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ORIGINAL ARTICLE

Structural, Thermal and Elemental Investigations in Epoxy Polymer Composites Reinforced with NaOH-Treated Short Areca Nut Fibers

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Abstract The major goal of this work is to create a process for extracting fibers from the husk of areca nuts, treating those fibers with ecologically friendly media, and turning those treated fibers into an epoxy polymer composite material. Fibers are chopped down to a length of 30 mm manually prior to its usage. Three different amounts of fibers, namely 20, 25 and 30 wt%, have been introduced in an epoxy matrix, fabricating the composite by compression molding technique. Structural, thermal and elemental investigations in epoxy-based composites reinforced with alkali-treated chopped areca nut fibers with different fiber weight percentages have been compared in this novel work. Scanning electron microscope was used for studying the morphological characteristics. Thermogravimetric analysis and derivative of thermogravimetric analysis have been utilized to carry out the thermal analysis. Energy dispersive X-ray analysis was used to study the elemental composition of the synthesized material. From the studies, it is concluded that the composition with 20 wt% areca nut fiber-reinforced epoxy polymer composites exhibits better properties.

Keywords Biodegradable · Compression · Chopped · Renewable

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1 Introduction

Sustainable development is the best way to prevent or mitigate environmental problems. Scientific evidence demonstrates that human consumption of natural resources is unsustainable and that unprecedented cooperation among all stakeholders is required to reverse this trend. Using Earth's resources at a rate that allows them to be replenished is critical to the survival of humanity. A growing need for environmentally friendly materials and a desire to reduce the cost of using traditional fiber reinforcing has led to the establishment of particular types of natural fiber-reinforced epoxy polymer composites. Due to environmental concerns, natural fibers have been designed to be employed in composites as single or hybrid reinforcing fibers. Natural fibers have been obtained from animals, plants, and minerals, utilizing various procedures, such as chemical and thermal, before being used in composites. For mechanical components that require lightweight, strength, stiffness, and fatigue resistance as well as ease of manufacture and a lower cost than synthetic fibers, the demand for eco-friendly composite materials made from natural fiber-reinforced polymers (FRPs) has been steadily rising in the industrial sector [1, 2]. Plant fibers are biodegradable, making the environment greener and suitable for the industrial sector because they are competitive in price and performance [3, 4]. Natural fibers typically play a significant role as reinforcing agents [5, 6]. Important natural fibers in producing automotive parts, such as dashboards, seat backs, clutch disks, and door panels, include kenaf, jute, sisal, bamboo, hemp, pineapple, areca, banana, and coir [7, 8]. To achieve a greener future, we require durable materials with the appropriate qualities, such as recyclability, reusability, and biodegradability. Using epoxy polymer composites reinforced with natural fiber in military applications (partition boards, construction industries, ceiling paneling

and building), transportation (railway coaches, aerospace and automobiles), consumer products, packaging and other industries is more environmental friendly than using synthetic fiber-containing composites.

The research conducted by Prasad et al. [9] shows that the characteristics of the coir fibers can be improved with the assistance of alkali treatment. The strength of these fibers was found to be enhanced by 15% after soaking them in an aqueous solution of NaOH at 5% for 72–76 h at 281° C. It was further found that the fibers were roughened [10] after the alkali treatment to improve mechanical interlock and increase cellulose exposure. Fibers and thermoplastic matrix interfacial shear strength were enhanced by worn morphological and silane chemical changes of the fiber surface. This was later proved eventually by Gassan et al. [11] through the work on jute fiber epoxy composites, wherein the treatment was done with NaOH, and the mechanical characteristics were found to be improved after the treatment.

Styrenated polyester was reinforced with short natural fiber from Hildegardia populifolia by Rajulu et al. [12]. A detailed comparison of the mechanical characteristics of the alkali-treated fibers to those of untreated fibers was made in his work. Furthermore, the mechanical characteristics of jute fiber composites (woven type) were conducted by Gowda et al. [13] in accordance with ASTM-D 3039 standards. The specimens were made by hand layup method, and the mechanical characteristics such as tensile, flexural, impact and hardness were conducted. Kumar et al. [14] examined the mechanical properties of areca fibers and compared them to coir fibers. The effects of alkali treatment on areca fiber strength were investigated after the fibers were chemically altered, and increment in the values were noted. Chemically processed areca nut fiber-reinforced natural rubber composites for applications requiring high tensile strength [15] and low-density features of raw betel nut husk fiber for applications requiring high-dimensional stability [16, 17] have both found to be used in lightweight applications. Hence, it can be concluded that the chemical treatment enhances the strength of the fibers through various aspects.

Snipped mat glass-polyester composites were judged superior in some situations, but betel nut polyester composites possess the same mechanical characteristics as glass and polyester composites [18]. Alternatively stated, betel nut fibers can be suggested as a replacement to usage of glass fibers [19] in some applications. Utilizing natural fibers is a fantastic way to save money and reduce the environmental impact. The eco-friendliness and biodegradability of natural fibers make them a potential alternative to carbon and glass fibers [20]. A wide variety of materials of biodegradable nature were employed in the fabrication of composite materials, including grain straw, corn cob, cotton waste [21], processed tea waste [22], corn, areca nut fiber and bamboo waste [23–25]. Although only a few agricultural waste products are commercially available, researchers worldwide are looking into new agricultural waste products [26]. As a result, thorough research is required to locate underutilized waste resources that can be used to make composite material and earn cash. Alkali treatment lowers the wetting ability and specific surface area while destroying the bonding within the fiber network structure [27, 28].

Woods and plants like areca nut and flax have been employed as sources of lignocellulosic fibers for the reinforcing of composite materials such as fiberboards. They are a viable alternative to carbon, glass, and other synthetic fibers because of their qualities, including low density, availability, renewable nature, low cost, and mechanical properties. About 40% of India's total areca nut production is in Karnataka [29]. Areca nut production in India is the largest in the world. Areca nut husk fiber must be studied and researched in depth to increase its usage. This palm, areca catechu, is a member of the arecaceae/palmae family; it grows vertically, reaching a height of ten to twenty meters. Its stem is 5 cm to 7.5 cm in radius and has scars from falling leaf sheaths. Because of its high strength to weight ratio, the areca husk fiber could be used to make structural components in engineering applications. Short areca nut husk fiber-reinforced composite's research is still in its infancy. Characterization of areca fiber-reinforced epoxy polymer composites is motivated from the fact that it provides insight into the behavior of these materials under varied loading circumstances, which is critical for their use in diverse engineering applications. Furthermore, the characterization of these composites can help in optimizing their properties by varying the fiber content, fiber orientation, and matrix properties. This can lead to the development of composites with tailored properties that are suitable for specific applications. For instance, composites with high strength and stiffness may be suitable for use in structural applications, while composites with high thermal conductivity may be suitable for use in heat transfer applications.

To summarize the above considerations, it is fair to say that areca nut husk fiber composites have some potential, especially in that the filler does represent an easily available agrowaste source; this has been scantily explored for, so far, though. In this current work, laminates containing areca nut husk fibers were fabricated by utilizing the compression molding technique. Chopped areca nut fibers were added in varying weight percentages of 20%, 25% and 30% over the weight of epoxy resin for this detailed investigation. In particular, different characterizations, which include testing of structural, thermal and elemental properties, were conducted.

2 Materials and Methods

2.1 Materials Used

Hand lay-up method uses areca nut fiber, hardener, and epoxy glue to make these composite panels. This process is used to break down long areca-nut fibers into smaller ones. Being a lignocellulosic fiber, areca nut husk fibers contain three basic chemical components, namely cellulose, hemicellulose and lignin. It is observed that areca nut husk fibers contain 56.8% cellulose, 22.4% hemicellulose and 6.3% lignin, respectively. Fibers collected from the husk of areca nut trees are chopped down to a length of 30 mm and either used as such or subjected to alkaline treatment by immersion in a 5% sodium hydroxide solution. Sodium hydroxide pellets (AR 500 g) used in this study were purchased from Labogens Fine Chem Industries, Ludhiana, Punjab.

For composite preparation, chopped areca nut husk fiber was mixed with epoxy resin and hardener. Resin used in this work was LY 556 and hardener used was HY 951. To handle tensile loads, the areca nut husk fibers in the composite material were held together by the epoxy resin. Epoxy resin shields the fibers of the areca nut husk from exposure to the outside environment. Epoxy resin was utilized to produce composite sheets because of numerous benefits that they hold such as strong adhesive ability with a wide range of materials, little shrinkage after curing, fast curing abilities, sufficient mechanical capabilities, greater electrical resistance, greater chemical inertness and water insoluble qualities [27].

2.2 Alkali Treatment

Areca nut husk fibers were initially cleaned using fresh water. To complete the chemical retting procedure, the areca nuts were immersed in a 6 percent by volume NaOH alkali solution for 24 h at a room temperature of 27ºC. NaOH-treated areca nut fibers were disinfected with water in order to remove any traces of chemical residue. The areca nut fibers were then dried in the sun for two days after washing, to reduce the amount of moisture absorbed from the fibers. This method of removing fiber was carried out by first drying and then sealing the husks of areca nuts. Figure 1a, b represents untreated short areca nut fibers and alkali-treated short areca nut fibers, respectively.

2.3 Fabrication of Composite

A widespread and simple technology used for the fabrication of the composite material is compression molding technique. Table 1 depicts the composite laminate codes developed for the experimental evaluation. The epoxy to hardener ratio of the final material was found to be 10:1. The ideal fiber volume percentage was identified to be 5% using 300 g epoxy

Table 1 Composite laminate codes of the materials developed for the experimental evaluation

Composite laminate code	Quantity of resin (g)	Quantity of hardener (g)	Natural FIBER weight percentage added
L	300	30	20
M	300	30	25
O	300	30	30

Fig. 1 **a** Areca nut fiber in shortened form, **b** chemically treated areca nut fiber in short form

and 30 g hardener. The liquid was poured over the areca fibers that had been treated with NaOH and then sliced. The mold of the metal was later immersed into the liquid, pressed, and left to cure for three consecutive hours. The dimensions of the composite material made was 200-mm long, 200-mm wide, and 6-mm thick. The ASTM standards were then machined with the assistance of a water jet after the curing process.

2.4 Wear Investigations

The surface morphology of NaOH-treated chopped areca nut husk fiber-reinforced epoxy polymer composites were analyzed by a scanning electron microscope (Make: Zeiss Sigma VP, USA). Pin-on-disk (POD) specimens after wear test were used to examine the morphology. A POD machine (wear and friction machine TR—20 LE) made by Ducom Material Characterization systems, USA, was used to test the dry sliding wear. In compliance with the ASTM G99-05 requirements, the specimens were being made. The experiments made use of 30-mm-long pins with a 5 mm × 5 mm square cross section. The applied stresses were 20 N, 15 N and 10 N, and the disk rolled at a speed of 3 m/s. Before running the tests, abrasive (SiC) papers were used to smooth down the surfaces of the specimens and counters. Acetone was utilized to clean and dry the test samples. Prior to and just after the dry sliding test, it was weighed. The decrease in the weight of the composite sample was calculated from the difference of these two weights. An electronic weighing equipment AUW. 220D made by Shimadzu, Japan, was used to measure the masses of the samples. Table 2 describes the wear values which depicts that combination with 20 wt% areca fiber-reinforced epoxy polymer composites exhibits the least values of wear.

In order to make the surface of the specimens conductive, a thin sheath of gold was used as a coating. This instrument had resolution of 1.3 nm, accelerated voltage of 0.1 kV to 30 kV and a magnification of 12 × to 1000,000 ×. The morphological images captured for the current study possessed an accelerated voltage of 10 kV, resolution of 10 µm, and a magnification of 1000 ×. The images taken were then studied to analyze the fiber epoxy bonding in different samples.

Table 2 Wear in grams obtained corresponding to different loads

Material Combination	Wear corresponding to 10 N Load (g)	Wear corresponding to 15 N Load (g)	Wear corresponding to 20 N Load (g)
L	.0014	.0016	.0018
M	.0029	.0033	.0036
O	.0040	.0045	.0048

Figure 2 displays SEM experimental set up used to generate images with high-energy electron beams.

2.5 Thermal Analysis

Thermogravimetric analyzer used in this work is TGA 8000 thermogravimetric analyzer (Make: PerkinElmer, USA). Figure 3 depicts the thermogravimetric analytical set-up utilized in the experiment. This machine provides a sensitivity of 0.1 µg, accuracy of 0.02%, precision of 0.01%, and a temperature range of − 20 °C to 1200 °C. Thermogravimetric analysis (TGA) is a thermal analysis technique used to investigate the thermal stability and decomposition behavior of materials. Here, the sample was heated in a controlled atmosphere while continuously measuring its weight loss as a function of temperature or time. This information can be used to determine the temperature at which the material begins to degrade, as well as the rate and extent of degradation. The method used for cooling was forced air cooled with an external fan. The technique can provide information on the weight loss and degradation temperature of the

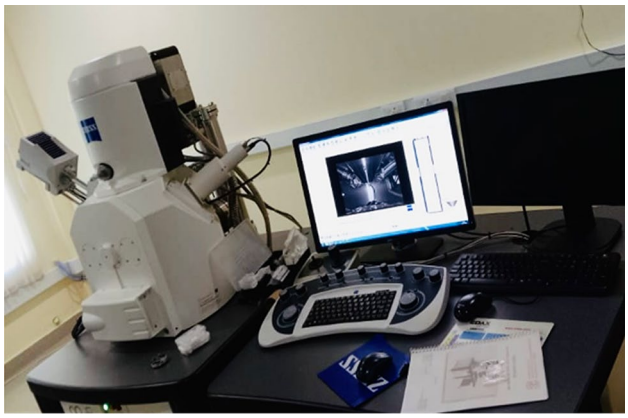


Fig. 2 SEM experimental set up

Fig. 3 Thermogravimetric experimental set-up

composites as a function of heating rate, as well as the thermal stability of the individual components of the composites. The derivative of thermogravimetric analysis (DTG) is a related technique that involves taking the first derivative of the weight loss curve obtained from TGA. Derivative sensitivity of the instrument used in this work ranged from 0.1 °C/min to 10 °C/min. DTG provides information on the rate of weight loss as a function of temperature, which can be used to identify different stages of thermal degradation and to determine the activation energy of the degradation process. In summary, TGA and DTG are useful techniques for investigating the thermal stability and degradation behavior [31] of areca fiber-reinforced epoxy polymer composites and can provide valuable information for the optimization of the properties and performance of these materials. Lignocellulosic fibers like areca nut fibers are temperature sensitive, and total thermal breakdown is expected at temperatures surpassing 400 °C [30, 32, 33]. Physical qualities of plant fibers are due in part to the presence of hemicellulose, lignin and cellulose all of which are found in high concentrations in plants. This was further seen on the graphs plotted between TG in % versus temperature in °C and DTG in µg/min versus temperature in °C.

2.6 Elemental Investigations

Elemental analysis of a composite sample can be done using energy dispersive X-ray analysis, which is also known as EDAX/EDX. The distribution and concentration of components within a composite material can be determined specifically with the aid of EDX analysis, which can reveal information about the material's mechanical, thermal, chemical, and electrical properties. It further helps us to determine the presence and distribution of reinforcement fibers in a composite material and to forecast its strength and stiffness. The performance and durability of the material can be impacted by the presence of contaminants or impurities, which can be detected using this method. In general, EDX analysis is a crucial method for examining the elemental composition of composites since it can provide vital details about their characteristics and behavior.

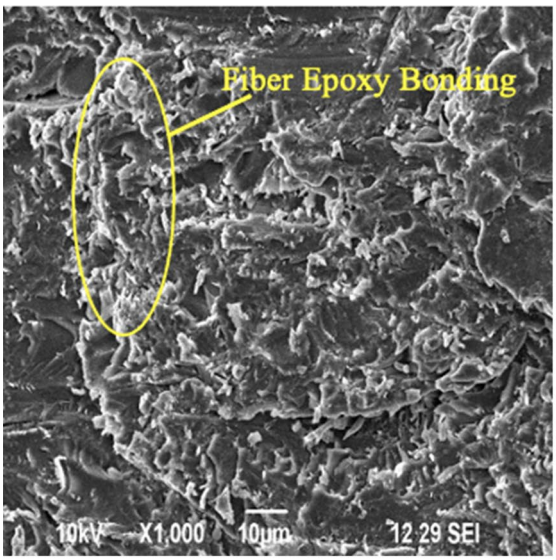
X-ray excitation and a sample are required for its operation. The fact that each element has a different atomic structure and, as a result, emits electromagnetic radiation with a different set of peaks lends itself well to its characterization. X-ray emission can be stimulated by focusing an electron beam on a specimen, which produces distinctive X-rays. Samples of atoms that are at rest contain electrons that are in the ground state (or unexcited) or bonded to the nucleus. It is possible that the incident beam will excite an electron in an inner shell, which will cause the electron to be released from the shell and create an electron hole in that place. In order to fill the hole, an electron from a higher-energy shell fills it, and this difference in energy may be emitted in the form of an X-ray.

3 Results and Discussion

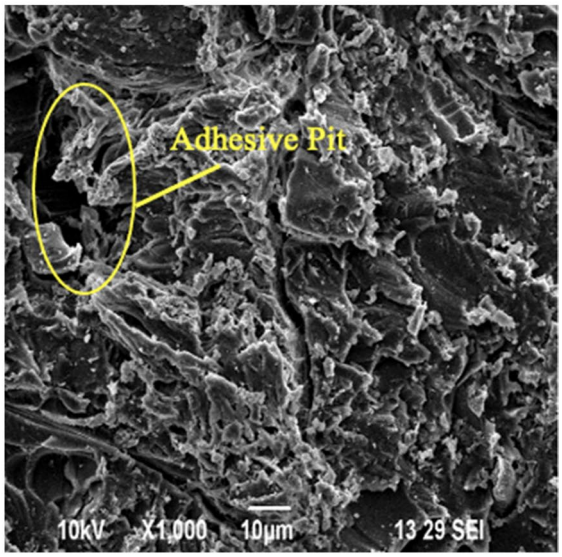
3.1 Structural Results

Figure 4 shows morphological images of worn surfaces after a wear test conducted on a tribometer. At a 500-fold amplification, secondary electron imaging (SE) was employed to produce images with 1000×1000 m resolution. Images indicate a bumpy, burr-like texture on the sliding surface. From the figures, it is evident that the introduction of larger amount of fibers does produce a marked difference into the modes of fracture of the composites. Figure 4a shows short areca fibers that have a strong interfacial connection with epoxy resin. The least amount of damage has been found in the form of wear scratches and debris in the low-intensity wear tracks. Adding 20 percent areca fiber makes the epoxy surface gritty and rough. In addition to debris and broken fibers, specimen shown in Fig. 4b has a worn surface with low interfacial connections between the areca fiber and its matrix, as evidenced by its worn surface. The principal cause of adhesive breakdown is the separation of large forms of flakes of material from the matrix by greater sliding force. Peeling pits appear as well on the friction surface of the material. Adhesive wears occur because of the combination of abrasion and fatigue. Figure 4b shows that significant strains cause debonding of the worn surface, as can be seen. There are pits and fissures evident on the laminate's worn surfaces, indicating that low-content fiber content does not improve the interfacial effects between the matrix and the fiber. The loss of fibrous areas at the points where the fiber is exposed to the counter face is observed as wear develops. Specimen shown in Fig. 4c depicts the agglomeration's worn surface that has microcracks, sticky pits, and hard particles. The composite matrix of specimen shown in Fig. 4c has an overall low wear strength and weak reinforcing ability because of its lack of reinforced fibers. A variety of thermo-mechanical stress conditions causes resinous areas of the composite surface to be more sensitive during sliding. Due to a lack of areca fiber, the polymer's surface characteristics erode, and its abrasive capabilities are affected. Furthermore, it can be concluded that the formation of cracks, pits, debris and agglomerates is more likely to be seen in the composites with lesser wear strength when compared with the materials with better wear properties.

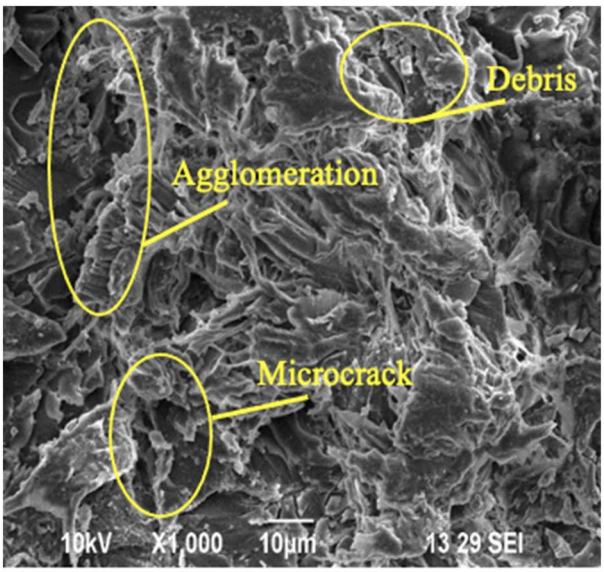
In comparison with the other composite laminate codes shown in Fig. 4b, c, the microstructure of Fig. 4a indicates greater bonding between the areca fiber and the epoxy. As a result, the laminate composite code R is dominated by morphological characteristics.



(a) Structural analysis of 20 wt %



(b) Structural analysis of 25 wt %



(c) Structural analysis of 30 wt %

Fig. 4 **a** Structural analysis of 20 wt%, **b** structural analysis of 25 wt%, **c** structural analysis of 30 wt%

3.2 Thermogravimetric Results

Deviation of thermogravimetry (TG) and derivative of thermogravimetry (DTG) with respect to temperature for an alkalized 20 wt% areca fiber-reinforced epoxy polymer composite is indicated in Fig. 5. The highest value of DTG obtained for an alkali-treated 20 wt% areca fiber epoxy polymer composite has been found to be − 10 µg/min corresponding to a temperature range of 200 to 250 °C. In contrast, the lowest peak value for DTG is obtained at 351.3 °C inhibiting a DTG value of − 721.7 µg/min. One more minimum peak value is indicated in the same figure, corresponding to 476.3 °C with a DTG value equal to − 326.3 µg/min. These values obtained are found to be sufficiently higher at each point corresponding to the same sets of values obtained for an untreated composition. From Figs. 5 and 6, it is evident that an alkalized areca fiber possesses predominantly better values for derivatives of thermogravimetry. From the figures, it is clearly observed that the treatment applied on areca nut husk fibers results in a reduction by around

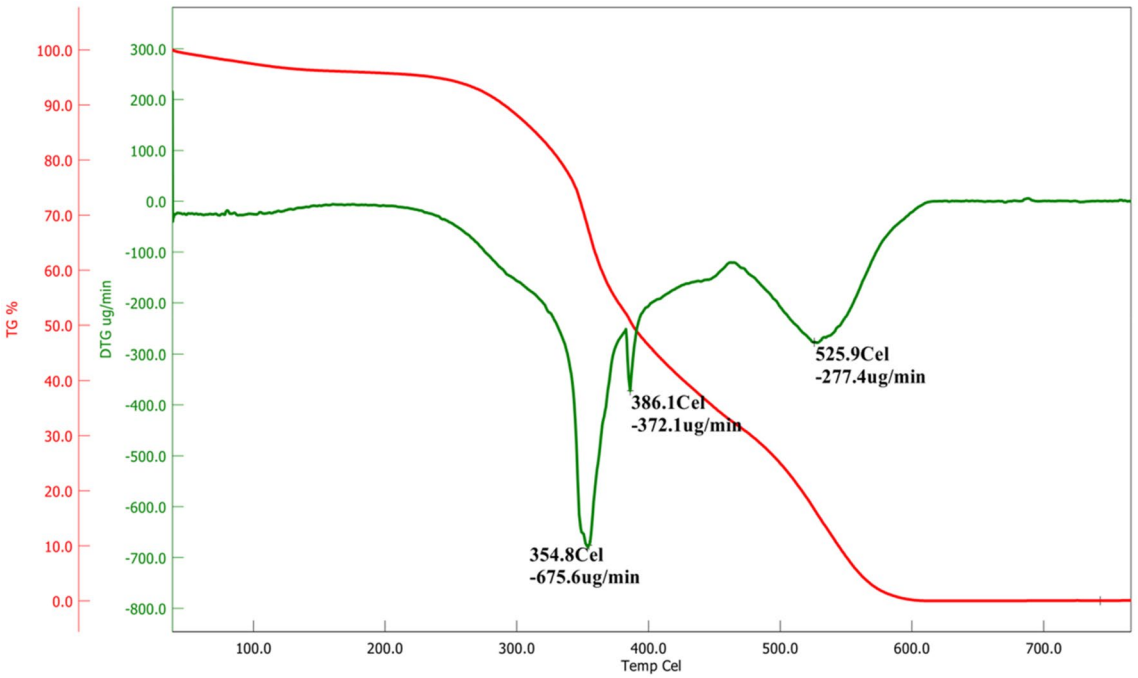


Fig. 5 Variation of DTG/TG v/s temperature for untreated 20% areca fiber volume %

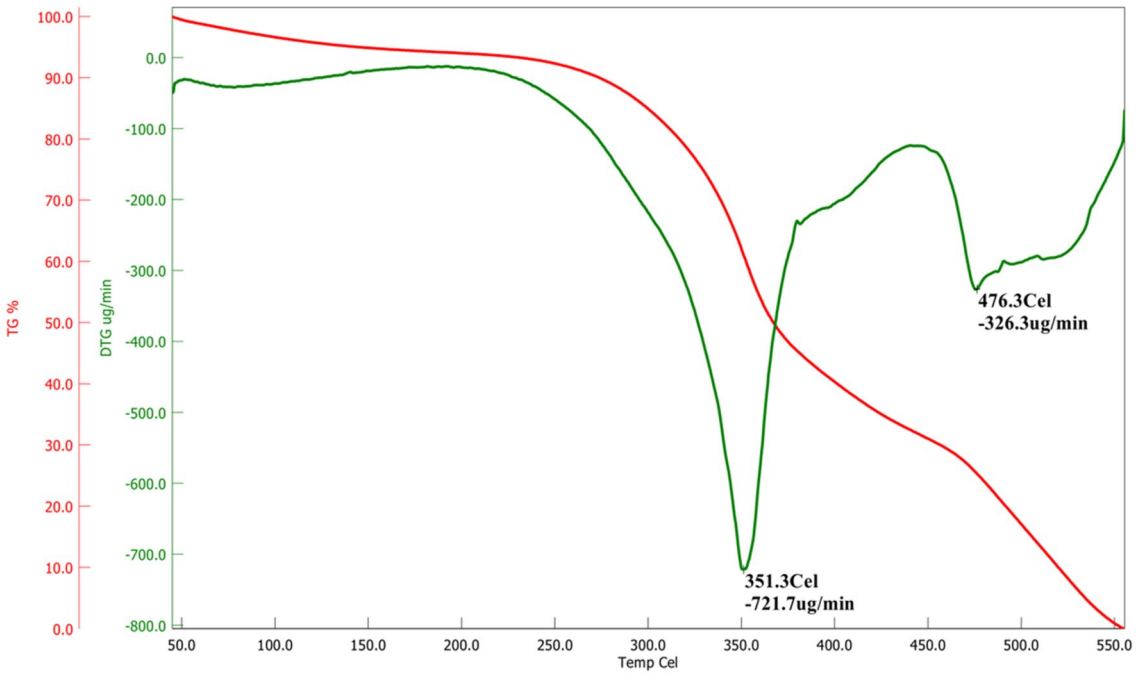


Fig. 6 Variation of DTG/TG v/s temperature for alkalized 20% areca fiber volume %

20 °C of the peak degradation temperature of the biomass. Also, a substantial decrease to almost zero of the residues is obtained at 550 °C. This is an expected outcome, which has been reported on the degradation of rice husk after alkali treatment and may be attributed to the dissolution of lignin and some hemicellulose.

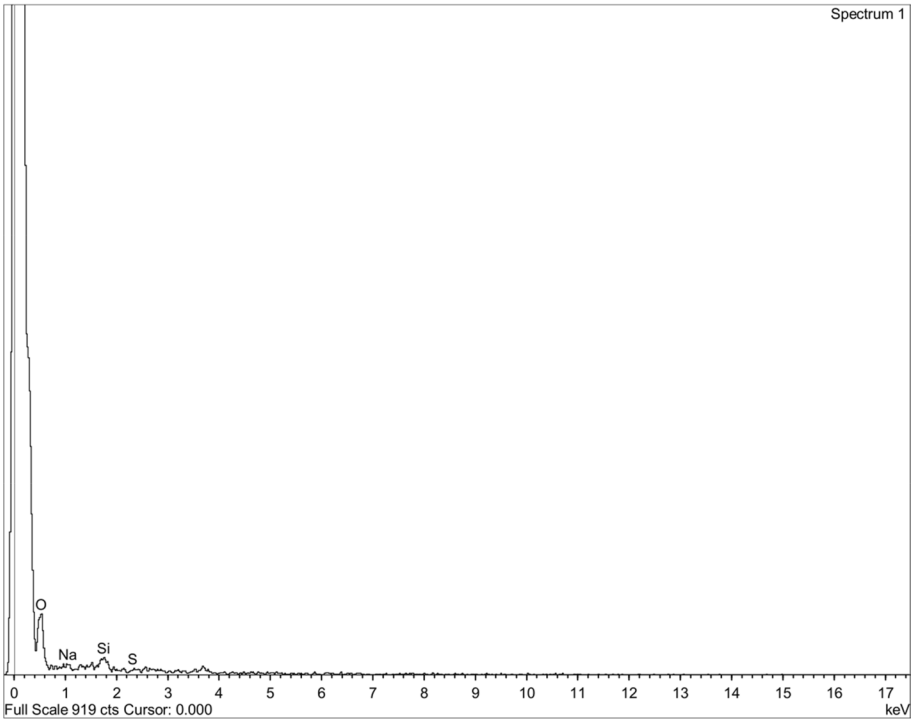


Fig. 7 EDAX analysis of 20 wt% treated composites

Table 3 Elemental composition obtained from EDAX analysis

Element	Intensity correction	Weight %	Atomic %
O	2.1098	87.46	91.83
Na	0.6409	5.53	4.04
Si	0.8058	6.22	3.72
S	0.8658	0.79	0.41

3.3 EDAX Analysis Results

Figure 7 depicts the EDAX analysis results obtained for the composite samples, whereas Table 3 describes the major elemental compositions in the short areca fiber-reinforced epoxy polymer composites. Elements identified were studied based on different standards. Elements like oxygen, sodium, silicon and sulfur were analyzed based on the standards like silicon dioxide, albite, silicon dioxide and ferrous sulfite, respectively. Oxygen weight % is found to be significantly higher (equal to 87.46%) as compared to other elements, analyzed namely sodium, silicon and sulfur. This further makes the composite material suitable for an oxide material or subsequent applications. Silicon weight and sodium weight are found to be 6.1% and 5.5%, respectively, in the material composition. The weights of the elements sodium and oxygen have been found to be significant, and it may be due to be addition of alkali treatment done on the areca nut fiber-reinforced epoxy composite material.

4 Conclusion

Following a thorough examination of the properties of NaOH-treated short areca fiber-reinforced epoxy polymer composites, the following conclusions were drawn:

- Hand lay-up method and compression molding method can be used in the synthesis of natural fiber-reinforced epoxy polymer composites. Better bonding between the fiber and the matrix can be ensured while performing these methods for ensuring better results.
- Worn morphological evaluation of epoxy polymer composites were conducted on the composite samples and the laminate with 20 weight percentage short areca fibers shows greater bonding when compared with other laminate codes. This is owing to the superior tribological qualities of this particular types of compositions when compared to others.
- Areca fiber-reinforced epoxy polymer composites with composite laminate code L exhibit better results in thermophysical characterization. Thermal characteristics are significantly higher for alkali-treated (20 weight percentage) areca fiber composites when compared with other compositions.
- Better thermogravimetric results were obtained when tests were carried out on an alkali-treated short areca fiber-reinforced epoxy polymer composite samples when compared to untreated ones. The treatment applied on areca nut husk fibers resulted in a reduction by around

20 °C of the peak degradation temperature of the biomass.

- From the EDAX analysis, elemental composition of aforementioned type of composite sample is identified, and it is found that oxygen holds the higher elemental weight percentage when compared with other elements identified. Significant values of silicon and sodium is also seen in the composition. This further makes this material suitable to be used in applications that require elements like oxygen, silicon or sodium.

The results of the morphological, thermal and EDAX analysis show that when compared to the other laminate codes, M and N, the composite laminate code L, which refers to 20 weight percentage alkali-treated short areca nut husk fiber-reinforced epoxy polymer composites, exhibits better characteristics. This particular composition of the areca nut husk fiber-reinforced polymer composites can be suggested as an alternative for a wide variety of applications as they are sustainable, renewable and of ecofriendly nature.

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