



Effects of layering variation on mechanical, thermal, and morphological properties of areca natural fiber mat reinforced epoxy biocomposites

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Abstract

Recent developments in reinforced plastics for a range of engineering applications have utilized natural fiber mat as a reinforcing material. The goal of the current work is to create a biocomposite material by adding a natural fiber mat with polymer. Areca fiber mats were combined with epoxy to fabricate five areca fiber mat-based biocomposites via the hand lay-up technique. These areca fiber mats were reinforced with epoxy in the number of mat layers or weight of fiber mat varying (0, 1, 2, 3, and 4). Tensile, flexural, and impact strengths of the manufactured areca fiber mat composite were investigated. We used SEM to conduct a morphological examination on specimens that had undergone tensile and flexural fracture. The thermogravimetric analysis (TGA) method was used to study the thermal strength of the novel areca fiber mat composites. We also conducted experiments on water absorption and biodegradability. The results indicated that the morphologies of the composites enhanced the mechanical characteristics by increasing the bonding between the epoxy and areca fiber mat. The three-layer areca fiber mat composite has better mechanical strength (tensile 41.8 MPa, flexural 192 MPa, and impact 2.9 J) and thermal qualities (highest thermal stability 17.9 %) than the other four composites. SEM scans also support the areca fiber mat composite.

Keywords Biocomposites · Areca fiber mat · Morphological · Water absorption · Biodegradability

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1 Introduction

The modern need for biomaterials in precise automobile industrial applications has led to a surge in research focused on various forms of biocomposite materials, including fibers, particles, and mats [1, 2]. Because it has got several key advantages that contribute to both environmental sustainability and improved product performance, some of these advantages include being renewable and sustainable, reducing environmental impact, being lightweight and cost-effective, and having good thermal insulation, biodegradability, low-energy manufacturing, etc. Long, thin strands called “natural fibers” come from either plants or animals [3]. On the other hand, the term “particles” designates tiny, solid fragments of various sizes and compositions [4]. Natural materials like grasses, reeds, or bamboo are weaved or layered to create natural mats [5]. Unique characteristics including comfort, breathability, and biodegradability are offered by natural fibers [6]. They come in a variety of fiber types and can be strong, delicate, or durable. Particles can have a number of features, such as absorbency, insulating properties, or structural reinforcement, depending on their composition and size. Natural mats can provide a flexible, level surface with a lovely natural feel. Natural carpets, fibers, and other materials are frequently regarded as being more sustainable and kinder to the environment than their synthetic counterparts. While the characteristics and uses of each vary, they all have the benefit of being constructed from natural materials, which encourages sustainable practices [7, 8].

Natural fiber and particle composites are less expensive and less harmful to the environment than synthetic fibers [9–11]. Composites formed from single natural fibers or particles do not, however, possess the same strength as those made from synthetic fibers. Jalil et al.’s (2021) [12] study on the use of current spinning machinery to spin blended cotton made from pineapple leaf fiber (PALF) and PALF. It discusses the physio-mechanical characteristics of the fibers as well as the extraction and degumming of PALF. The purpose of the study is to investigate PALF’s and its mixes’ spinnability for use in both home and commercial settings. It also contains details on FTIR and XRD investigations, yarn characterization, fabric property evaluation, chemical analysis, and more.

According to Alim et al. (2022) [13], the melt-blending approach is used to manufacture and compatibilize binary blends of superheated steam-treated jute and poly (lactic acid) (PLA) biocomposites. With the goal of improving these biocomposites’ dimensional and physico-mechanical qualities and their commercial feasibility,

the study modifies jute surfaces using the superheated steam (SHS) treatment. The efficacy of the SHS treatment on the jute-PLA biocomposites was evaluated by a range of studies, such as mechanical, morphological, and water uptake assessments. According to Mishfa et al. (2024) [14], the creation and characterization of a composite material reinforced with epoxy resin from snake plant fibers is covered. The purpose of the study is to investigate these sustainable composites’ mechanical, morphological, and water-absorbing qualities. The procedure of making these composites, the tests done to assess their qualities, and the conclusions drawn about their water absorbency, mechanical strength, and bonding qualities are all highlighted in the paper.

Mats made of natural fibers can help you get the results you want. The mechanical and thermal properties of biocomposites may be enhanced by using different natural fiber matting. In recent years, researchers have carried out research on the mechanical and thermal characteristics of biocomposites that are strengthened by natural fiber mats, including but not limited to straw, coir, seagrass, jute, areca, kenaf, hemp, and sisal. Areca is one of these natural fibers; it is a member of the *Areca catechu* family and is extensively utilized in the building, packing, marine, and sporting goods sectors. It is a more effective replacement for glass fibers and is lightweight. The outer husk of the nut is removed, and the fibers from the areca palm are treated to make mats. Usually woven or braided, these mats have a flat, pliable surface. The natural golden-brown color and texture of areca fiber mats are well-known. Native to tropical Asia, the areca palm is now grown for its nuts, which are frequently used to chew betel quid [15, 16]. Currently, there is less information on the mechanical, thermal, biodegradability, and water absorption capabilities of natural areca fiber mat biocomposites reinforced with epoxy. Still, research herein field has produced encouraging outcomes in terms of creating composite materials that are eco-friendly and sustainable but still have superior qualities.

Very few researchers use fiber mats; the majority of researchers exclusively use natural fiber or particles. The main focus of this study is the potential use of areca fiber mat, which is readily available in India and has to be exploited to manufacture things that benefit society and compensate the farmers. The purpose of this research is to use natural fiber composites produced in India to make various manufactured items. Areca fiber mat may be used to create strong, reasonably priced, environmentally friendly, and recyclable biocomposite products. Limited articles are accessible, particularly with regard to the number of fiber mat layers. Thus, it is decided to change the number of fiber mat layers.

This study's objectives are to (1) prepare areca fiber mats and treat them with 5 wt% NaOH, (2) fabricate one neat polymer and biocomposite material using epoxy and a number of mat layers of fiber mat that varies (0, 1, 2, 3, and 4) areca fiber mat as reinforcement, and (3) examine the mechanical, morphological, thermal, water absorption, and biodegradability characteristics of both neat polymers and areca fiber mat composites.

2 Methods

2.1 Materials

We used epoxy and HY 951 (Araldite) from M/s Go Green Products (Tamil Nadu, India) as the matrix and hardener for this investigation. Alkaline NaOH was supplied by Indian Scientific Solution (India). Areca fiber in the mat (Fig. 1) layout with an aerial density of 290 GSM (thickness 0.38 mm) was purchased from Metro Composites, Pvt. Ltd. (Tamil Nadu, India). Table 1 illustrates the properties of areca fiber mat.



Fig. 1 Areca fiber mat

Table 1 Properties of areca fiber mat [5, 17]

Description	Units	Areca fiber property
Density	GSM	300
Tensile strength	MPa	320–876
Hemicellulose	%	34.5–63.8
Lignin content	%	12.5–25
Young's modulus	GPa	42.5–48.4

2.2 Alkaline NaOH treatment in areca fiber mat

Areca fiber mats that were ineligible for this investigation were used. The areca fiber mats were dried for one day at 95 °C in a hot air furnace to eliminate moisture from them [18]. NaOH treatment is the technique that is most frequently used to clean and bleach the surface of natural fiber mats in order to obtain premium fiber mats [19]. Distilled water was mixed with 5% NaOH (related to the weight percent of the areca fiber mat), and the areca fiber mat was then submerged in the distilled water for one day. In regular water, the fiber mat was washed. The areca fiber mat was then given a final day of 90 °C oven drying [20]. The purpose of this treatment was to reduce the hydrophilic groups and enhance the surface area. This hydrophilic character can result in poor wet ability, incompatibility, and weak bonding at the fiber/matrix interface in a hydrophobic polymer matrix.

2.3 Fabrication of the areca fiber mat biocomposites

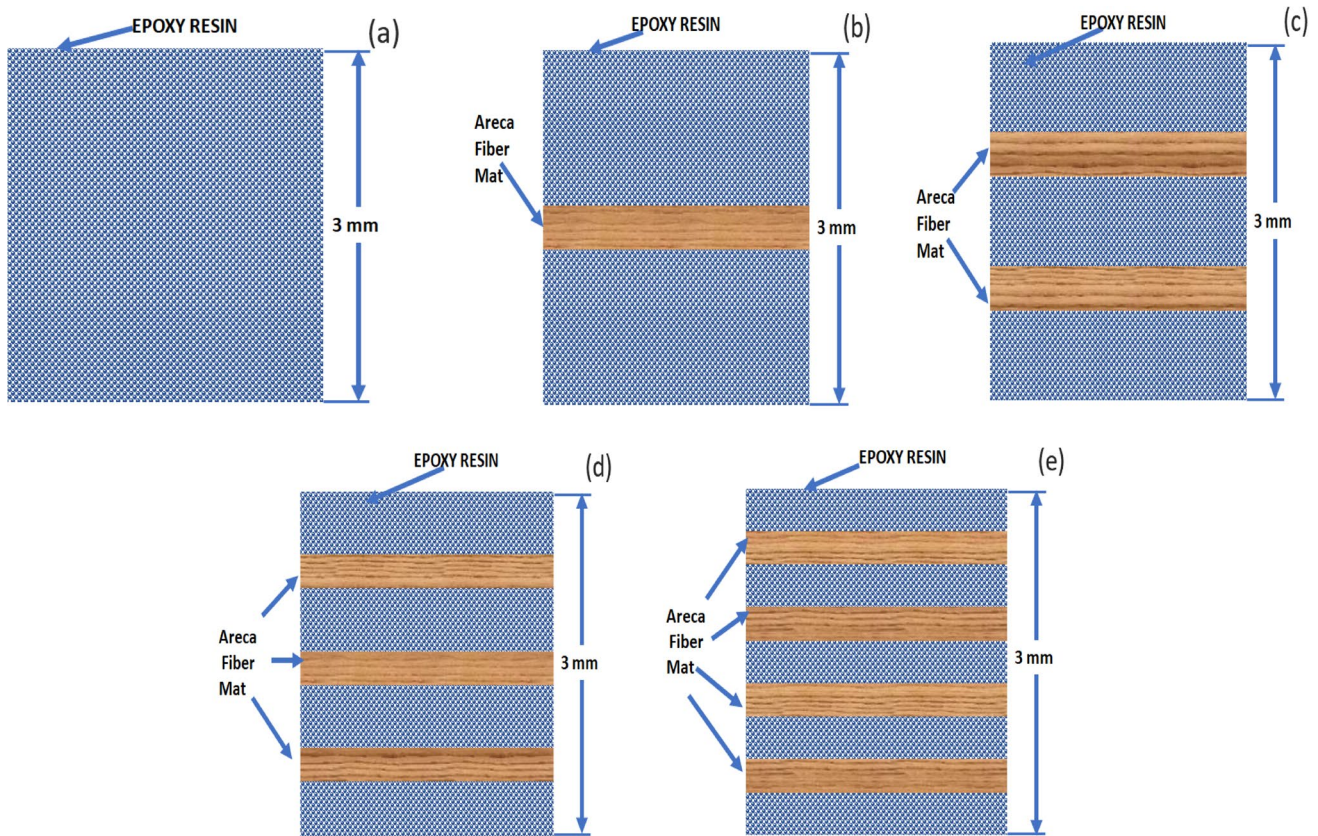
The fabrication process of areca fiber mat biocomposite involves the utilization of the hand lay-up technique. Five biocomposite laminates with varying numbers of areca fiber mats and reinforcement materials are produced. Table 2 displays the different sequences used in this work. The hand lay-up mold size for the areca fiber mat biocomposites was 300 × 300 × 3 mm. To easily remove the laminate from the mold, attach a lean, visible sheet to its base and cover it with wax. The coupling agent HY951 (Araldite) was mixed with the epoxy in a 10:1 (by weight) ratio, and the mixture was manually agitated [21]. Transferred into the mold and distributed throughout the frame is the resin mixture. To release air bubbles trapped between the fiber mat layers, the first layer is placed on top of the resin mixture and further rubbed with a roller. The matrix material is once again transferred from the mat's top to the mold and then back to the roll. The top layer is covered with a second transparent sheet that has been wax-coated, and the mold is then loaded with a 20 kg dead weight [22, 23]. The entire setup is left undisturbed for 24 h at room temperature to allow for curing. The following day, these composites were post-cured in a hot air oven for 8 h at 110 °C. We repeat this process until we reach the biocomposite laminate's 2, 3, and 4 layer mats. Figure 2 shows a schematic diagram of fiber mat layer arrangement. Figure 3 shows the actual developed areca fiber mat composites.

2.4 Biocomposite characterizations

Conducting tensile and flexural tests on composite materials is made easier by the Tinus Olsen Universal Testing Machine ((Model 150 ST) Met Mech Engineers Laboratory, Chennai).

Table 2 Composition of areca fiber mat biocomposites

S. no	Laminate code	Composition of biocomposite laminates	Wt % of areca fiber mat
1.	S1	Epoxy (neat polymer)	0
2.	S2	Epoxy + 1 areca fiber mat layer	7.8
3.	S3	Epoxy + 2 areca fiber mat layer	15.6
4.	S4	Epoxy + 3 areca fiber mat layer	23.4
5.	S5	Epoxy + 4 areca fiber mat layer	31.2

**Fig. 2** Areca fiber mat composites: **a** neat polymer composite, **b** single layer fiber mat, **c** double layer fiber mat, **d** triple layer fiber mat, and **e** four layer fiber mat

This testing device has a load range that can go anywhere from 0 to 150 kN and a cross-head rate that can go anywhere from 5 to 500 mm/min. The ASTM D 638 and ASTM D 790 standards were utilized in order to conduct their respective evaluations of these two tests. Each composite underwent the evaluation of five samples, followed by the presentation of average evaluation results. We conducted the Izod impact test using the EMIC pendulum machine, as per ASTM D 256. Each composite displayed the average results from each of the five used samples.

Water absorption in percentage is determined in accordance with the standard (ASTM D570) for specimens of areca fiber mat

composites. Before being immersed in distilled water, samples were weighed on a computerized scale. They were subsequently immersed in the distilled water beaker. After 24 h, they were carefully removed from the beaker, moisture removed using tissue paper, and weighed again to measure water absorption. The samples reached equilibrium using the same method. Calculate the water absorption percentage:

$$W(\%) = \{(W_{\text{wet}} - W_{\text{dry}})/W_{\text{dry}}\} \times 100\% \quad (1)$$

where W_{wet} and W_{dry} were wet and dry weight of samples, respectively.



Fig. 3 Actual developed areca fiber mat composites

The biodegradability test of the samples (ASTM D6400) is experiential by following the weight loss over time in a soil environment system (environmental resources, waste disposal, etc.). The samples are first weighed before being buried in the ground. After 40, 60, and 80 days, the buried specimens are unearthed. After being taken out of the soil, the specimens are cleaned with tissue paper, and they are then weighed to see how much weight has been lost.

$$W(\%) = \{(W_{In} - W_{Fi})/W_{In}\} \times 100\% \tag{2}$$

where W_{In} and W_{Fi} were initial and final day weight of specimens, respectively

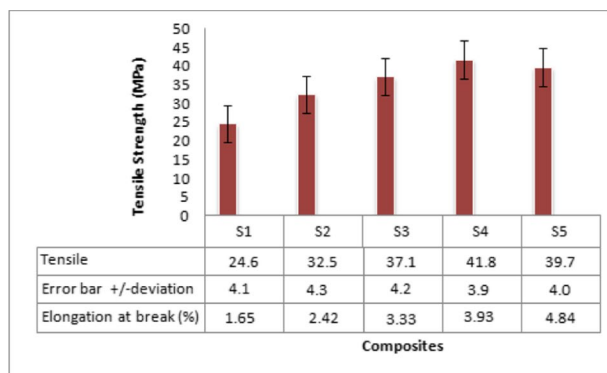
Studies on morphology included scanning electron microscopy (SEM) examinations of the flexural fractured surfaces of areca fiber mat biocomposites. These examinations were carried out with an Indian-made JEOL JSM 6610 LV. After being properly cleaned and air-dried, the composites were then coated with platinum to give improved conductivity and inspected by scanning electron microscopy (SEM) at 15 kV.

Using a NETZSCH STA 449F3 thermogravimetric analyzer, thermogravimetry, often known as TGA, was used to study the thermal stability and thermal degradation of areca fiber mat biocomposites. 10 g of each sample was deposited into a platinum pan, which was then heated in a nitrogen environment at a rate of 5 °C/min from ambient temperature to 600 °C.

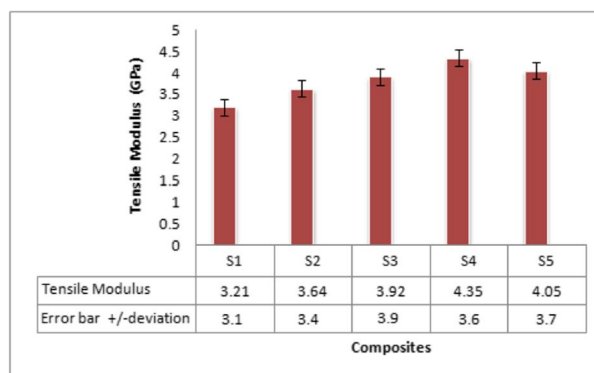
3 Results and discussion

3.1 Tensile strength

The outcome of the areca fiber mat reinforcement on the tensile strength of the epoxy matrix is confirmed by Fig. 4a. Figure 4a demonstrates that the maximum tensile strength of laminates



(a) Influence of areca fiber mats on tensile strength



(b) Influence of areca fiber mats on tensile Modulus strength

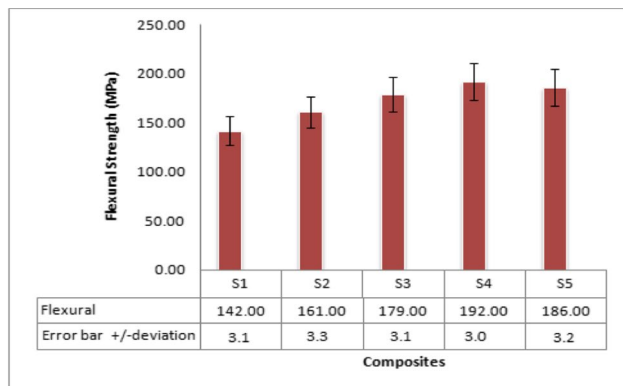
Fig. 4 **a** Influence of areca fiber mats on tensile strength. **b** Influence of areca fiber mats on tensile modulus strength

rose from 24.6 MPa for the epoxy resin to 41.8 MPa for three mat layers. The tensile strength enhanced linearly up to three layers of fiber mat reinforcement. But after that, it started to go downhill. The mechanical strength of a fiber mat-reinforced polymer composite influences a variety of elements, including the adherence of the fiber mat to the matrix, the number of mat or weight of mats, the characteristics of the fiber mat, and the orientation of the fiber mat [24]. In some circumstances, processing conditions may have a significant impact on some of these variables. In this instance, it significantly conforms to improved matrix and fiber mat adhesion, which can support weight and strengthen matrix bonds by covering the full fiber mat’s surface area. However, as the number of fiber mat layers increases above three, the reinforcing reduces, which in turn causes the tensile to decrease [25, 26]. It is also true that it is more difficult for the resin to completely permeate the fibers at high reinforcement percentages, which results in poor interfacial bonding and consequently worse mechanical qualities. Therefore, inadequate wetting causes ineffective stress transmission at the fiber mat-resin contact, which causes agglomeration and prevents stress transfer. As a result, the tensile strength decreased as the number of fiber mats in the composite increased past three. The tensile modulus results,

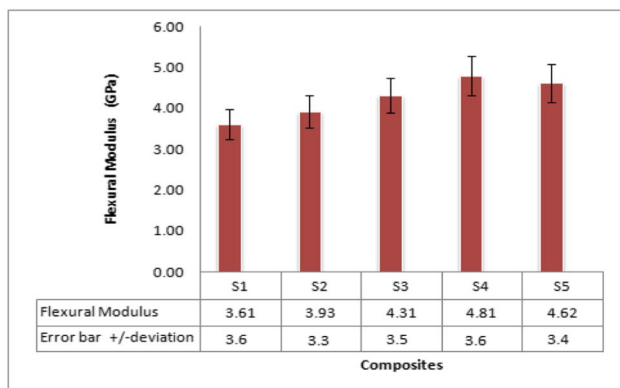
as illustrated in Fig. 4b, the tensile strengths exhibit identical tendencies. The tensile modulus is observed to increase as the addition of the areca fiber mat layer increases up to three layers further increasing the layer strength degradation, which is a result of the composite's increased tautness. As a result of the fiber structure's rigidity, the rigidity of the fiber mat is further enhanced by the potential intermolecular hydrogen bonding between the cellulose in the fiber mat and the resin.

3.2 Flexural strength

Average flexural strength also steadily rises up to three layers of reinforcement in an areca fiber mat before significantly declining for a four-layer areca fiber mat. Figure 5 illustrates the three mat layers that were used to achieve a highest flexural strength of 192 MPa. The tensile strength of the composites was found to be greater than that of the other materials. This might be a result of the flexural test's induced tensile and compressive stresses. This is also related to how the fiber mats are arranged in the composites' outer layer. The fracture starting points are bigger as you go above the three fiber mat layer (ideal fiber matrix ratio) because there is more stress



(a) Influence of areca fiber mats on flexural strength



(b) Influence of areca fiber mats on flexural modulus strength

Fig. 5 a Influence of areca fiber mats on flexural strength. b Influence of areca fiber mats on flexural modulus strength

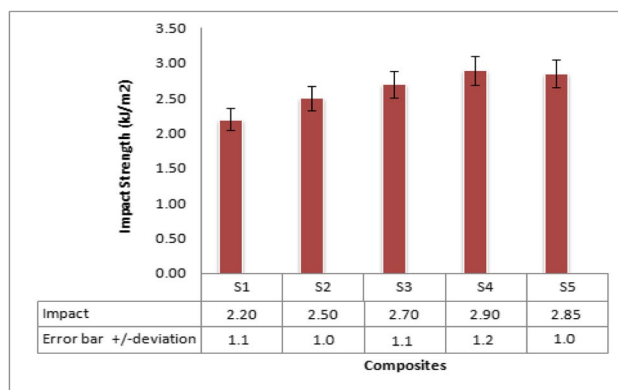


Fig. 6 Influence of areca fiber mats on impact strength

concentrated at the fiber ends [27, 28]. Due to the epoxy's poor adherence to the areca fiber mat, the flexural strength will suffer as a result. Figure 5b illustrates the flexural modulus. The flexural strengths reflect identical proclivities.

3.3 Impact strength

The energy required to break a neat epoxy resin (one that does not contain fiber mat composite, S1) is 2.2 J. The impact of the fiber mat-reinforced epoxy resin composites is shown in Fig. 6 because the fiber mats initiated some brittleness as an enhancement in hardness, which resulted in a loss in impact strength. The impact strength of three-layer fiber mat-reinforced composites has a maximum improvement of 2.9 kJ/m² over composites reinforced with other fiber mat layers [29]. The fiber matting introduced some brittleness, which is why the impact strength decreased [30]. The trends for tensile and flexural strengths are the same. The strength ratings of these composites are only marginally better than those of composites reinforced with areca fiber or particles.

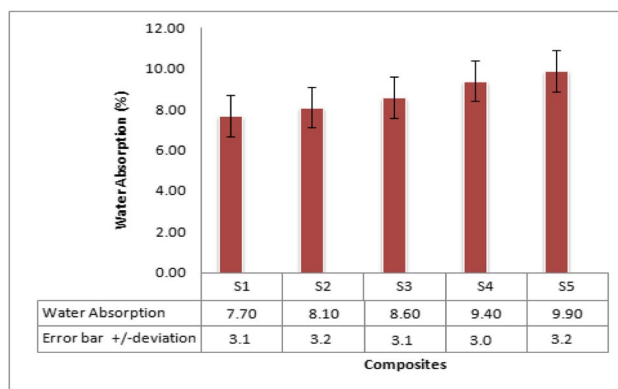


Fig. 7 Influence of areca fiber mats on water absorption

3.4 Water absorption

Figure 7 illustrates how the duration of exposure time affects the proportion of water that the fiber mat absorbs. It was discovered that composites with more fiber mat content have greater water absorption properties. Generally speaking, materials comprised of natural fibers have the capacity to absorb moisture. This is due to the increase in apertures caused by the filler material, which also improved the composite’s capacity to absorb water. It is also possible that agglomerations form more frequently as filler content increases, due to the challenges of ensuring a homogenous dispersion of filler content within the matrix [31–33]. The clumping of filler particles in composites leads to increased hydration.

3.5 Biodegradability testing

Due to moisture intake, the sample weight increased for the first 40 days before gradually declining over the following days, as shown in Fig. 8. Although there were more mat layers, the weight of composites only gradually dropped. The weight loss is caused by the interface of micro as well as macro-organisms with the composite samples over a prolonged period of time. Compared to other 2, 3, and 4 mat layers loading in the composite, single fiber mat reinforced composites are said to have a higher biodegradability [34].

3.6 TGA analysis of areca fiber biocomposites

Thermogravimetric analysis (TGA) was used to examine how heating affected the biocomposite materials used in areca fiber mats. Figure 9 compares the weight loss percentage and the amount of waste material for the neat polymer (S1) and areca fiber mat (S2-S5) composite samples. All areca fiber mat composites (S2 through S5) lose weight significantly when compared to clean polymer composites (S1). Figure 7

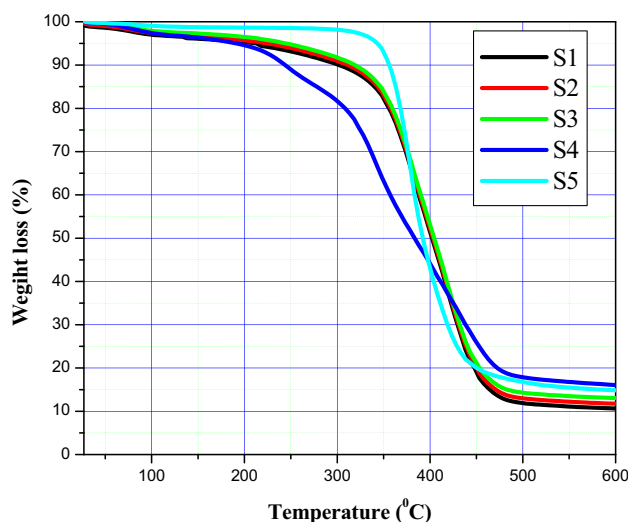
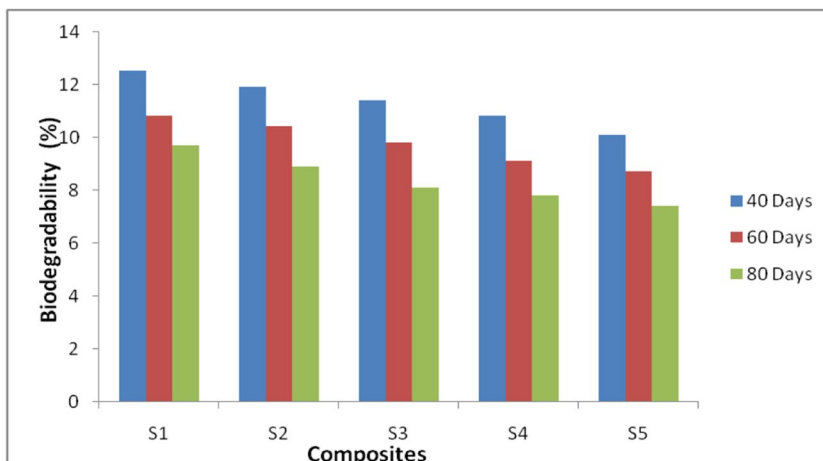


Fig. 9 TGA for areca fiber mat biocomposites

demonstrates that compared to other areca fiber mat layer and neat polymer composites, the areca fiber three mat layer (S3) biocomposites show much better thermal stability. All composites demonstrated improved heat stability with 2.5% weight loss up to 100 °C. In the S1 laminate and other areca fiber mat composites, the primary low-temperature weight loss, which is brought on by the solvent being pulled out of the polymer matrix, begins to happen at 220 °C and 280 °C, respectively [35, 36]. The first weight loss occurs when the composites’ absorbed moisture content vaporizes. Beyond this temperature, thermal stability gradually deteriorates and fiber mat decomposition takes place. The subsequent weight loss of 82% to 88.3% between 220 and 450 °C was brought on by the hemicelluloses, cellulose, and lignin thermally decomposing [37]. Following decomposition, the total weight losses of the S1, S2, S3, S4, and S5 composites were 88.3, 86.1, 85.8, 82.1, and 83.38 wt%, respectively, compared to alternative composites.

Fig. 8 Influence of areca fiber mats on biodegradability



3.7 Tensile fracture surface morphology of areca fiber mat biocomposites

Figure 10a–e shows the tensile shattered surface of the S1, S2, S3, S4, and S5 biocomposites. The fibers mat sharing and orientation are shown in the composite by the matrix and shattered fibers mat. Fiber mats and small virgins are absent, as seen in Fig. 10a. The single-layer and double-layer fiber mat biocomposites are depicted in Fig. 10b, c. The SEM micrograph showed the porosity, fiber mat pull-out,

void in the composite, and void coalescence. As a result, the existence of weaker void surfaces and fiber mat pullout was the deciding factor, and it is possible that the failure mechanism was caused by the ease with which cracks spread across void areas. It denotes poor compatibility between the filler and matrix because of the excessive porosity that leads to air being trapped inside the composite or on the surface [38, 39]. Accordingly, the composites' mechanical characteristics were less than those of other fiber mat composites. Figure 10d displays three mat layer composites (S4); macro-voids, not

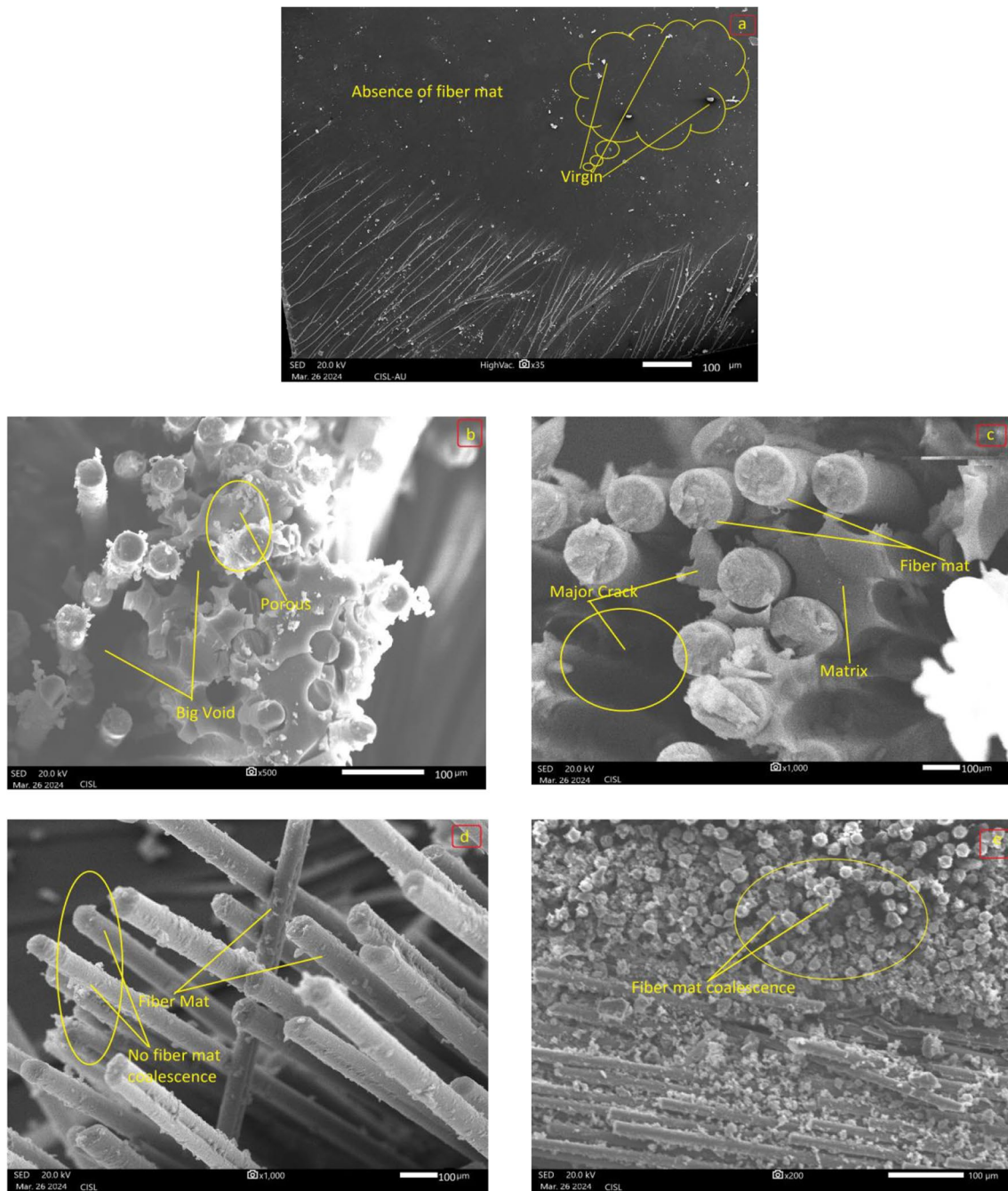


Fig. 10 a–e SEM images of tensile fracture specimen of areca fiber mat composites

fiber mat coalescence, or fiber interference are not visible. As a result, fiber mats were visibly embedded in the dominant matrix. This demonstrates exceptional compatibility and an evenly distributed, uniform fiber mat across the matrix [40]. As a result, the three-layer fiber mat composite has a higher tensile strength (41.8 MPa). The minimal porosity structure and increased fiber mat coalescence seen in Fig. 10e can be used to explain the modest loss in tensile strength seen in four-layer fiber mat composites.

3.8 Flexural fracture surface morphology of areca fiber mat biocomposites

Figure 11a–e illustrates the flexural cracked surface of the S1, S2, S3, S4, and S5 biocomposites made from areca fiber mat. This SEM picture exhibits consistent patterns in the morphology of tensile fractures. Figure 11a (0 layer mat) demonstrates the absence of cavities in the fiber mat. Figure 11b, c, and e depicts the biocomposites made from areca fiber mats, with

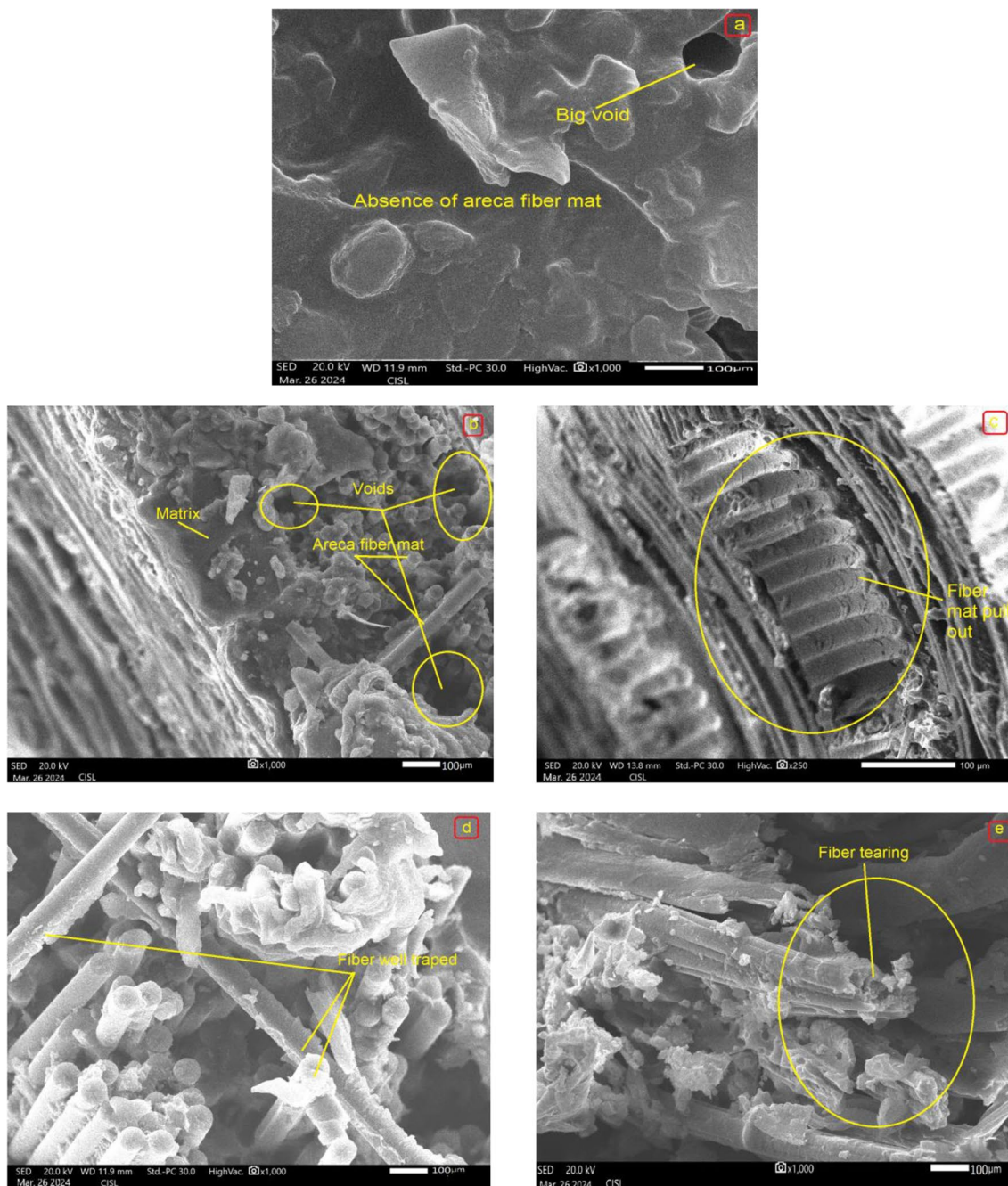


Fig. 11 a–e SEM images of flexural fracture specimen of areca fiber mat composites.

single, double, and four layers. The SEM micrograph revealed the presence of fractures in the composite, fiber mat pull-out, significant voids in the composite, and void coalescence [41]. Figure 11d shows a trapped fiber without any cavities. As a result, three layer areca fiber mat composites (S4) have a greater flexural strength (192 MPa).

4 Conclusions

The present study investigated the mechanical, thermal, biodegradable, and water-absorbing characteristics of epoxy biocomposites reinforced with areca fiber mats in varying layer counts. We draw the following conclusions based on the findings and the relevant discussion.

- This study reveals that the biocomposite reinforced with areca fiber mat in three layers (S4 sample) has the highest tensile (41.8 MPa), flexural (192.00 MPa), and impact (2.9 J) strength.
- The water absorptions increased linearly as the number of areca fiber mat layers increased. The biodegradability decreased linearly as the number of areca fiber mat layers increased at the directly proportional level.
- TGA revealed that the three-layer areca fiber mat (S4) biocomposite outperformed other composites in terms of thermal strength and resilience to temperature deterioration.
- SEM photographs exposed that the three-layer areca fiber mat (S4) composite revealed well-embedded areca fiber mats, strong fiber-matrix adhesion, not fiber mat coalescence, and the absence of voids.
- Three-layer fiber mats were generally the most advantageous areca fiber mat reinforcement for reinforcing epoxy matrix under the current exploratory settings.
- Based on the findings of this study, it is recommended that the areca fiber mat layer composite be used in the manufacturing of furniture and car parts.

Author contribution Katakam Satyanarayana—conceptualization, methodology, data curation, and writing—original draft. Ganesh K Jadhav—supervision and review and editing. D. R. Srinivasan—investigation and formal analysis. Manepalli. Sailaja—project administration and demonstration. A. Sunny Kumar—supervision and validation. Pathem Uma Chaithanya—data curation and English correction. R. G. Padmanabhan—testing and validation. A. Joseph Arockiam—visualization.

Declarations

Conflict of interest The authors declare no competing interests.

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