



Mechanical, thermal and fatigue behaviour of surface-treated novel *Caryota urens* fibre–reinforced epoxy composite

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Received: 11 June 2020 / Revised: 30 July 2020 / Accepted: 4 August 2020 / Published online: 12 August 2020
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Abstract

Epoxy biocomposites were prepared using acid-, base- and silane-treated novel *Caryota urens* natural fibres (CUFs). The primary aim of this research is to reveal a better surface treatment method to achieve improved mechanical, thermal and fatigue properties of *Caryota* fibre epoxy composite system. The composites were prepared using hand layup method and post cured at 120 °C for 48 h. The tensile, flexural and impact results show that the silane surface–treated *Caryota urens* fibre–reinforced epoxy composite possesses improved mechanical properties than the base- and acid-treated *Caryota urens* fibres in the epoxy composite. Similarly, the inter-laminar shear strength (ILSS) of silane-treated *Caryota urens*–reinforced epoxy composite gives the highest value of 28 MPa. The TGA shows a large mass loss for both acid- and base-treated *Caryota urens* epoxy composites whereas the silane-treated *Caryota urens* in epoxy composite retains the thermal stability. The fatigue behaviour of silane surface–modified *Caryota urens* epoxy composite shows the highest fatigue life cycle of 18,315 for 25% of maximum tensile stress. The SEM micrographs show improved adhesion for silane-treated CUF than those treated with acid and base. This *Caryota urens* fibre–reinforced epoxy composite could be used in automobile body parts, domestic appliances, defence products and lightweight mini-aircrafts.

Keywords Biocomposite · Natural fibre · Surface treatment · TGA · Mechanical · Fatigue

1 Introduction

In recent years, huge attention is being given to natural fibres as a sustainable alternative to artificial fibres in composite-making process [1]. Environmental concerns like gradual increase in non-degradable plastic waste have generated significant interest in biocomposites from renewable and environmentally friendly natural sources [2, 3]. Many researchers have made biocomposites using natural fibres such as jute, flax, hemp, kenaf, ramie, sisal, pineapple leaf, banana, bamboo and bagasse

and studied their mechanical, wear, thermal and time-dependent behaviour. The products like seat base, door panels, noise insulator panels, headliner panel, engine insulator and internal engine covers are the specific outcome of these composites; thus, natural fibre–reinforced polymer composites are having predominant application in the automotive industry [4]. Obi et al. [5] studied the feasibility of using *Borassus* fibre in polymer composite. Alkali and coupling agent–based chemical modification of *Borassus* fibres was successfully accomplished to demonstrate the effect on mechanical properties. The interfacial bonding between matrix and reinforcement was found to be improved by surface modification of fibres. The author concluded that the treatment of coupling agent in composite led to increment in mechanical properties such as tensile, flexural and impact toughness.

Uma Maheswari et al. [6] studied the effect of alkali treatment and polycarbonate coating on natural fibre. The tensile strength analysis has been carried out for the composites, and it was concluded that the interfacial bonding between the polymer matrix and fibre fabric was found to be improved by alkali treatment. The alkali treatment produced a rough surface on fibre via leaching, which improved the adhesion. Alavudeen et al. [7] reviewed the use of woven banana and

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kenaf fibre as reinforcing agent in a polymer-based composite. The authors concluded that the strength of composite was modified based on the weaving pattern, chemical modification and curing time. Thus, most of the researchers have done their studies in acid- and base-treated natural fibre-reinforced polymer composites and their relative effect in tensile, flexural, impact, wear and time-dependent properties. Very few researchers only studied and compared the silane surface modification and its effects with acid- and base-treated fibres. However, no research based on new fibre *Caryota urens* has been done by the researchers.

Based on the previous literature, the research gap for the present study was made. The present study is aimed to explicate the significant advantage of surface treatment process on novel *Caryota urens* fibre and its relative effect on mechanical, thermal and fatigue behaviour of epoxy composite. *Caryota urens* fibre could be selected as a sustainable fibre material for making high-performance polymer composites. This fibre is from the palm family as a flowering species and native of the Indo-Malayan region. They used to grow in the fields and rainforest ranges. It usually has a solitary trunk that measures up to 1500 cm in height and 25–30 cm wide. It has a density of 0.84 g/cm³ and is highly sustainable than other natural fibres in its class. Its tensile strength is higher than many natural fibres like *areca*, *spinifex littoreus* and coir. Krishnan et al. [8] have done a research study on making automobile break pad using *Caryota urens*, CaCO₃ and SiC fillers. The authors confirmed that the novel *Caryota urens* fibre-based epoxy composite was the best alternate for toxic asbestos material. Shetty et al. [9] investigate the mechanical and tribological behaviour of chopped *Caryota urens* fibre in polyester composite. The novel *Caryota urens* fibre in polyester resin improved the wear and mechanical properties. This improvement is attributed to the presence of hard hemi-cellulosic substance in the novel *Caryota urens* fibre. Antony et al. [10] investigate the effect of adding silane surface-modified *Caryota urens* fibre along with aluminium 2024-T3 foil as multi-stacking sequenced form. The author concluded that the addition of *Caryota urens* fibre along with aluminium 2024-T3 improved the toughness of composite by effective load sharing phenomenon. Thus, all previous studies revealed that the novel *Caryota urens* fibre has a significant role in high-strength composite-making process. Moreover, this fibre could acts as a highly sustainable material for composite-making industries. These mechanical, thermal and fatigue properties improved fibre-reinforced epoxy composite could be used as an alternate material for structural, automobile and domestic applications [11].

2 Materials and methods

2.1 Materials

The epoxy resin used in this present investigation was an Araldite LY556 type of resin with a density of 1.18 g/cm³ and a molecular mass of 190.1 g/mol. A curing agent used for curing the composites is TETA HY951 with a density of 1.04 g/cm³. Both the resin and hardener were purchased from Huntsman India Pvt. Ltd. The silane surface modifier 3-aminopropyltrimethoxysilane (APTMS) was purchased from Sigma Aldrich, USA. The other chemicals and salts (H₂SO₄ and NaOH) of 1 N were prepared in Metro Composites R&D Centre, Chennai, India. Other elements like ethanol, acetic acid and distilled water are purchased from Merck India, Ltd.

2.2 Fibre extraction

The *Caryota urens* plant for preparation of short fibre was collected from Kanyakumari District of Tamil Nadu, India. The sized fibre sheath bundles were separated from the dried flower and immersed in water for 7 days to perform microbial degradation. The dirt and foreign material in the *Caryota urens* fibre sheath were removed in water during the immersion process. The leftover sheath on the fibre bundle was removed by the hammering process and the separated fibre from the sheath was dried at an elevated temperature of 110 °C to remove moisture [12, 13]. The prepared fibres are in chopped form having a length of about 100 mm. The fibre has cellulosic, hemi-cellulosic and holo-cellulosic content values of 62.1, 8.2 and 0.4 wt% with a density of 0.84 g/cm³. Similarly, the lignin content measures 14.2 wt% with ash content of 8.42 wt%. Figure 1 shows the *Caryota urens* fibre extracted from the source. Figure 1(a) shows the fibre plant, b shows the fibre sheath bundle after immersion, c shows the extracted fibre after hammering and d shows the SEM image of a single fibre. From the figure, it is observed that the average diameter of single fibre was 175 µm.

2.3 Chemical treatment of fibre

Various chemical treatments such as acid (H₂SO₄) and base (NaOH) were done by simple immersion technique. In this, 1-N concentration of H₂SO₄ and NaOH was prepared using wet chemical method. The required quantity of *Caryota urens* fibre was immersed in the acid and base solution of 1 N for about 10 min and separated out. The separated surface-treated fibre was then oven-dried to remove the moisture and ready for making composites. Similarly, the silane surface treatment was done using aqueous solution method. In this, the silane substance of 4 wt% was mixed with ethanol-water solution and mixed gently. The *Caryota urens* fibre of the required quantity was then immersed into the aqueous-silane solution

Fig. 1 Fibre extraction stages. (a) Fibre fruit, (b) fibre with sheath, (c) extracted fibre and (d) SEM image of a single fibre



for 10 min. Finally, the treated fibre was separated out from the solution and dried off using a hot oven at 110 °C [14, 15]. Figure 2 shows the graphical infograph of silane surface treatment process. Scheme 1 shows the condensed silane reaction on *Caryota urens* fibre.

Figure 3 shows the SEM images of (a) acid-, (b) base- and (c) silane-treated *Caryota urens* fibres. It is observed that the after acid treatment, there are micro-pits on the fibre surface, which may enhance the adhesion between fibre and resin. Similarly, the base-treated fibre's surface shows a leached portion after treatment. This leached portion on the surface is the outcome of the removal of cellulose and hemicellulose. However, in the silane treatment, the surface remains smooth due to the coverage of silane substance, which could possibly improve the adhesion phenomenon between fibre and resin during composite-making process. Figure 3(d) shows the EDAX report of silane surface-treated fibre. The presence of oxygen, silicon, carbon and nitrogen confirms the presence of NH_2 functional group on silane-treated fibre.

Figure 4 shows the FT-IR spectra of (a) as-received, (b) neat amino silane (APTMS) and (c) silane-treated *Caryota urens* fibre. Figure 4(a) shows no functional peaks, which indicates an untreated form of fibre. Figure 4(b, c) shows significant peaks. A peak at 3422 cm^{-1} represents the presence of a functional amine group (NH_2) on both the spectral graphs of silane and silane-treated fibre. A peak at 2900 cm^{-1} represents the C-H stretch on both neat silane and silane-treated fibres, which is from the propyl group of silane. Similarly, peak at 1470 cm^{-1} indicates the C-H bending of the attached

propyl group in amino silane and silane-treated fibre's surface. Finally, the peak at 828 cm^{-1} represents the presence of Si-OCH_3 structure in pure silane but it is missing in the silane-treated fibre. Thus, the process of silane surface treatment induced amine (NH_2) functional group on *Caryota urens* fibre's surface, which could possibly improve the adhesion of fibre with resin [16].

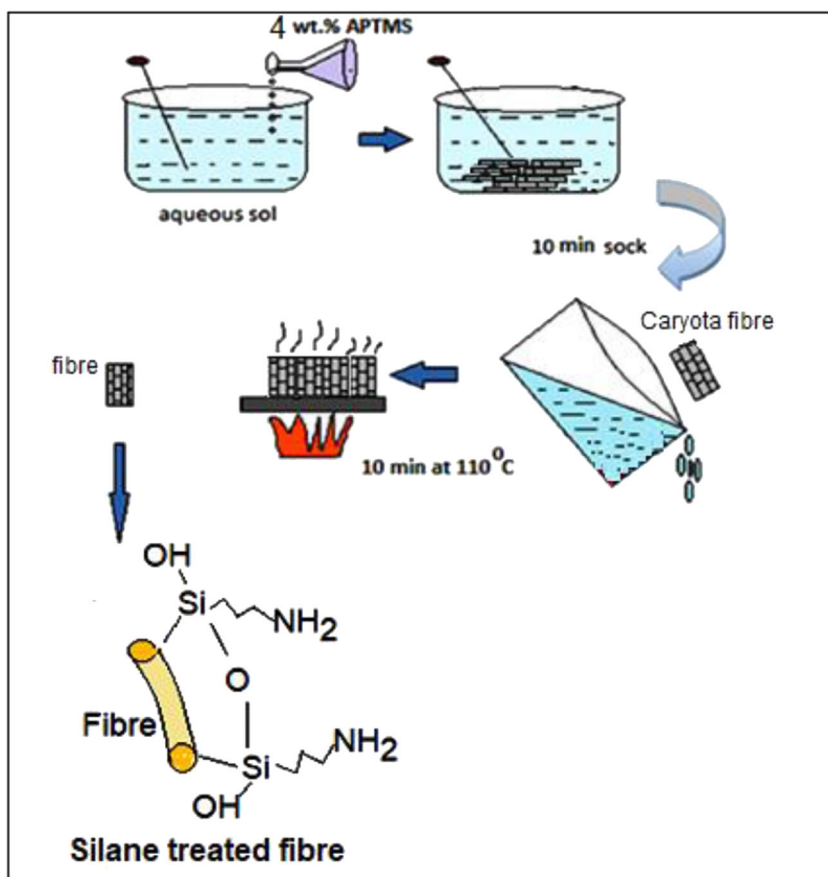
2.4 Formation of composite

The composite laminates were prepared using hand layup method. Firstly, a liberal coat of wax was applied on the mould and the hardener mixed epoxy resin was then poured into the mould. 30 vol.% of surface-treated chopped fibre was laid into the resin mix and cured completely. The room temperature-cured epoxy composites were post cured at 120 °C using a hot oven for 48 h [17, 18]. Figure 5 shows the fabricated composite in this present study. Table 1 shows the various compositions of treated and untreated fibre-reinforced epoxy composite.

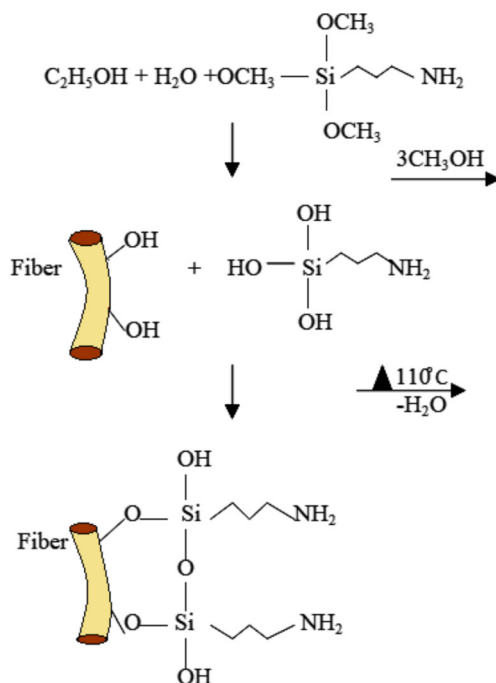
3 Characterization of composites

The hand layup-prepared fibre-reinforced epoxy composites were checked for surface defects. The tensile, flexural and inter-laminar shear strength (ILSS) testing was performed on the composites in accordance with ASTM D-3030, 790 and 2344 respectively. The test was performed using a universal

Fig. 2 Graphical representation of the silane treatment process



testing machine (INSTRON 4355, UK) with a cross-head speed of 1.5 mm/min. The Izod impact study on composite was done based on ASTM D-256 using a mini-impact tester



Scheme 1 Condensed silane reaction on natural fibre surface

(Krystal Equipment, India, Pvt. Ltd) having 20-J measuring capacity. The thermogravimetry study on epoxy composite was done using a TGA thermo-scanner NETZSCH STA Jupiter, 409 PL Luxx, Germany. The samples were scanned from 30 to 700 °C at the heating rate of 10 °C/min. Similarly, the fatigue behaviour of epoxy composites was tested using a fatigue load frame (MTS, Landmark 370, USA). Working stress values of 32.5 (25%), 65 (50%) and 97.5 (75%) MPa of ultimate tensile stress, frequency of 5 Hz, stress ratio of 0.1 and temperature of 28 °C were set as testing parameters to find the variation in the fatigue life cycle of composite and to plot S-N curve. The micrograph images were captured using a scanning electron microscope (HITACHI, S1500, Japan).

4 Results and discussion

4.1 Mechanical properties

Table 2 shows the tensile strength, tensile modulus, flexural strength and flexural modulus of as-received and various surface-treated epoxy composites. Figure 6 shows the stress-strain relationship graphs of various composites tested. It is observed that the pure epoxy resin gives tensile strength and modulus of 71 and 2624 MPa. The extreme brittleness of

Fig. 3 SEM image of (a) acid-, (b) base-, (c) silane-treated *Caryota urens* fibre and (d) EDAX spectrum of silane-treated fibre

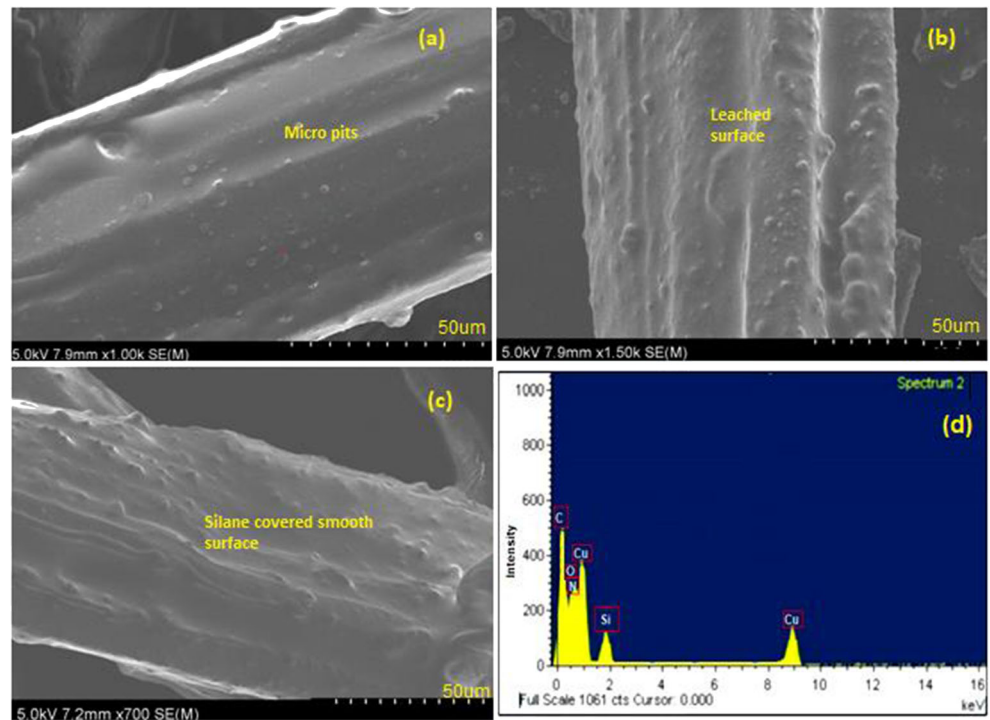


Fig. 4 FT-IR spectra of (a) untreated, (b) pure silane and (c) silane-treated fibre

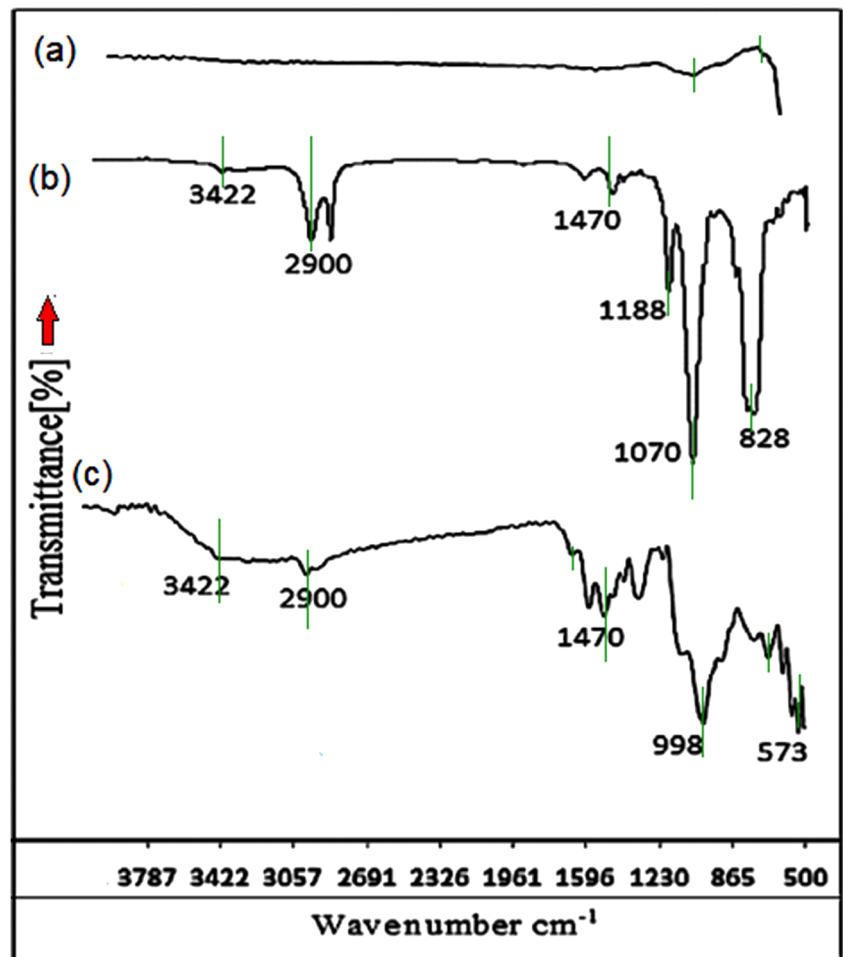




Fig. 5 *Caryota* fibre–reinforced epoxy composite

epoxy matrix gives this significant lower result in tensile properties. But addition of *Caryota urens* fibre into epoxy resin improved the tensile strength and modulus. The improved tensile strength and modulus of 98 and 4351 MPa were observed for EU composite designation. This improvement is the cause of adding cellulosic *Caryota urens* fibre into epoxy resin, which improved the smooth load transfer throughout the matrix [19].

It is observed that further addition of base-, acid- and silane-treated *Caryota urens* fibre into epoxy resin improved the tensile properties than untreated fibre epoxy composite. The tensile strength and modulus of EU composite designation measure 118 and 5782 MPa. Similarly, the EA composite designation measures 138 and 6174 MPa respectively. This marginal improvement in acid-treated *Caryota urens* fibre–reinforced epoxy composite is the reason of improved adhesion of fibre with the matrix. In acid treatment, small micropits were produced on the fibre surface, which in turn improves the adhesion with epoxy resin. But in base treatment, the fibre surface is rough and gets leached higher. Thus, the cross-sectional reduction in the fibre led the composite to deform at the lower implication of load itself [20].

But it is observed that the addition of silane surface–treated fibre into epoxy resin significantly improved the tensile

strength and modulus. The improvement in strength and modulus is near 57 and 60% on comparing with pure epoxy resin. Moreover, the improvement is much higher when comparing with EB and EA composite designation. This notable improvement is the reason of chemically modified *Caryota urens* fibre in epoxy resin, which improved the bonding behaviour between fibre and matrix. In both base and acid treatments, the process makes surface leaching and weakening of surface molecules. But in silane surface treatment, the silane substance not disturbing the original cross-section of fibre and just covers it with a highly reactive functional group (NH_2). Thus, improvement in tensile strength and modulus is observed [21].

It is observed that the flexural strength and modulus of pure epoxy resin measures 113 and 2142 MPa. This lower bending strength and modulus is the cause of highly cross-linked epoxy molecular chains, which restrict the stretching of molecules during bending. Thus, lower in flexural strength and modulus is observed. But further addition of surface-modified *Caryota urens* fibre of 30 vol.% into epoxy resin improved the flexural strength. The improvement in flexural strength and modulus of 36.4, 41.4 and 52.3% and 60.6, 62.5 and 67.1% were observed for composite designations EB, EA and ES respectively. This improvement is the cause of the effective load sharing ability of *Caryota urens* fibre in epoxy resin and also the adhesion improvement due to the surface treatment process. It is noted that among all surface-treated fibres in epoxy resin, the silane surface–treated *Caryota urens* natural fibre (CUF) gives improved flexural strength and modulus. This improvement is the cause of improved chemical bonding due to the reaction of NH_2 functional group with matrix [22]. Figure 7 shows the SEM tensile fractograph of various surface-treated epoxy composites. Figure 7(a) shows the fractograph of as-received fibre in epoxy resin matrix. The fibre pullout on the fractured portion indicates poor adhesion of fibre with matrix. This is because of no adhesion mechanism on as-received fibre’s surface. Figure 7(b, c) explicates marginal fibre pullout but not much as untreated fibre. This improvement is because of mechanical locking of fibre with matrix, which could possibly improve the adhesion. But Fig. 7(d) shows the fractograph

Table 1 Compositions of treated and untreated fibre epoxy composites

| Composite Designation | Untreated fibre (vol.%) | NaOH-treated fibre (vol.%) | H ₂ SO ₄ -treated fibre (vol.%) | Silane-treated fibre (vol.%) | Epoxy (vol.%) |
|-----------------------|-------------------------|----------------------------|---|------------------------------|---------------|
| E | - | - | - | - | 100 |
| EU | 30 | - | - | - | 70 |
| EB | - | 30 | - | - | 70 |
| EA | - | - | 30 | - | 70 |
| ES | - | - | - | 30 | 70 |

E epoxy, U untreated, B base, A acid, S silane

Table 2 Mechanical properties of *Caryota urens* fibre epoxy composites

| Composite designation | Tensile strength (MPa) | Tensile modulus (MPa) | Flexural strength (MPa) | Flexural modulus (MPa) | Izod Impact (J) | I.L shear strength (MPa) |
|-----------------------|------------------------|-----------------------|-------------------------|------------------------|-----------------|--------------------------|
| E | 71 | 2624 | 113 | 2142 | 0.43 | - |
| EU | 98 | 4351 | 139 | 4982 | 3.42 | 19 |
| EB | 118 | 5782 | 177 | 5446 | 3.22 | 23 |
| EA | 138 | 6174 | 193 | 5721 | 3.14 | 25 |
| ES | 164 | 6557 | 237 | 6522 | 4.65 | 28 |

of silane-treated fibre in epoxy resin matrix. The matrix phase is finely mangled as fibre phase, which indicates the improved adhesion of fibre with matrix.

It is noted that the Izod impact toughness shows conclusive results. The pure epoxy resin gives very lower impact toughness of 0.43 J. This poor energy absorption is the cause of no load-bearing micro-mechanisms with in epoxy matrix [23]. It is noted that further addition of untreated *Caryota urens* into epoxy resin improved the sudden load-bearing capability. The presence of fibre observes the sudden load and reduces the stress intensity factor in the matrix, thus improving the energy absorption rate. It is further noted that the addition of acid- and base-treated *Caryota urens* into epoxy resin reduced the impact of energy absorption. The reduction of 6% and 9% was observed for composite designations EB and EA on comparing with EU designation. This reduction in Izod impact strength is the reason for reduction in cross-sectional area of *Caryota urens* fibre after acid and base surface treatments. During these treatments, the fibre surface gets leached and eroded. Thus, the eroded cross-section could not bear the applied load and store high energy [24]. But the addition of silane surface-treated *Caryota urens* into epoxy matrix improved the Izod impact toughness. The improvement of 26.4% was observed for ES composite designation. This improvement is the reason for improved adhesion of fibre, which could transfer the sudden load very

smoothly throughout the matrix. Thus, high energy could be stored in the composite. There is no reduction in cross-sectional area of fibre; thus, the load-bearing capability is higher for ES composite designation [25].

The inter-laminar shear strength of composite shows a significant result in laminar strength. It is observed that the untreated *Caryota urens* in epoxy resin gives ILSS of 19 MPa. But the addition of base-, acid- and silane surface-treated fibre in epoxy resin improved the ILSS. Among all surface-modified composite designations, the silane-treated *Caryota urens*-reinforced epoxy composite shows the highest ILSS of 28 MPa. This is near 19% of improvement when compared with EU composite designation. This improvement is because, in acid and base treatment, the adhesion was improved via mechanical locking of fibre with matrix using the treated porous surface. But in silane treatment, the chemical reaction between the fibre and matrix ensures good bonding strength than the mechanical locking of fibre [26]. Thus, improvement in ILSS was observed for ES composite designation than other composites.

4.2 Thermal properties

Figure 8(a, b) shows the TG thermogram and derivative TG of epoxy resin and its composites. It is observed that the pure epoxy resin gives the initial, rapid and final decomposition temperatures of 323 °C, 420 °C and 492 °C. It is noted that further addition of untreated and surface-treated *Caryota urens* in epoxy resin gives reduced thermal stability. The untreated fibre in epoxy resin offers a marginal decrement of 3.5% in initial decomposition temperature. This reduction is the cause of poor thermal stability of natural fibre, which could allow evaporating the inbuilt moisture and the soft lignin [27]. Similarly, the addition of acid- and base-treated *Caryota urens* fibre in epoxy resin further reduces the thermal stability. The surface leached fibre via acid and base treatment further reduced thermal stability. This poor thermal stability is the cause of leaching phenomenon during the acid and base treatment process. The leaching could remove all thermal barriers in the outer shell of *Caryota* fibre; thus, poor thermal stability is observed. The increases in temperature induce

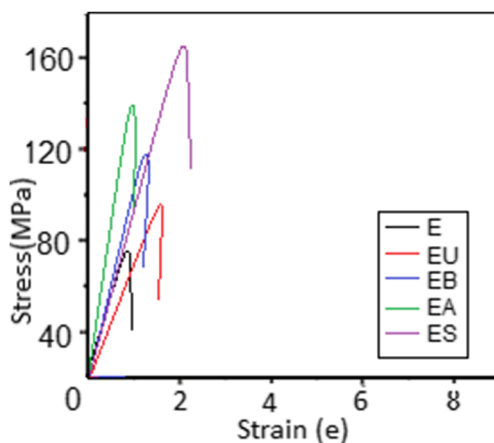
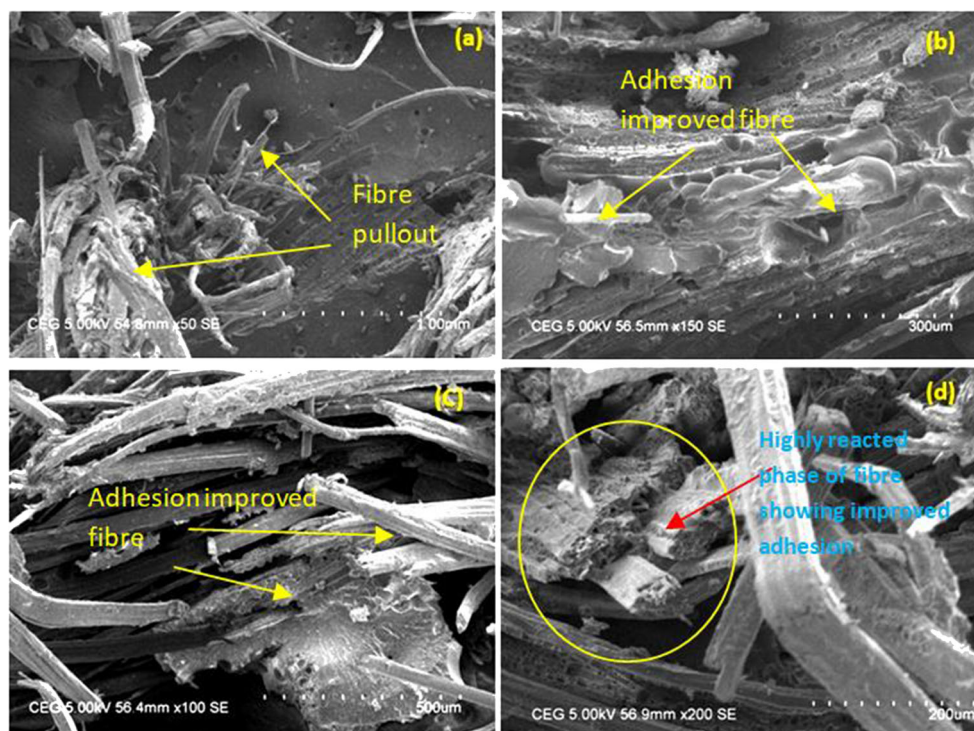
**Fig. 6** Stress-strain relationship graph

Fig. 7 SEM tensile fractograph of (a) as-received, (b) acid-treated, (c) base-treated and (d) silane-treated *Caryota urens* fibre-reinforced epoxy composite



evaporation of moisture and other volatile content at early temperature itself. Thus, the composites show large mass loss at early temperature. It is noted that further adding of silane surface-modified *Caryota urens* into epoxy resin maintains the thermal stability. The ES composite designation does not improve the thermal stability but it maintains the lasting of thermal stability even after the addition of *Caryota urens* fibre. This high thermal stability is the cause of silane coverage on *Caryota* fibre. The silane-coated *Caryota* fibre needs higher temperature to deplete the silane layer since the evaporation of silane needs a temperature more than 320 °C; thus, the initial decomposition temperature never reduced for ES composite designation [28].

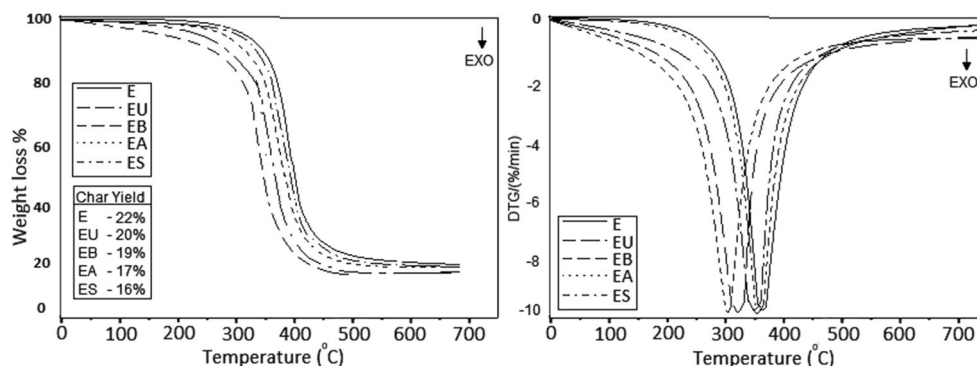
It is observed that the char yield of various composite gives significant results. The pure epoxy resin gives the char yield of 22% after complete burning. Similarly, the EU, EB and EA composite designations show the char yield of 20%, 19% and

17% respectively. This lower percentage shows there is no remaining portion of fibre, which retained at this final temperature [29]. But in ES composite designation, the char yield is 16%, which indicates the chemically bonded fibre is still present with the epoxy matrix and maintains higher char [30]. Thus, the process of silane treatment improves the thermal stability of *Caryota urens* fibre-reinforced epoxy composite.

4.3 Fatigue behaviour

Figure 9 shows the S-N curve of pure epoxy resin and its composites in 25, 50 and 75% of maximum tensile stress. It is observed that the pure epoxy resin gives very lower fatigue life counts of 621, 340 and 272. This lower fatigue life count is the cause of high brittle nature of epoxy resin. The retention of plastic strain in the epoxy molecular chain is the cause of this

Fig. 8 TG and DTG graphs of *Caryota urens* epoxy composite



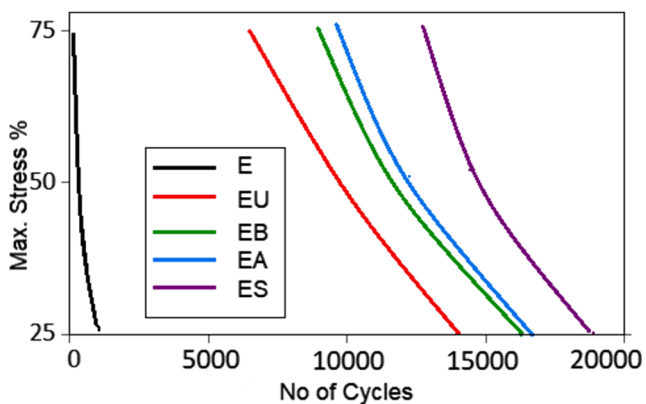


Fig. 9 S-N curve of composites

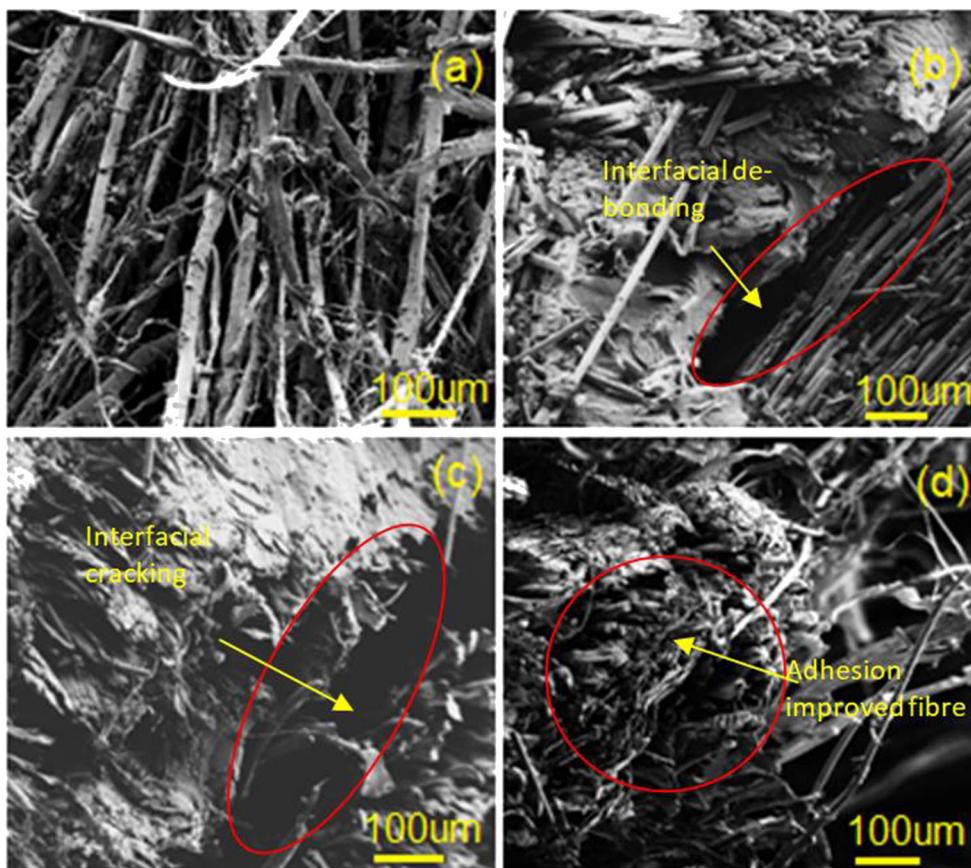
lower fatigue life cycle. It is noted that when applied stress percentage is increased, the composite’s life decreased. This is because at higher applied stress, the degree of microcrack formation and growth is high, which leads the composite to deform plastically [31].

It is observed that the addition of as-received, base-, acid- and silane-treated *Caryota urens* fibre into epoxy resin gives a notable improvement in fatigue life count. The fatigue counts of 14,763, 16,720, 17,455 and 18,315 were observed for EU, EB, EA and ES composite designations at 25% of maximum stress. Similarly, fatigue counts of 12,744, 13,711, 14,280 and

16,773 and fatigue counts of 7122, 9220, 10,980 and 13,872 were observed for EU, EB, EA and ES composite designations at 50 and 75% of maximum stress, respectively. This improvement is the reason of effective load absorption behaviour of *Caryota urens* fibre in epoxy resin. The presence of micro-fibril *Caryota urens* effectively transfers the load and made the matrix tougher. Thus, the storage of plastic strain in every loading cycle reduces, which in turn improves the fatigue life count of epoxy composite. Moreover, the propagation of microcracks is another cause of fatigue failure in composites [32]. It is observed that the base- and acid-treated *Caryota urens* in epoxy resin improved the fatigue behaviour on comparing with EU composite designation. This improvement is because of improved adhesion due to the mechanical self-locking of resin with fibre surface. The adhesion improved fibre transfer the load uniformly and reduces the stress intensity factor as well as accumulation of plastic strain. The toughness of matrix also increases by lowering the crack density and crack growth. Thus, improvement in fatigue behaviour is observed [33].

It is observed that further addition of silane surface-modified *Caryota urens* into epoxy resin gives the highest fatigue life cycle of 18,315 at 25% of maximum tensile stress. This is near 19.4% of improvement than EU composite designation. This improvement is the cause of effective bonding

Fig. 10 SEM fractographs of (a) as-received, (b) base-, (c) acid- and (d) silane-treated *Caryota urens* fibre epoxy composite under fatigue load



of *Caryota urens* fibre with matrix via a chemical reaction. The functionally modified *Caryota urens* reacted with epoxy resin during the curing process; thus, the load transfer from matrix to fibre becomes smooth, which in turn reduces the crack growth at the fibre matrix interface and suppresses the interfacial delamination [34]. Thus, on comparing with acid and base treatments, the silane surface treatment fetches improved fatigue count for epoxy composites. Figure 10 shows the scanning electron microscope image of a fatigue fractured composite sample. Figure 10(a) is the fractured sample of EU composite specimen. The fibre shows almost nil adhesion with matrix. The fibre surface is not having any evidence for matrix debris. Figure 10(b, c) illustrates the fatigue fractured composite specimen of EB and EA. Both the composites show marginally improved fibre adhesion due to mechanical locking. The fractographs revealed that fibre matrix debonding is the primary cause of fatigue failure in composites. Figure 10(d) shows the fatigue fractured ES composite specimen under repeated cyclic load. The fibre fractured finely without any fibre pullout, which indicates improved adhesion of fibre with matrix via a chemical reaction.

5 Conclusions

Novel *Caryota urens*-reinforced epoxy composites have been prepared with untreated, base-treated, acid-treated and silane-treated fibre forms. The significance of various surface treatment processes was investigated via mechanical, thermal and fatigue behaviours. The fibre was successfully prepared from its bio-source and used in this study as a sustainable material. The composites were prepared using hand layup method and post cured at elevated temperature. The summary of the present study as follows.

- 1) The tensile and flexural results show that the composite, which is made of surface-treated *Caryota urens*, gives improved tensile, flexural strength and modulus. Among all surface treatment, the silane surface-treated composite designation ES gives the highest tensile and flexural strength.
- 2) The SEM micrographs of silane surface-modified *Caryota urens* shows improved adhesion with matrix than other surface-treated *Caryota urens* fibres.
- 3) The Izod impact toughness of EB and EA composite designation gives bit lower values than untreated *Caryota urens* composite designation (EU). But the silane surface-treated (ES) composite designation gives improved Izod impact toughness.
- 4) The ILSS of untreated *Caryota urens* epoxy composite shows a lower value of 19 MPa. But the silane surface-treated *Caryota urens* in epoxy resin gives higher ILSS of 28 MPa.
- 5) The thermal stability of untreated, base- and acid-treated *Caryota urens* gives large mass loss than pure epoxy resin whereas the composite reinforced with silane surface-treated *Caryota urens* fibre retains the thermal stability as equal as epoxy resin.
- 6) The silane surface-treated *Caryota urens*-reinforced epoxy composite gives the highest fatigue life count of 16,773, which is near 24% of improvement on compared with EU composite designation. The other two surface modifications (acid and base) fetched lower value than silane surface treatment.
- 7) Thus, in the process of making high-strength and high-toughness natural fibre composite, the silane surface treatment would be a preferable method than other chemical methods since the other treatments affect the original cross-section of fibre, whereas in silane treatment, the actual cross-section is not altered and also boosts up the mechanical, thermal and time-dependent properties.

Acknowledgments The authors of this research work have deeply acknowledged the work rendered from Metro Composites Research and Development Centre, Chennai, India. www.metrocomposites.org, metrocompositesrd@gmail.com.

Compliance with ethical standards

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

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