



Processing and Properties of Biodegradable Composites to Strengthen Structures

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Abstract Advantages of composite materials based on natural fibre have warranted increased utility of these products and have significantly offset the use of artificial fibre composites. This work on preparation of fibre-reinforced polymer (FRP) composites has been carried out by utilizing naturally occurring fibres from sisal, jute, banana, and ramie plants. Two sets of FRP composites have been prepared (1) by chemical treatment and (2) without chemical treatment. Detailed characterization of FRP composites prepared has been carried out. Detailed procedures for preparation of the samples have been explained. Parameters like tensile strength, flexural strength, water absorption, density, and microstructure analysis are performed. For density measurement, Archimedes concept using air and water weight measures and ASTM C830-00 guidelines have been followed. KIC-2-1000-C is employed for tensile tests, and digital weighing method is adopted for water absorption analysis. The FRP prepared have a great potential as natural fibre polymer composites (NFPCs) and constitute for a wide range of applications. A significant improvement in these properties has been obtained by heat and chemical treatment of these composites. Satisfactory improvements of the properties were noticed with heat and chemically treated fibre-reinforced composites. Tensile and flexural strength tests were carried out on untreated and treated sisal, jute, ramie and banana fibre-reinforced composites, which revealed significant improvement of treated composites (tensile

strength 73.59, 41.70, 36.30 & 33.87 N/mm² and flexural strength 60.77, 40.47, 72.05 and 31.54 N/mm², respectively) as compared to untreated composites (tensile strength 57.24, 37.72, 35.19 and 21.83 N/mm² and flexural strength 50.51, 40.27, 41.77 and 21.35 N/mm² respectively).

Keywords Sisal fibre · Jute fibre · Ramie fibre · Banana fibre · Tensile strength · Flexural strength

Introduction

Composite materials of FRP are more durable, lighter in weight and have higher strength-to-weight ratio than traditional reinforcement materials such as steel. Natural fibres like kenaf, sisal, abaca, silk, etc., are available and used for this purpose. Natural fibre polymer composites (NFPCs) are a neoteric category of polymers that has a strong ability to replace wood in the future and are mainly focused on building applications. The mechanical properties of composites depend on the characteristics of the fibre, matrix, fibre–matrix bond, the quantity of the fibre and its alignment [1, 2]. NFPCs can be conveniently reprocessed than glass or carbon material. It is 10–30% lighter by weight as compared to other conventional materials and also has an outstanding acoustic absorbing characteristic [3]. Another significant factor shaping the future growth potential of NFPC is the commitment of researchers to discover innovative ways to boost the quality of natural fibres to the highest level while taking into account the state of equilibrium between sustainable development, finance and efficiency. Significant factors that decide the commercial performance of NFPCs are shown in Fig. 1.

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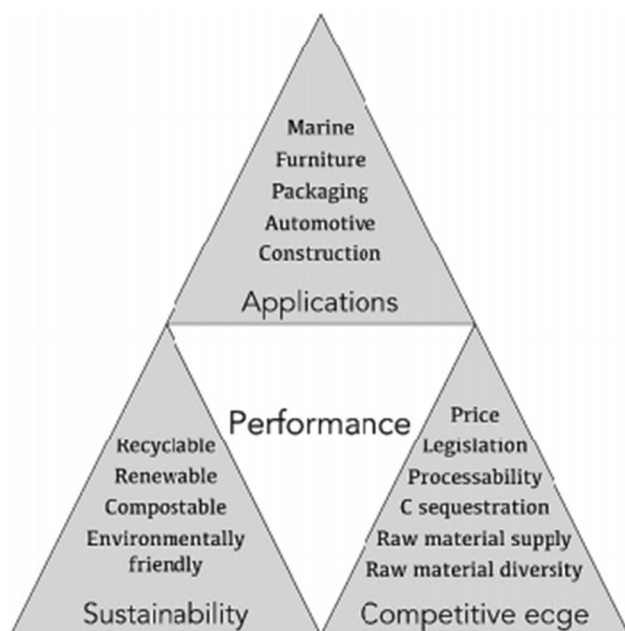


Fig. 1 The triangle of commercial success for NFPCs [4]

The durability of the composite material largely depends on the modulus and strength of the fibres, chemical strength of the matrix, the efficacy of bonding intensity among the matrix and the fibres in stress transfer at the interface [5]. In FRPs, increase in mats grammage encourages a heavy increase in ultimate load, regained energy, perforation impact energy, as well as a marked decrease in deformation [6]. Depending on the hardness, each fibre shows their own fracture work [7]. Strong fibres with large strain of failure impose large fracture work on composites. With the rise in its fibre volume fraction, the tensile and flexural strength increases. The impact strength rises with the rise in fibre loading to some extent. The mechanical properties rise with the rise in thickness in small arbitrarily aligned FRP composites [8]. As the FRP composite thickness increases, the rate of water absorption and fire resistance also rises [9]. However, rising fraction of the fibre volume results in reduction of linear density rate. Glass and banana fibre-reinforced hybrid epoxy composites were also analysed [10] and found that the mechanical properties of sample fibre with 10 mm length and 15 percent fibre loading were stronger. Relative to the remainder of the specimens, composite of bamboo-banana epoxy resin displayed greater impact strength, while the composite of bamboo epoxy resin recorded the minimum impact strength. Maximum hardness was observed in bamboo linen epoxy resin composite, while minimum hardness was found in bamboo epoxy resin [11]. A relatively larger tensile strength was found for optimum loading value of roselle fibre in the matrix, which is enhanced by 136 percent relative to the

remainder of the fibre loading percentage [12–14]. The thermal analysis showed enhancements in the residual content of the composite materials, thereby improving the thermal stability. There was no major difference seen in the degradation temperature. Naveen et al. [15] noticed in their study that the coir fibres with 5 % by its volume showed a significant result compared to the rest of the volume used due to the effect of material stiffness. Currently, the usage of natural fibres has tremendously increased to enhance the strength and stiffness of structural members. Tidarut et al. [16] investigated concrete cylinders confined with natural fibres, which are jute, hemp and cotton natural fibre-reinforced polymers. In their study, mechanical properties of NFPC confined concrete were intensively evaluated. They have successfully proved that NFPC is effective and suitable to enhance the confinement effect of concrete. Bahja et al. [17] studied the effect of different treatment procedures for the betterment of properties of the sisal fibres and their applicability with cement mortar. Their results indicated treated fibres had better properties (high crystallinity index, high thermal resistance, eliminates hemicelluloses and pectin made parts) than untreated ones. Also they have noticed a reduction in flexural and compressive strengths of sisal fibre-reinforced cement mortar composites compared to the control cement mortar, and this may be because of decrease in the density of the mortar due to the increase in its porosity.

In recent developments, NFPCs are being used to rehabilitate the deficiencies and strengthen the existing structure [13, 18, 19], which is considered as an effective external strengthening material, due to their outstanding performances like high strength-to-weight ratio, lightness and resistance to corrosion. This has piloted a new technique which has great potential in the field of upgrading structures. This helps in load carrying as a composite member with concrete. Limited research on various types of natural fibres for the retrofitting of structures has been reported in the literature. Preferable experimental studies of local species of many other natural fibres are still necessary, particularly as reinforced concrete for structural elements. Therefore, by retrofitting, it will become the latent replacement of the expensive and unsustainable synthetic fibre for existing structures. Use of banana, sisal, jute and ramie fabrics composites is meagre in the literature. This gap has motivated the present work to prepare a composite material from these materials. The preparation, mechanical characterization, analysis is hence performed in detail for these materials. The above issue of lack of research work in the area of local species is addressed in this work by using sisal, jute, banana and ramie fabrics to prepare composite materials and is based on the experimental results obtained from previous studies conducted by the investigators [20]. It was decided to use fabrics such as

sisal, jute, ramie and banana fabrics for preparation of composites to enhance the properties of the NFPCs.

Materials and Methods

Raw Materials

The raw materials used here are: sisal, jute, banana and ramie fabrics. Figure 2 shows the plants, their extracted fibres and micrographs. K-6 hardener and LAPOX L-12 (epoxy resin) are employed. In Table 1, the specifications of resin and hardener are provided. The subject hardener and epoxy resin have been procured from Yuje Enterprises (Bengaluru, India). Fabrics are purchased from Extra Weave Private Ltd., Cherthala (India). The sisal plant, jute plant, banana plant and ramie plant are shown in Fig. 2a, and their respective fibres in Fig. 2b. SEM micrographs of the typical section of the fibres are shown in Fig. 2c.

Pre-treating Natural Fibres

For mechanical testing of all fabrics and their alkali treatment, they were made into pieces as per the standard method. 4 % NaOH solution was taken in which the cut pieces of fabrics were immersed at normal temperature (room temperature), allowing sufficient soaking time. Following this, the fabric was washed using clear water to eliminate any NaOH. The remainder is neutralized using dilute acetic acid. Later, the fabrics treated with NaOH are cleaned once more using distilled water, confirming to maintain a final pH of 7 [21, 22]. In Fig. 3, the procedure adapted for alkali treatment of this fibre is shown. As revealed in Fig. 3c, at room temperature the fabric is dried for 2 complete days and then dried in an oven for 1 complete day at 50 °C. Heat treatment is a physical process that results in the modification of fibre surface morphology by increasing cellulose crystallinity due to the rearrangement of molecular structure at high temperatures. Chemical reactions are triggered and intensified by high temperatures. Heat treatment of cellulosic fabrics, such as sisal and jute natural fibres, resulted in thermal modifications of cellulose, which caused changes in lignin and hemicelluloses [23], resulting in the formation of hydroxyl groups and an increase in the number of carbon–carbon double bonds. It is the carbon–carbon double bonds which enhanced cross linkages and further lead to the formation of radicals [24].

Composite Fabrication

The pre-treated and untreated fabrics of natural fibres were cut in sizes, as per the requirement, which are all utilized

for the composite FRP fabrication. A mould made of wood is employed having appropriate dimension to prepare the composite FRP plates. To prepare the sample, the method of hand lay-up was followed. Matrix of epoxy resin with vol. fraction 30 % is considered. A thoroughly mixture of epoxy resin and hardener in the ratio of 70:30 by volume was mixed by stirring gently in order to reduce the entrapment of air. The dielectric strength of gases is much lower than dielectric strength of water, and partial discharge may occur in the air bubbles at the lower field strengths than is necessary for a breakdown of water. The interest in the insulating properties of two-phase media arose due to their damping properties. Propagation of perturbations of various amplitudes in gas-liquid media has been studied extensively since the mid-twentieth century. Therefore, research in dependency of the breakdown voltage of water on concentration of air bubbles of different gases (air, sulphur hexafluoride, inert gases) is employed.

To easily remove the mould, an agent was used for the composite plates as depicted in Fig. 4. The release agent, i.e. silicone grease, was used on the inner mould surface underneath the electrical insulating paper for easy removal. A thin layer of epoxy and hardener mixture was poured after the mould was deposited on the insulating sheet. The fabric was then put on the mixture of the resin. On top of the fabric, epoxy hardener mixture was then poured into the mould to form alternate layers of mixture and fabric. Ample care has been ensured to prevent air bubble formation. In the next step, electric insulating paper was applied over the manufactured FRPs and weights were placed on it, a pressure of 0.2616 kgf/cm² was attained, to ensure no air bubbles remained in the reinforced polymer epoxy fibre mix. At room temperature, it was allowed to heal for 48 hours with the applied pressure on top of the composite. This entire process is schematically illustrated in Fig. 5. The sewing and final preparation of fibre mats are shown in Fig. 6 for the purpose of illustration.

Characterization

Density

The naturally occurring fibre density has been estimated with the Archimedes concept using air and water weight measures by following ASTM C830-00 guidelines and employing a 0.1 mg precision balance. Consideration was given to masses of fibre in dry and wet condition. For a certain time, the mass of dried fibre was weighed and then the fibre was submerged in water. Saturated weight of the fibre was then weighed.

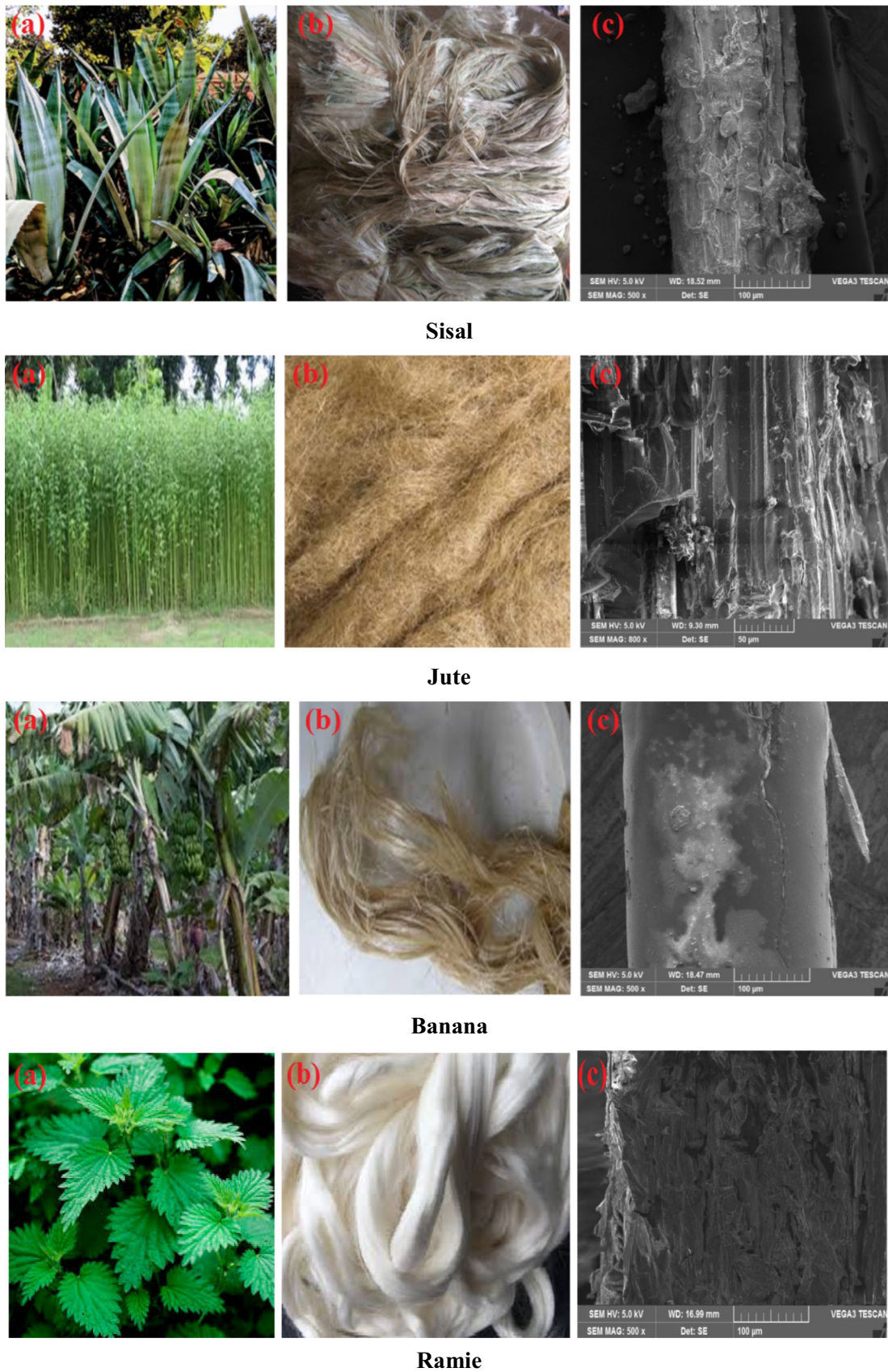


Fig. 2 a Natural fibre plant, b extracted fibre, c scanning electron micrograph

Water Absorption Test

According to the ASTM D570 standard, the test of water absorption was performed. Thoroughly dried specimen was weighed using a digital scale (mass m_1) and then immersed

in beaker containing water. After 12 h, the sample was weighed (mass m_2) after removing from the water. Over 3 days, this process of water absorption by the specimens was repeated to estimate their weights. The % water absorption was computed with the difference in weight between samples considering before and after the immersion in water. The % absorption is given according to:

$$(\%) \text{absorption} = ((W_{at} - W_{ao}) / W_{ao}) * 100$$

The term W_{at} is the specimen weight in grams after taking out from water, and W_{ao} is the specimen weight before soaking in water. Using simple mass balance experimentation, the weight of the specimen was calculated having a precision of 0.0001 g.

Table 1 Hardener and resin specifications

Material	Chemical name	Trade name	Density (g/cm ³)
Resin	Diglycidyl Ether Bisphenol A (DGEBA)	LAPOX L12	1.12
Hardener	Triethylene Tetro Amine (TETA)	0.954 K-6	0.955

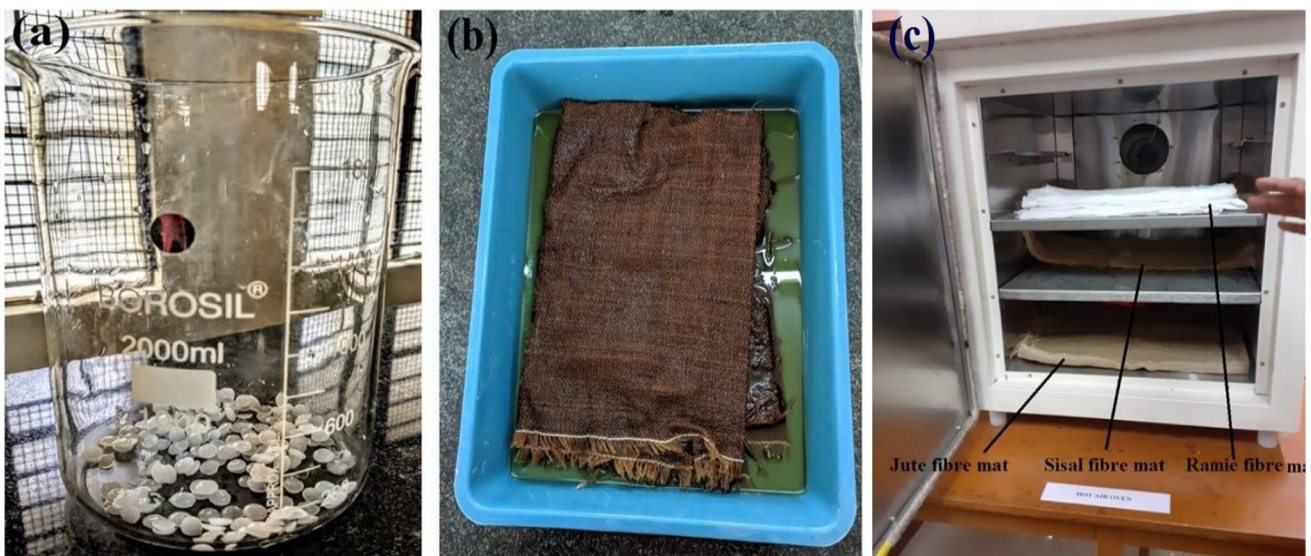


Fig. 3 Alkali treatment of fibre fabric **a** NaOH tablets to prepare 4% NaOH, **b** jute mat immersed in 4% NaOH solution, **c** heat treatment of fibre mats in an oven for 24 h at 50 °C



Fig. 4 Chemicals used in the study **a** epoxy resin—LAPOX L-12, **b** hardner—Lapox K6, **c** releasing agent—polyvinyl alcohol]



Fig. 5 Stepwise procedure followed in the study for preparation of composites by hand layup process **a** fibre mat, **b** releasing agent applied on the surface, **c** resin application, **d** application of insulating paper, **e** alternative layers of fibre and resin placed, **f** load applied

Mechanical Properties

Employing KIC-2-1000-C with capacity of 100 kN (Universal Testing Machine, Make: Kalpak Instruments and Controls, Pune), the composite's sample tensile strength test was carried out as per ISO 527-4:1997(E) Part-4. Using the same machine, the flexural strength was calculated as per ISO 14125:1998. For each

composition, a total of three samples were prepared and tested. Average of three test results obtained for flexural and tensile strength is utilized for further analysis. Loading rate of samples during these tests was 2 mm/min. The mechanism of sample failure and microstructure analysis is performed using the Vega 3 TSE (Tescan Scanning Electron) microscope operated at 26 KV. As per the ISO 527-4:1997(E) Part-4 standards, the specimens for tensile

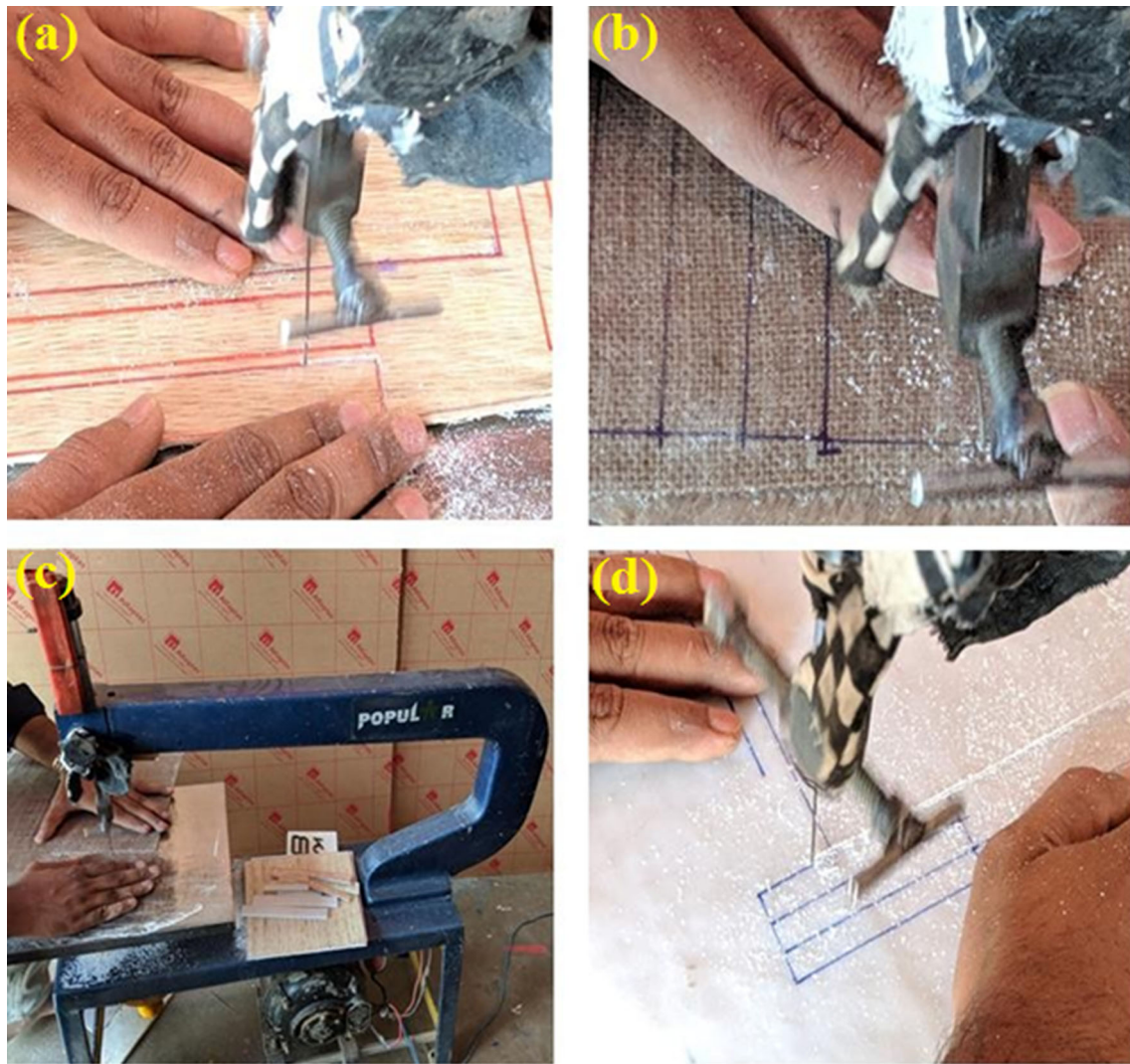


Fig. 6 Specimen preparation for tensile and flexural test as per standards **a** sisal/epoxy mat, **b** Jute/epoxy mat, **c** banana/epoxy mat, **d** ramie/epoxy mat

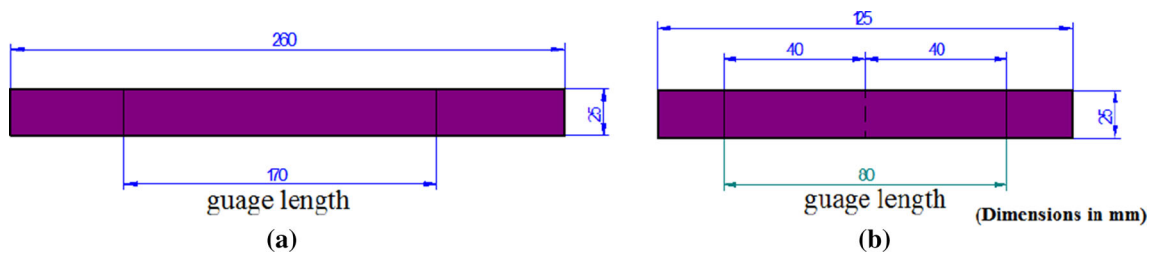


Fig. 7 Details of **a** the tensile test specimen **b** the flexural test specimen

tests were organized as depicted in Fig. 7a. Uni-axial load was applied at both the ends of samples using specific jaw arrangements in the testing machine. Since fibres have been classified as Class II type material, all the limitations of the

sample proportions for flexural testing of class II type material were practiced as per the ISO 14125:1998 code. Details of the specimen are shown in Fig. 7b.

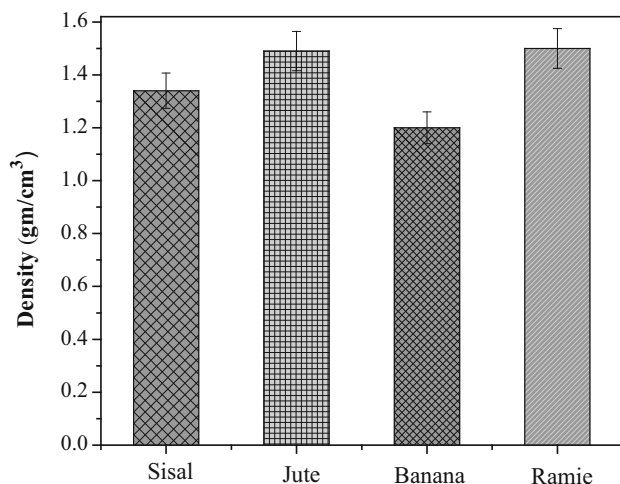


Fig. 8 Density characteristic of natural fibres

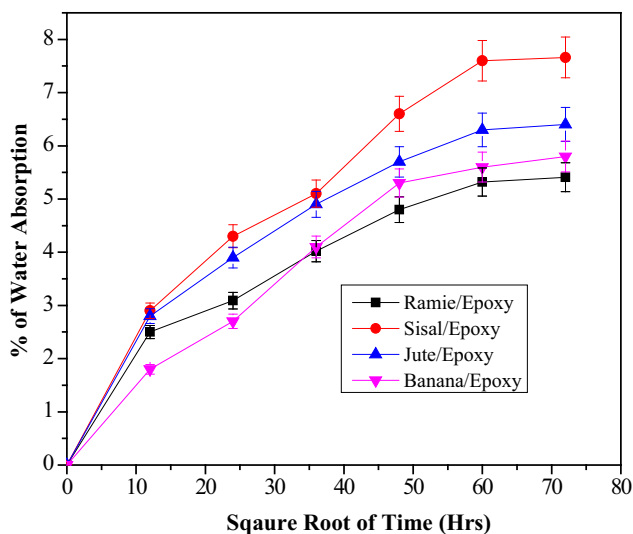


Fig. 9 Water absorption characteristics of composites

Results and Discussion

Density

The densities of sisal, jute, banana and ramie were calculated to be 1.34, 1.49, 1.2 and 1.5 g/cm³, respectively, as shown in Fig. 8. The calculated density values for Sisal, Jute and Ramie were found to be in agreement with literature values (1.32–1.5 g/cm³ [21] for sisal; 1.51 g/cm³ [22] for jute; 1.5 g/cm³ [21] for ramie), whereas banana fibres (1.35 g/cm³ [25]) were found to be slightly lower.

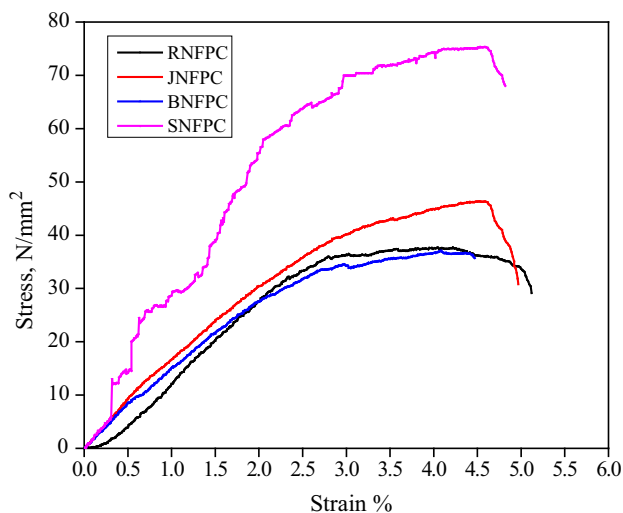


Fig. 10 Tensile stress–strain curve for different composite samples, recorded during tensile test

Water Absorption

Figure 9 shows the relation between the immersion time in hours and water absorption in percentage. Readings were obtained periodically for every 12 h for a total of 72 h. It can be noticed that the absorption of water gradually increases in accordance with increase in immersion time (hours) in all the combinations. The hydrophilic characteristic of sisal fibre caused the sisal/epoxy composite to absorb a larger amount than others. As shown in Fig. 9, the water absorption trend in later stages of the immersion (80 h) was similar to that in early stages, indicating that the mass gain of all specimens due to the hydrolysis of epoxy resin was more than the mass gain of water absorption in later stage. The loss of mass for thinner specimen (Sisal) was greater than that of thicker specimens (Ramie and Banana) in later stages, meaning that a thinner specimen had a greater hydrolysis degree at the given ageing time. It is noteworthy that the water absorption of these laminates was studied by considering the epoxy resin and the natural plant fibre as a whole in this study. It is significant to determine water absorptions of the resin and natural plant fibre, respectively, for a better understanding of the water absorption mechanism [26, 27].

Tensile Strength

Tensile stress–strain curve for different composite samples is shown in Fig. 10. The tensile strength values are tabulated in Table 2. The variation of tensile strength of epoxy/natural composites is illustrated in Fig. 11. It is seen that the composites reinforced by treated fibres produce

Table 2 Tensile and flexural strength of different composites

Composites	Tensile strength (MPa)						Flexural strength (MPa)					
	Untreated			Treated			Untreated			Treated		
	T1	T2	Avg	T1	T2	Avg	T1	T2	Avg	T1	T2	Avg
SNFPC	64.09	50.39	57.24	73.18	74.01	73.59	56.24	44.94	50.59	66.09	55.45	60.77
JNFPC	36.38	39.07	37.72	37.19	46.22	41.70	42.59	37.96	40.27	34.26	46.67	40.47
RNFPC	37.39	32.98	35.19	38.5	34.1	36.30	48.32	35.22	41.77	68.76	75.34	72.05
BNFPC	20.69	22.97	21.83	33.73	34	33.87	21.85	20.85	21.35	32.43	30.66	31.54

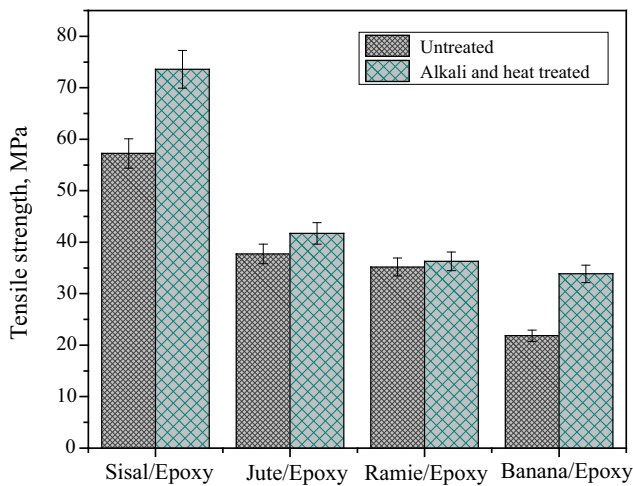


Fig. 11 Tensile strength of fabricated fibre/epoxy composites

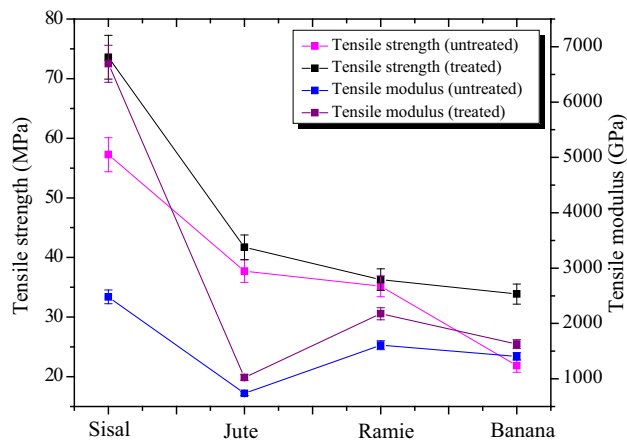


Fig. 12 Tensile strength and modulus for fibre/epoxy composites in untreated condition and treated condition

relatively improved properties in all the cases. This is because the alkali treatment eliminates matrix incompatible waxes (Tyloses) and impurities from the fibre surface and gives a rough surface for improved interlocking. The

natural fibres were treated with maleic anhydride. The fibres were placed in a flask equipped with water condensers. The calculated amount of maleic anhydride was added to the flask containing fibres and refluxed on a heating mantle that maintained a temperature of 120 °C for 2 h. Processing with the maleic anhydride binding agent eventually breaks down the fibre structure. In Fig. 12, the untreated fibre’s tensile strength and modulus are represented for comparison. The tensile strength of the composite with chemical and heat treatment increases by 28.56%, 10.55%, 2.84%, and 55.15%, respectively, for sisal (SNFPC), jute (JNFPC), ramie (RNFPC) and banana fibre-reinforced polymer composites (BNFPC) as shown in Fig. 12. As is evident from Fig. 12, there is a slight increase in tensile strength of the composite, adjusted with the chemical treatment, which is primarily because of the implementation of the inorganic compound ‘sodium hydroxide’. It enhances the interaction between the fibre and the resin matrix. Therefore, the tensile strength of the composites processed with chemicals and heat increases more dramatically as compared to untreated composite fibres. It is evident that a composite when fabricated using jute, banana, sisal, and ramie with epoxy matrix exhibits the best tensile strength among the prepared lot. Thus, this FRP composite is incorporated with heat-treated material to check its tensile properties. Also, it can be observed that with the incorporation of alkali and heat treatment epoxy composites, an increment of nearly 43% in tensile strength has taken place. This increment on addition of other bio-waste can be attributed to the improvement in transfer of stress from matrix phase to the reinforced material. Figure 13 shows the test specimens before and after the tensile test.

A detailed SEM micrograph assessment was conducted on the tensile fractured surfaces of the specimens studied which showed the disappearance of (a) phenomena of debonding, (b) pull-out and delamination, (c) confirming strong bonding of the fibre matrix or interface as depicted in Fig. 14a–d. The fibre is not quickly drawn out of the matrix, if it gets an external force. It is also observed that

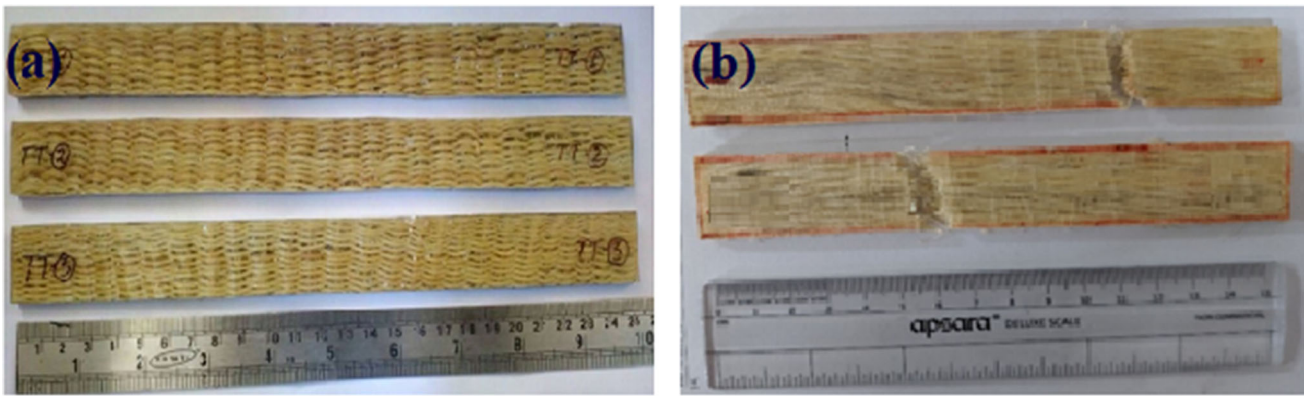


Fig. 13 Tensile test specimen of sisal fibre-reinforced epoxy composites **a** before and **b** after the test

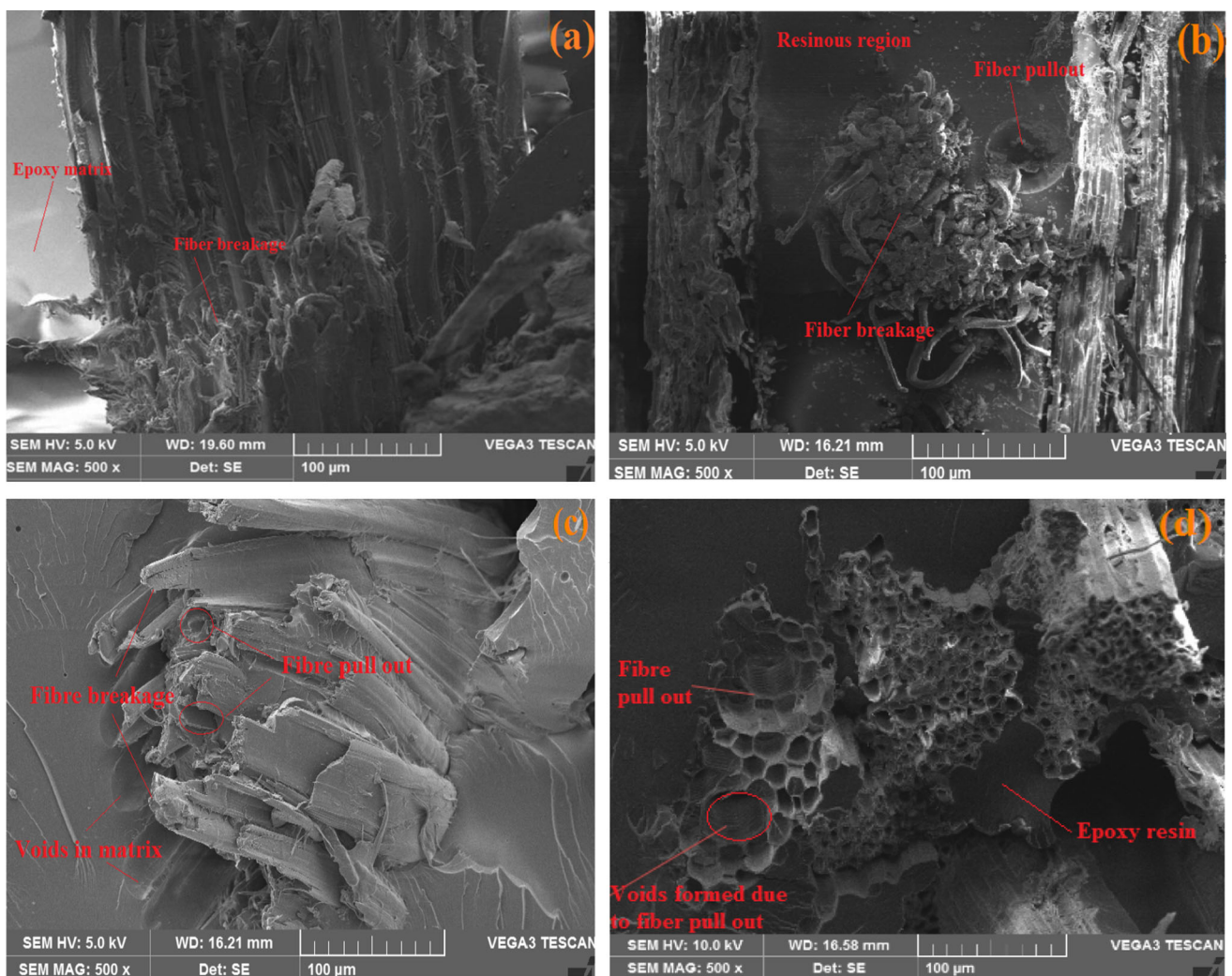


Fig. 14 Fractography of **a** sisal/epoxy **b** jute/epoxy **c** ramie/epoxy **d** banana/epoxy composite after tensile test

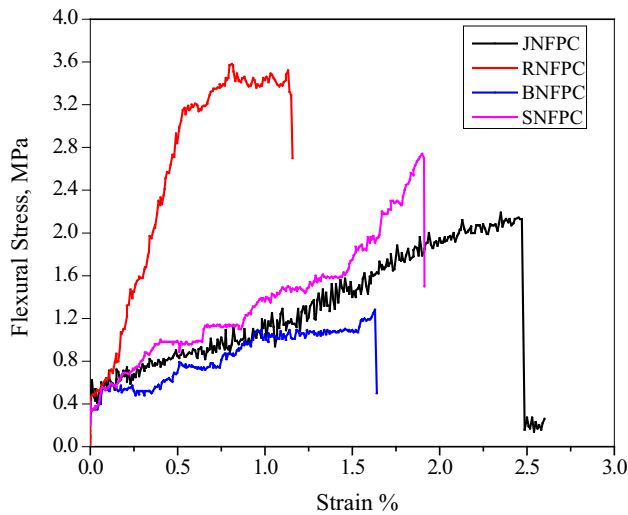


Fig. 15 Flexural stress–strain curve of different composite samples, recorded during flexural test

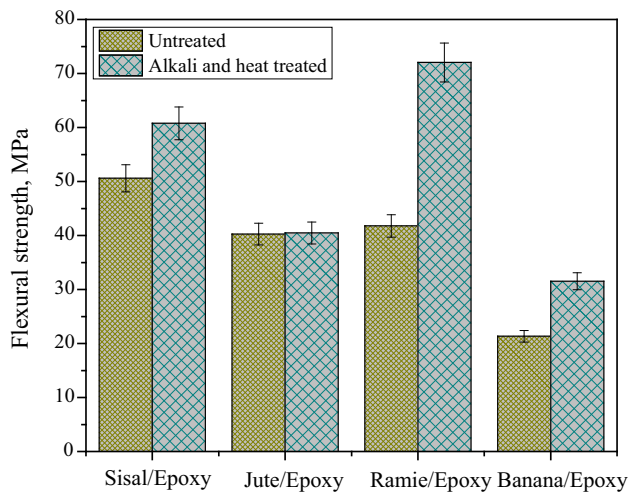


Fig. 16 Flexural strength of fibre/epoxy composites

the holes are not visible in the composites subjected to the combined treatment as several fibres and the matrix are bonded together. Thus, the best interface is for this composite. It is deduced from the figure that the load transfer between the fibres and the matrix is reduced by a weak matrix bond. It will therefore lead to poor resistance and delamination among layer patterns. Different researchers have studied the impact of weak fibre/matrix mechanism on mechanical activity [28, 29]. In addition, this bad relation between the fibres, which is verified by the presence of so many vacuums presented in Fig. 14d, could not sustain the fibre in its place.

Flexural Strength

Figure 15 shows flexural stress–strain curve for different composite samples after a three-point bending test. Depending on flexural testing, Fig. 16 was drawn to assess the ability of composites to resist bending before breakage happens. In all the cases, treated fibre-reinforced composites resulted with comparatively better properties. The flexural strength increases by 20.12%, 1%, 72%, and 47.72%, respectively and modulus increases by 19.55%, 47.55%, 87.9%, and 69.9%, respectively, for sisal, jute, ramie and banana fibre-reinforced composites. This may be because the alkali treatment eliminates waxy substances from the fibre exterior and gives a rough surface for improved interlocking. Processing with the maleic anhydride binding agent eventually breaks down the fibre structure. The banana fibre showed the lowest flexural strength out of all composites. Due to the hybridization, there was not much improvement compared to pure fibre-reinforced composites. Consequently, it will lead to an earlier delamination failure between each layer of fibre composites. But in the case of pure fibre composites (CCC, BBB, SSS), it is possible to achieve a better compatibility and dispersion than hybrids and also the earlier delamination is comparatively minimum.

Figure 17 shows the flexural strength and corresponding flexural moduli for composites reinforced with untreated and treated fibres. The characteristics of the chemical and heat-treated composites are improved more significantly, as compared to the raw composite’s fibre. Figure 18 indicates specimens before and after test. This increase may have been the combination of improvement in transfer of stress from matrix phase to fibre reinforcement along with advancement in adhesion property between epoxy and jute fibre.

Figure 19a–d presents the fracture exterior look of the composite specimens examined for flexural strength. The examination of the SEM micrographs demonstrates the lack of debonding and very few pull-outs. The fracture exterior of epoxy composites is actually identified by the occurrence of precisely fragmented fibres on the fracture plane. Few of the holes are produced by the fibre pull-out, mostly due to low integration occurring from the untreated fibre hydrophilicity and the polypropylene matrix hydrophobicity. The following micrographs suggest that the holes are decreased and many fibre cracks could be observed after the chemical agent adjustment, suggesting that the composite has a strong interface. This indicates the fibre–matrix detachment and breakage of fibre. The epoxy composite with four-layer jute fibre exhibits as the best material that is capable of resisting maximum amount of flexural load as seen in Fig. 19. With the increase in weight fraction of the jute fibre in the composites, the ability to

Fig. 17 Flexural strength and moduli for fibre/epoxy composites in untreated and treated condition

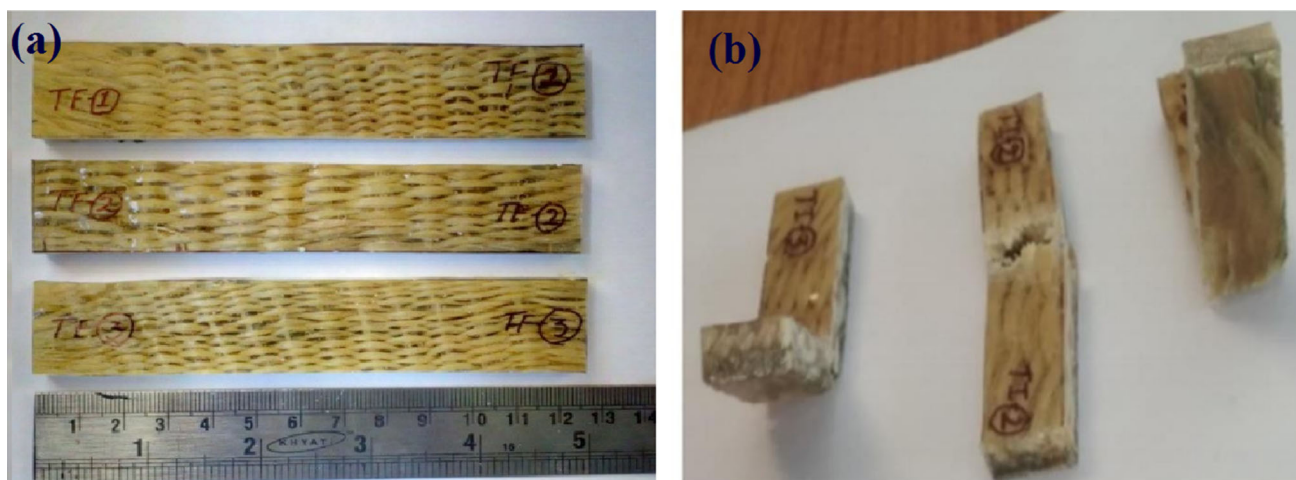
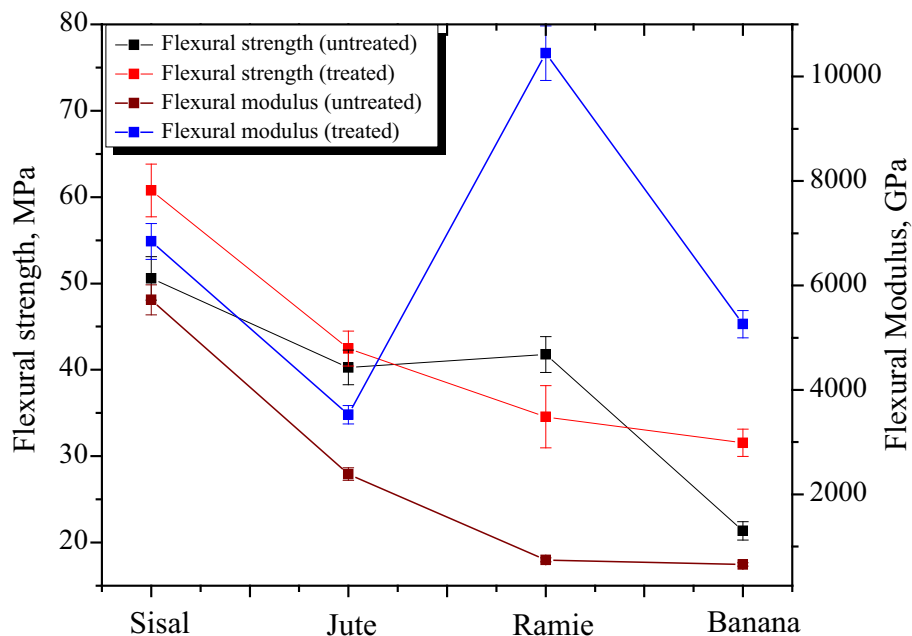


Fig. 18 Flexural test specimen of sisal fibre-reinforced epoxy composites **a** before and **b** after the test

absorb greater flexural peak load also increases. Due to lack of proper impregnation of epoxy in this composite, flexural strength decreases phenomenally.

Cleaner Production Approach of Developed Composites

Cleaner perception is not only aimed at minimizing waste components but also enhancing the efficiency of produced materials. This is achieved in the current research, which shows better performance of manufactured hybrid composites. These composites improved mechanical characteristics enabling them for use in lightweight cars and

structural projects. The results obtained from the NFPC show the possibility in further improvement of composite property. The final composite produced through the use of natural fibre provides cleaner processing, sustainable development and environmental protection. While the work at hand showed that NFPCs are viable, several restrictions remain to be addressed. However, the use of natural fibre in polymers poses many difficulties, such as excess water absorption and low thermal stability, which have to be addressed in the manufacture of similar materials to traditional composites.

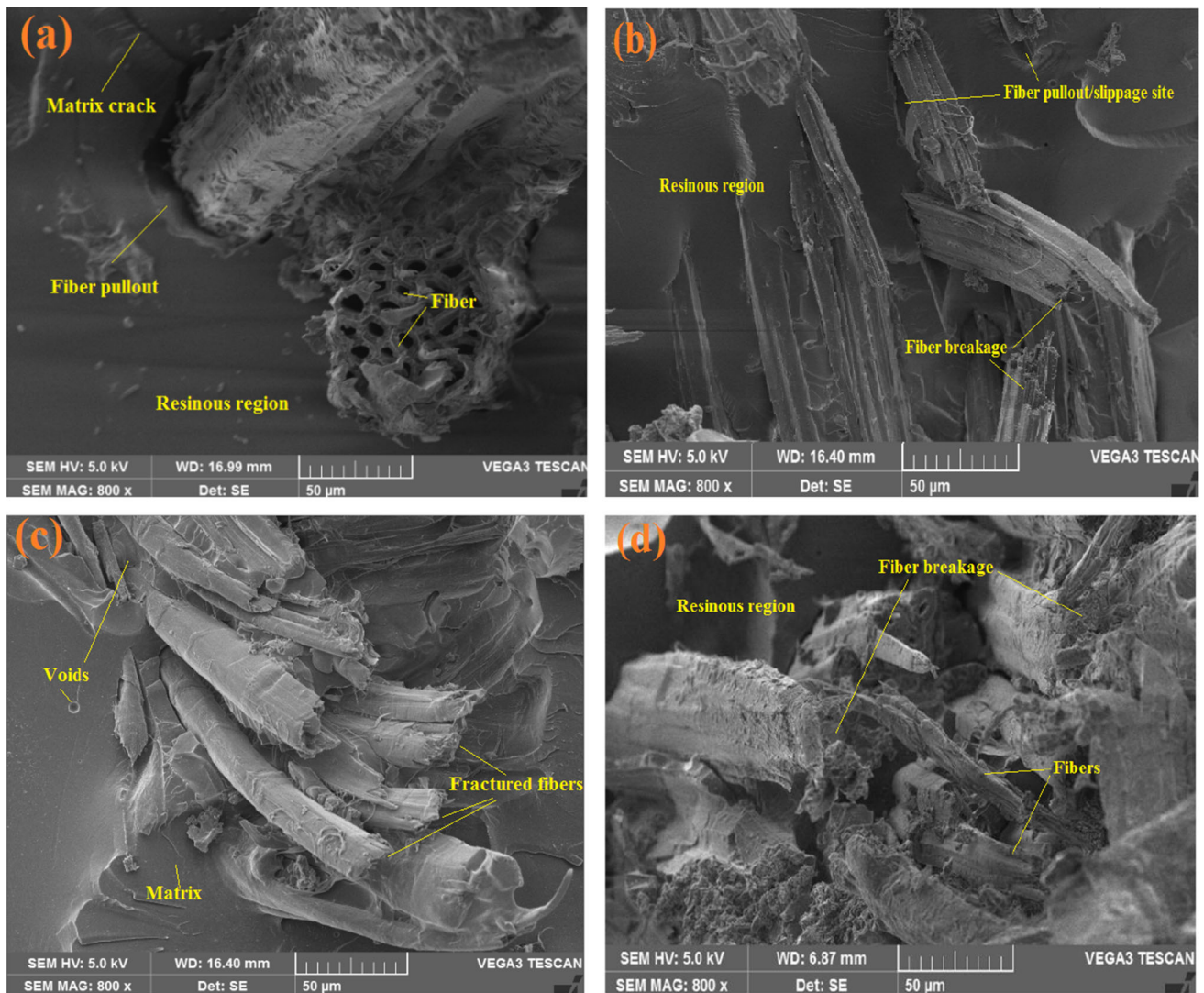


Fig. 19 Fractography of **a** sisal/epoxy, **b** jute/epoxy, **c** ramie/epoxy, **d** banana/epoxy composite after flexural test

Conclusions

The work presented is pertinent to preparation of natural fibre-reinforced polymer using bio-natural fibres and testing of different characteristics. Characterization in terms of tensile strength, flexural strength, water absorption, density and microstructure analysis are performed in detail. The following are the important conclusions drawn:

- The heat- and alkali-treated samples showed a greater improvement in their properties compared to untreated samples.
- The alkali and heat treatment natural fibre-reinforced polymer influences the performance of composites on both static and dynamic characteristics. Among all, the alkali -treated sisal and ramie combination is found to

be the optimal composite and has offered better tensile, flexural, and absorption properties.

- The FRP prepared have great potential as natural fibre polymer composites (NFPC's) and constitutes to be used in wide range of applications. The present study shows that the bio-fibre-epoxy composites could be a competitive material in mechanical properties for specific application. Though strength of fibre composites may be lower as compared to the glass fibre-reinforced composites, their lower density, biodegradability and cost make them comparable with glass fibre composite in terms of specific and economic values.

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