The minimum value obtained to absorb water in treated C/G/C fiber mats 4% nanoclay feeding in HR composites is 7% in comparison with other fiber composites. It is observed that untreated J/G/J fiber mats 2% nanoclay feeding in HR has a higher water absorption ability up to 11.1%. Natural fibers had a high-water absorption capacity in general [36]. The natural fiber resin matrix interface might also have an important contribution in the amount of water absorption in the percentage of hybrid composites and degradation of mechanical strength caused by the effects of water [37].

4 Conclusion

Hand-lay-up compression technique is used to fabricate locally available materials such as natural fibers (cotton fiber mats, bamboo fiber mats, and jute fiber mats), artificial fibers (glass fiber mat), hybrid resin (Lannea Coromandelica blended epoxy matrix) and bentonite nanoclay powder in various combinations to achieve the synthesis of environmentally and economically approachable biocomposites.

Hybrid nanocomposites and hybrid resin are produced in the present investigation. The thermal behavior of the produced composites is investigated using TGA and DSC. Some of main conclusions drawn from the analyses are as follows:

- In total, 18 compositions of hybrid composites are prepared. NaOH-treated 4% nanoclay filler-reinforced HR matrix in C/G/C fiber mats has better mechanical properties such as higher tensile strength (87 MPa), tensile modulus (3701 MPa), flexural strength (274.6 MPa), flexural modulus (4713 MPa), impact value (5.4 J), and hardness (35.1 MPa) compared to other fiber hybrid nanocomposites.
- SEM photograph also confirms for the same hybrid composite (NaOH-treated 4% nanoclay filler-reinforced HR matrix in C/G/C fiber mats) finding with no voids, uniform distribution, well trapped, and good interfacial bonding.
- TGA indicates higher thermal stability for the specimen of NaOH-treated 4% nanoclay filler-reinforced HR matrix in C/G/C fiber mats because it has the largest residual weight of 18.4% at 595 °C compared to other hybrid composites.
- DSC indicates a higher thermal stability for hybrid composites compared to hybrid resin because hybrid

composites have obtained the maximum degradation temperature due to the incorporation of nanoclay and fiber in the HR.

- In comparison with other hybrid fiber composites and hybrid resin, the minimum value obtained for water absorption in treated C/G/C fiber mats 4 vol% nanoclay mixed in HR composites is 7%. The NaOH treatment can effectively remove non-cellulosic materials in C/G/C fiber mats, which will reduce the water absorption and help to increase the mechanical strength of composites.
- Finally, among all of the hybrid composites, the hybrid composite (NaOH-treated 4% nanoclay filler-reinforced HR matrix in C/G/C fiber mats) has the best properties.

Author contributions P.Prabhu—Conceptualization, methodology, data curation, and writing of the original draft.

B. Karthikeyan—Supervision, review & editing, and validation.

R. Ravi Raja Malar Vannan—Investigation, formal analysis, and visualization

A. Balaji—Project administration and demonstration. The authors read and approved the final manuscript.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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