Development and characterization of jute composites for sustainable product: effect of chemical treatments and polymer coating

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Abstract
In this work, woven jute fibres were subjected to ecofriendly and chemical treatments (alkali, benzoylation and sodium bicarbonate) and PLA coating in order to improve the adhesion with epoxy thereby improvement in the performances of their composites. Treated and coated jute fibres reinforced epoxy composites were prepared by hand lay-up technique keeping constant 30 wt% of fibres content in the each composite. Mechanical properties (i.e. tensile, flexural and impact) and dynamic mechanical properties (i.e. storage modulus, loss modulus, damping and glass transition temperature) of prepared composites were studied. The outcomes from the experimental results suggested that benzoyl chloride treated and PLA coated jute composite exhibited the best performance as compared to other all the composites. There were 21%, 40.6% and 27.5% improvement in tensile strength, flexural strength and impact strength respectively for this composite as compared to that of untreated jute fibre reinforced composites. Moreover, storage and loss modulus of jute composites were also significantly enhanced by treatment and coating. The present developed composites can be used for medium strength application in the field of automobile, building and construction and packaging.

1. Introduction

Many researchers have shown a deep interest in natural fibres for the development of ecological and environmental friendly materials owing to its characteristics like low density, low cost, high specific strength, abundantly available, non-toxicity and biodegradability [1, 2]. Because of such good properties of natural fibres, their polymer-based composites are being used in many applications such as packaging, furniture, office and house products, building and constructions, sports and electrical equipments [1, 3, 4]. Now in current product development, natural fibres composites have also been used in the various parts of automobiles such as door panels, seat covering, seat surface, floor panels and insulation components [1, 3, 5]. In spite of such good properties, they have some drawbacks also such as higher water absorption, moderate strength, poor compatibility and durability [1, 5]. Therefore, researchers have the challenges of reducing these drawbacks of the natural fibres. A good number of research works has been already carried out to overcome these limitations using chemical treatments.

The main effect of chemical treatments is to reduce the polarity of the surface of natural fibres to increase interfacial adhesion with polymer matrix. Arthanarieswaran et al [6] found that embedded composites with alkali treated fibres demonstrated a better mechanical and thermal properties. Young's modulus and tensile strength alpha fibre/polypropylene composite were improved by 23% and 16% respectively after alkali treatment [7]. Some other researchers also found improvement in the properties of the bio composites after alkali treatment [8–10]. Tensile strength and tensile modulus of bamboo composite [11] and palmyra palm leaf stalk fibre composites [12] were also improved by benzoyl chloride treatment. In some other studies, benzoyl chloride treatment was found effective to enhance the performances of the composites [13, 14]. Sever et al [15]
observed enhancement in tensile, flexural and interlaminar properties of jute/polyester composites by silane treatment. In some other studies, silane treatment was found effective to enhance the performances of the composites [16–18]. Instead, chemical treatments of fibres, ecofriendly treatment (i.e. sodium bicarbonate treatment) was also found effective to enhance the performances of the composites; hemp/polyester [18], sisal/epoxy [19], flax/epoxy [20], sisal/PLA [21] and coir/polyester [22]. Mechanical and morphological properties of coir fibres [23] and mechanical and thermal properties of sugar palm fibre were also improved by sodium bicarbonate treatment [24]. In recent few years, the researchers have used polymer coating on the surface of natural fibres to enhance its performances as well as its composite’s also; sisal fibres [25], sisal/polyester [26], jute/polyester [27] and jute/vinyl ester [28].

Now, it can be concluded that the limitations of the natural fibres were found to be significantly overcome resulting in enhanced properties by the various surface modification methods. Chemical and ecofriendly treatments are found to be useful to improve the properties of the natural fibres and its composites up to some extent. However, a much improvement is needed for advanced applications of the natural fibres and its composites which might be achieved by using polymers coating on treated natural fibres. To the best knowledge of the authors, it can be reported that such type of work as effect of a novel surface modification (alkali, sodium bicarbonate and benzoyl chloride treatment along with PLA coating) on the properties of jute/epoxy composite has not been attempted by any author. Hence, effect of the various chemical treatments and PLA coating on the properties of jute/epoxy composite has been presented in present work.

2. Materials and methods

2.1. Materials

Epoxy resin LY556 (Araldite) as the matrix along with Hardener HY951 (Araldite) is provided by universal Enterprises Kanpur, Uttar Pradesh, India. The properties of Epoxy are: density = 1.15 g cm\(^{-3}\), tensile strength = 83–93 MPa, tensile modulus = 31–33 GPa and viscosity = 10 000–12 000 MPa s. Woven jute fibres were supplied by Vruksha Composites and Services, Chennai, Tamil Naidu. The properties of jute fibres are: density = 1.3 g cm\(^{-3}\), elongation = 1.7%, tensile strength = 230 MPa and tensile modulus = 26.5 GPa.

2.2. Surface modification of woven jute fibre

2.2.1. Alkali treatment

Oushabi et al [8] has observed that 5% NaOH treatment is the optimum concentration for the highest properties of date palm fibers/polyurethane composite. Therefore, 5% NaOH treatment is applied on the woven jute fibre in the present work. The fibres were immersed in alkaline solution at 30 °C for 4 h. After removing for the solution, fibres were washed many times with clean running water then immersed into very dilute HCl solution to remove the NaOH adhering from the surface of the fibres. Finally, fibres were again washed many times with water and then dried in a hot air oven maintained at 70 °C for 24 h. The reaction of alkali treatment with fibre is provided below:

\[
\text{Fibre–OH} + \text{NaOH} \rightarrow \text{Fibre–O}^\cdot\text{Na}^+ + \text{H}_2\text{O}
\]

2.2.2. Sodium bicarbonate treatment

Since Fiore et al [19] suggested that for optimum properties of sisal/epoxy composite 10% NaHCO\(_3\) treatment is perfect. On basis of their recommendations, 10% NaHCO\(_3\) treatment is applied on woven jute fibre in the present work. The fibres were immersed in 10% NaHCO\(_3\) solution at 30 °C for 120 h. The fibres were then washed several times with clean running water followed by immersion of these fibres in very dilute HCl solution in order to remove the NaHCO\(_3\) adhering from the surface of the fibres. Then treated fibres were dried at room temperature for 24 h, subsequently dried in a hot air oven maintained at 70 °C for 24 h. Reaction of sodium bicarbonate treatment with fibre is provided below:

\[
\text{NaHCO}_3 + \text{H}_2\text{O} \rightarrow \text{Na} + \text{HCO}_3^-
\]

\[
\text{HCO}_3^- + \text{H}_2\text{O} \rightarrow \text{H}_2\text{CO}_3 + \text{OH}^-
\]

\[
\text{Fibre – OH} + \text{NaOH} \rightarrow \text{Fibre – O}^\cdot\text{Na}^+ + \text{H}_2\text{O}
\]

2.2.3. Benzoyl chloride treatment

For benzoyl chloride treatment, 18% NaOH treatment followed by benzoyl chloride treatment on the fibres have been already applied [10]. Therefore, 18% NaOH treatment is applied on woven jute fibres followed by benzoyl chloride treatment. The fibres were immersed into 18% NaOH solution and agitated vigorously with 50 ml benzoyl chloride at room temperature. The mixture was allowed to stand for 15 min, followed by filtering,
washing with water, and drying at room temperature. The fibre were then soaked in ethanol for 1 h to remove the unreached benzoyl chloride and finally washed with water and dried in an oven at 60 °C for about 24 h. Reaction of benzoyl chloride treatment with fibre is provided below:

\[
\text{Fibre–OH} + \text{NaOH} \rightarrow \text{Fibre–O}^-\text{Na}^+ + \text{H}_2\text{O} \\
\text{Fibre} - \text{O}^-\text{Na}^+ + \text{CIC} \rightarrow \text{Fibre} - \text{O}^-\text{C} + \text{NaCl}
\]

2.2.4. PLA coating on treatment fibres
Initially, PLA pellets were immersed into the chloroform solution for 8 h. Subsequently, the solution was stirred manually and heated to 60 °C to make sure the uniform dispersion of PLA into chloroform solution. After making PLA solvent, woven jute fibres were dipped and then taken out after 5 min soaking time. Finally, coated fibres were dried at room temperature for 24 h and then finally dried at 60 °C for 4 h in a hot air oven. A schematic diagram of PLA coating on jute fibre is provided in figure 1.

2.3. Fabrication method
For fabrication of the composites, first of all matrix material was prepared by mixing epoxy resin and corresponding hardener in a ratio of 10:1. Treated and coated woven jute fibres with constant 30 wt% loading were reinforced into epoxy matrix to prepare the composites by hand lay up method followed by static compression. A stainless steel mould of dimensions of 200 mm × 200 mm × 3 mm was used to prepare the composite laminate of 3 mm thickness. A releasing agent was also used to facilitate easy removal of the laminates from the mould after curing. The cast of each composite was cured under a load of 50 kg for 24 h before it was removed from the mould. The specimens were cut in the various dimensions for various testing as per ASTM standards. A schematic diagram of fabrication process is provided in figure 2. Nomenclature used for prepared composites is provided in table 1.
2.4. Tensile test
UTM (Biss, Nano plug and play Servo hydraulic machine, 100 Ton) was used to perform the tensile test of the fabricated composites’ specimens as per ASTM D638. Tensile test on the specimen (dimension: 165 mm × 20 mm × 3 mm) was performed at a crosshead speed of 1 mm min⁻¹. The average values with standard deviations of tensile strength and tensile modulus were reported after testing five specimens of each composite.
2.5. Flexural test

Flexural test of the composite was carried out using a three point bending test on Tinius Olsen H10 K-L (Bi-axial testing machine). The samples were prepared for this test in dimensions 80 mm × 13 mm × 3 mm with 48 mm span length. The flexural test was carried out at room temperature with the crosshead speed of 1 mm min⁻¹ as per ASTM D790. Calculation of flexural strength and flexural modulus were made using the following equations (7) and (8).

\[
\text{Flexural strength} = \frac{3PL}{2xy^2} \tag{7}
\]

\[
\text{Flexural modulus} = \frac{6L^3}{4xy^3} \tag{8}
\]

where \( P \) = maximum load applied (N), \( L \) = span length of the specimen (mm), \( \theta \) = slope of the tangent to the initial straight portion of the load-extension curve, and \( x \) and \( y \) are width and thickness of specimen respectively (mm). The average values with standard deviations of flexural strength and flexural modulus were reported after testing five specimens of each composite.

2.6. Impact test

Digital impact testing machine (Presto Izod/ Charpy Impact testing machine) was used to perform the Izod impact test on the notched specimens as per ASTM D256. Specimens were prepared for this test in dimensions of 65 mm × 13 mm × 3 mm and 2.5 mm notch thickness. The average values with standard deviations of the impact strength and energy were reported after testing five specimens of each composition of the composite.

2.7. Scanning electron microscopy

The SEM, scanning electron microscope (Carl Zeiss EVO MA 15) was used to study the morphological analysis of the fractured samples of tensile, flexural and impact test. All samples of the composites were coated with a very thin layer of gold to make them conductive and prevent electric charging during examinations.

2.8. Dynamic mechanical analysis

The dynamic mechanical properties of the composites were studied using the Dynamic mechanical analyzer (Seiko instruments DMA 6100). The dynamic mechanical properties were determined in 3 point bending test as a function of temperature as per ASTM D 5023. The laminates were cut into samples in dimensions of 50 mm × 13 mm × 3 mm subjected to dynamic mechanical test at 1 Hz frequency and within temperature range of 25 °C–200 °C at heating rate 5 °C min⁻¹.

Figure 3. Tensile load v/s displacement graph of untreated and modified jute composites.
3. Result and discussion

3.1. Tensile testing
The tested values of tensile properties (i.e. tensile strength and tensile modulus) obtained from tensile load v/s displacement diagram (figure 3) for pure and modified jute composites are presented in figure 4. Tensile properties of the present composite was found close to those of reported in the previous published works \([27, 29, 30]\). A positive effect of surface modifications by treatments (alkali, benzoyl chloride and sodium bicarbonate treatment) with PLA of jute fibres were observed in terms of enhancement in tensile properties of its composite. All treated and coated jute composites revealed the higher values of tensile properties as compared to untreated one. Sodium bicarbonate treated with PLA coated composite S(T1) offered the higher values of tensile strength and modulus by 8% and 10% respectively when compared with untreated composite S(T0). Enhancement in tensile properties by sodium bicarbonate treatment could be due to effectiveness of treatment that overcome the polarity of surface of fibres that could be cause of strong bonding \([19]\). Further, alkali treated with PLA coating composite S(T2) has 11% and 22% higher values of tensile strength and modulus as compared to untreated composite S(T0). This could be due to removal of impurities by alkali treatment leads to strong bonding between fibres and epoxy resin thereby improvement in tensile properties \([27]\). Benzoylation reduces the thickness of the fibres by removing unwanted elements (lignin, wax and hemi cellulose) and hence increase in aspect ratio thereby increase in effective area of contact between fibres and matrix. Benzoylation also makes the fibres more reactive due to attachment of benzoyl group \([17]\).

Figures 5(a)–(c) show the SEM images of fracture surface of the specimens subjected to tensile load. Figure 4(a) shows the SEM image of untreated jute composite wherein fibre debonding due to poor adhesion and voids can be observed. Figures 5(b)–(d) shows the SEM images of fractured surfaces of treated and coated composites SC(T1), SC(T2) and SC(T3) respectively. In all figures 5(b)–(d), fractures of the fibres due to strong interfacial bonding credited to treatment and coating can be seen. Moreover, a strong bonding between fibres and matrix was seen for composite SC(T3) credited to benzoyl chloride treatment.

3.2. Flexural test
The experimental values of flexural properties (i.e. flexural strength and flexural modulus) obtained from flexural load v/s extension graph (figure 6) for pure and modified jute composites are presented in figure 7. Flexural results of the present composite was seen close to those of reported in the earlier published works \([27, 28, 30]\). In case of flexural analysis, a similar trend like tensile test result was observed. Each modified composite has higher values of flexural strength and flexural modulus as compared to untreated composite.
S(T0); shows a fruitful effort of treatment and coating. The higher values of flexural strength and modulus mainly depends upon improved interfacial bonding which provides an effective stress transfer [24]. The composite S(T3) offered the maximum value of flexural strength (110.35 MPa) and flexural modulus (8.89 GPa) due to improved stiffness of fibres and its strong bonding with matrix which was 41% and 29% higher than that of composite S(T0). The flexural strength of composite S(T3) was 22.24% and 14.67% more than those of composites S(T1) and S(T2) respectively, whereas flexural modulus was 28.65% and 25.56 more than those of composites S(T1) and S(T2) respectively. It can be observed that benzoylation treatment with PLA coating causes the highest improvement in flexural properties followed by alkali and sodium bicarbonate.
Figures 8(a)–(d) shows the SEM images of fracture surface of the composites samples subjected to flexural load. It can be seen that fracture behaviour of composite samples subjected to flexural load are found quite similar to those of under tensile load. Fibres pull out and voids can be seen in figure 8(a), whereas fracture of fibres, good interfacial bonding and uniform distribution of fibres can be observed in figures 8(b)–(d).

### 3.3. Impact test

Table 2 shows the experimental data of impact energy and strength for untreated and modified jute composites, these related data are also plotted in figure 9. The results of impact test was found similar to tensile and flexural test results. All modified composites showed enhancement in impact strength as compared to untreated composite. It was interesting to observed that benzyl chloride treated and PLA coated composite S(T2) did not
show the highest values of impact strength that was shown by alkali treated and PLA coated composite S(T2) followed by sodium bicarbonate treated and PLA coated S(T1). Strong interfacial bonding might be because of poor fibre pull out and hence lower impact strength for the composite S(T3). The composite S(T2) had the highest value of impact strength (18.7 kJ m\(^{-2}\)) which was 56% more than that of composite S(T0). This could be due to increased area of contact of fibres owing to treatment, which provides the higher capacity to observe the impact energy. The enhancement in impact strength by alkali treatment has been already reported in past literatures \[10, 18\]. Further, enhancement in impact strength by sodium bicarbonate treatment has also been reported by Gupta et al \[18\].

Figures 10(a)–(d) shows the SEM images of fracture surface of the composites samples subjected to impact load. Figure 10(a) presents the poor compatibility of fibres with polymer. On the other hand, a partial fibre pull out can be seen in figure 10(c). In figures 10(b) and (d), fracture of fibres and good bonding can be seen.

### 3.4. Dynamic mechanical analysis

The dynamic mechanical analysis (DMA) was performed to study the viscoelastic properties such as storage modulus (\(E'\)), loss modulus (\(E''\)), damping (\(\tan \delta\)) and glass transition temperature (\(T_g\)) of the untreated and modified jute composites.

#### 3.4.1. Storage modulus

It is amount of energy stored by the materials during one cycle of oscillation, and estimates the temperature-dependent stiffness behaviour and the load-bearing capacity of the materials \[8\]. Figure 11 shows the variation of storage modulus with temperature of untreated and modified jute composites at 1 Hz frequency. It was noticed that all treated and coated composites S(T3), S(T2), and S(T1) have higher values of storage modulus than untreated jute composites S(T0) in glassy region. This could be due to enhanced stiffness of fibres in the composites after treatment and coating. In the glassy region, increase in values of storage moduli followed the order S(T3) > S(T2) > S(T1) > S(T0). In transition region, the storage modulus of the composites was found to be decreased rapidly as the temperature increased, probably due to the loss in stiffness of the jute fibres at evaluated temperature \[8, 27\]. In the rubbery region, above to 90 °C storage modulus of the composites were saturated because at very high temperature treatment and coating could not control the stiffness of the composites. It was also observed that storage moduli of the composites didn’t show much variation with each other at very high temperature.
Figure 10. SEM images of impact fractures surfaces of untreated and modified jute composites: (a) S(T0), (b) S(T1), (c) S(T2), and (d) S(T3).

Figure 11. Storage modulus v/s temperature graph of untreated and modified jute composites.
3.4.2. Loss modulus

It is very useful to obtained the glass transition temperature and represents the viscous response of the materials \([8, 26, 27]\). The variation of the loss modulus with temperature of untreated and modified jute composites at 1 Hz frequency is shown in figure 12. On increasing the temperature the values of \(E''\) for all the composites were found to be increased up to \(T_g\) and then decreased. Since temperature corresponding to highest peaks of \(E''\) curves shows the values of \(T_g\) of corresponding composites. All the treated and coated composites have the higher values of \(E''\) as compared to untreated composite; peaks of \(E''\) curves follows the order: \(S(T3) > S(T2) > S(T1) > S(T0)\). It could also be observed that \(E''\) curve for the composite \(S(T3)\) has covered greater area as compared to other all the composites due to strong interfacial bonding. The values of highest peak of \(E''\) curves and \(T_g\) of untreated and modified jute composites are provided in table 3. All treated and coated composites exhibited the higher values of \(T_g\) than untreated one. Among all the composites, the highest value of

![Figure 12. Loss modulus v/s temperature graph of untreated and modified jute composites.](image)

![Figure 13. Tan δ v/s temperature graph of untreated and modified jute composites.](image)

| Composites | Loss modulus (MPa) | Tan δ | Loss modulus (MPa) | Tan δ |
|------------|--------------------|-------|--------------------|-------|
| \(S(T0)\)  | 997.97             | 0.606 | 60.12              | 70.64 |
| \(S(T1)\)  | 1025.45            | 0.546 | 61.77              | 67.80 |
| \(S(T2)\)  | 1079.51            | 0.538 | 61.36              | 68.74 |
| \(S(T3)\)  | 1216.00            | 0.417 | 61.19              | 69.31 |

3.4.2. Loss modulus

Table 3. Dynamic mechanical properties of untreated and modified jute composites.
$T_g$ was shown by composite S(T3) due to strong bonding that resist the polymer movement and hence increase in value of $T_g$.

### 3.4.3. Damping parameter

It is the ratio of lost and stored energy during one cycle of oscillation, and depends upon fibre- matrix bonding and stiffness of the fibres [8, 27]. The variation in $\tan \delta$ with temperature of untreated and modified jute composites at 1 Hz frequency is shown in figure 13. Damping shows the impact properties of the materials [8].

Highest value of $\tan \delta$ was found for the untreated composite S(T0); shows the better damping and poor interfacial bonding [8, 27]. On the other hand, lowest value $\tan \delta$ was found for the composite S(T3); shows better load bearing capacity and strong interfacial bonding. Intermediate values of $\tan \delta$ were offered by the composites S(T1) and S(T2); shows the moderate load bearing capacity and damping. The values of $T_g$ obtained from peak of $\tan \delta$ curves of untreated and modified jute composites are mentioned in table 3. The composite S(T0) had the highest value of $T_g$ followed by composite S(T3) as shown in table 3.

### 4. Final remarks

Based on the results obtained from the present study, following conclusions can be drawn.

- A positive effect of treatments (alkali, sodium bicarbonate and benzoyl chloride) and PLA coating on jute fibres were observed in terms of a significant enhancement in mechanical and thermal properties of their composites.
- The superior tensile and flexural properties were seen for the composite reinforced by benzoyl chloride treated and PLA coated jute fibres. However, greatest impact strength was exhibited by the composite reinforced by alkali treated and PLA coated jute fibres.
- The highest values of storage and loss modulus, and glass transition temperature was offered by the composite reinforced by benzoyl chloride treated and PLA coated jute fibres.
- PLA coating on treated fibres could be consider as an effective approach to enhance their composite’s performances for advanced applications.

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