Studies on thermo-mechanical properties of chemically treated jute-polyester composite

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Abstract. The effect of chemical treatments on jute-polyester composites is studied in this paper. The jute fabrics are chemically treated with NaOH and benzoyl chloride and its tensile and visco-elastic properties are compared with untreated jute composite. The NaOH treated jute-polyester composite show superior tensile strength and modulus compared to other jute-polyester composites. The glass transition temperature obtained from DMA shift to higher temperature for composites in comparison to polyester resin, this is due to restriction of mobility in chains due to introduction of jute reinforcement. The DMA results also show favourable results towards NaOH treatment i.e. higher storage modulus and lower tan δ values relative to untreated jute-polyester composite. The benzoyl treated jute-polyester composite however do not show promising results which may be attributed to the fact that the adhesion properties associated with similar ester functional groups in the benzoyl treated jute fabric and polyester resin were not obtained.

1. Introduction

Plant based fibre composites have been very popular in recent years mainly owing to their ease of availability, low cost and environment friendly characteristics. The jute fibres are most commonly used bast fibres and are considered as partial substitute to synthetic fibres. The jute fibre composite applications have been explored in the automobile, construction and consumer goods application. Unsaturated polyester resins (UPR) are most commonly used thermoset resin due to its versatility and low cost. The general purpose UPR is a quick curing based on orthophthalic raw material, suitable for both hand lay up as well as gun spray up. General purpose UPR is used where moderate structural properties are desired.

The jute fibres bear hydroxyl groupings which make these fibres hydrophilic in nature and make them incompatible with the polymer resin used in the composite. A number of chemical treatments like alkali, benzoylation, acetylation, peroxide treatment, permanganate treatment, silane treatment, cyanoethylation [1, 2] of natural fibres are known which decrease the hydrophilic nature and make them compatible with various polymers. Pretreatment also ensures removal of other impurities in the natural fibres.

The mechanical behaviour of various natural fibres with polymer matrix with various chemical treatments/coupling agents have been studied: NaOH treated jute-vinylester composite [3], maleic anhydride grafted Jute-HDPE composites [4], silane treated Ramie/PLA composites [5], micro-emulsion silicon treated Jute- Polyester composite [6] and cyanoethylated jute-polyester composites [7]. Extensive mechanical testing on untreated jute-polyester composites were carried out [8]. The
benzoylation treatment of jute fibres enhances the mechanical properties and has seen much success in Jute-Epoxy and Jute-PLA composites [9, 10]. In case of benzoyl treated jute composites, the interaction of similarities in the ester groupings of the matrix with that of benzoyl treated jute fabric is expected to yield better mechanical properties. In the present work, the modification of natural fibre OH groups using NaOH treatment and benzoyl treatment is carried out and their thermo-mechanical properties are compared with untreated jute composite.

2. Experimental
2.1 Material
Industrial grade jute in the form of unbalanced plain weave 58 × 54 (yarns per 100 mm in warp and weft direction respectively) fabric with an average area density as 250 g/m² and average thickness 0.7 mm was procured from Chandra Prakash & Co., Jaipur, Rajasthan, India. General purpose unsaturated polyester resin NRC Polyflex GR 200-108, catalyst Methyl Ethyl Ketone Peroxide (MEKP) and accelerator Cobalt Octoate was supplied by Naphtha Resins and Chemical (Pvt.) Ltd., Bangalore.

2.2 Alkali treatment
The industrial jute fabric cut to 275 mm (warp direction) × 150 mm (weft direction) dimension was soaked in 5% NaOH solution for a duration of 4 hours at ambient temperature. Later the fabrics were washed using distilled water to get rid of residual NaOH. The fabrics were then finally neutralized using dilute acetic acid. The fabrics were washed with distilled water till the washings showed neutral pH. The fabric was dried for 6 hours at 80°C in an oven. The jute fabric upon NaOH treatment undergoes the following changes [11] as per equation 2.1 and illustrated in figure 1.

\[
\text{Fibre-OH} + \text{NaOH} \rightarrow \text{Fibre-O}^{-}\text{Na}^{+} + \text{H}_2\text{O} + \text{Surface impurities} \quad (2.1)
\]

**Figure 1.** Schematic of NaOH-treated fibre surface.

2.3 Benzoyl chloride treatment
The jute fabrics were initially soaked in 18% NaOH for 1 hour. Later they were subjected to filtration and washing. This is done to get rid of surface impurities on the jute fabrics. Next the jute fabrics are suspended in 10% NaOH which facilitates in activating the hydroxyl groups on the jute fibre surface and after that fibres are dipped in the benzoyl chloride solution for a duration of 15 minutes. The fibres are kept in ethanol for 2 hours to remove excess benzoyl chloride which has not reacted. Finally the jute fabrics are washed with distilled water. The jute fabrics are dried for 6 hours at 80°C in an oven. Upon this treatment the fabric shall undergo the following changes [11] as per equation 2.2 & 2.3 and illustrated in figure 2.

\[
\text{Fibre-OH} + \text{NaOH} \rightarrow \text{Fibre-O}^{-}\text{Na}^{+} + \text{H}_2\text{O} + \text{Surface impurities} \quad (2.2)
\]

\[
\text{Fibre-O}^{-}\text{Na}^{+} + \text{ClC} \rightarrow \text{Fibre-O} - \text{C} + \text{NaCl} \quad (2.3)
\]
2.4 Composite preparation
A suitable quantity of unsaturated polyester resin is taken. First the accelerator cobalt octoate 0.8 wt% is mixed with the resin, then 2 wt% MEKP is mixed with the resin-accelerator mixture (The accelerator to catalyst ratio of 0.4/1 was used). The unsaturated polyester resin upon curing is shown in figure 3.

The jute fabric of size 275 ×150 mm were cut to size and dried in oven to get rid of moisture. The conventional hand lay-up technique was used to make composite plates. Poly Vinyl alcohol (PVA) was applied on the mould surfaces before the start of the process which serves as a releasing agent. First the resin coat is applied then placing the jute fabric and repeating the process till sufficient laminate thickness is achieved. Eventually the laminate is compressed in a hydraulic press at 10 MPa constant pressure for 2 hours followed by exposure to atmosphere for almost 48 hrs which aids in curing.

Four plates were prepared: (1) Polyester plate (2) Untreated jute-polyester composite plate (3) NaOH-treated jute-polyester composite plate and (4) Benzoyl treated jute-polyester composite plate. The fibre weight % for the above composite plates was 25 ±3%.

2.5 Static Tensile test
A computer-controlled servo-hydraulic BISS machine with 100 kN capacity was used to carry out the static tension test. The specimens for the test were prepared as per ASTM D3039 standard. The extensometer was used to get precise readings. Constant displacement rate of 1 mm/min was maintained throughout the test. Tensile strength and modulus of elasticity are obtained from tensile tests. Figure 4 & figure 5 show the tensile specimens and test setup.

2.6 Dynamic Mechanical Analysis.
The dynamic properties of the various specimens are obtained using TA Instruments Q800 Dynamic Mechanical Analyzer (DMA). It is equipped with several optional clamps namely single cantilever, dual cantilever, 3-point bending or 4-point bending to conduct DMA experiments. A single cantilever clamp was used in present study. The dimensions of specimen were 35 mm long, (12.5 ± 0.2) mm width and (3 ± 0.2) mm thick. The test was conducted in air atmosphere from 25 to 150°C with a temperature ramp rate of 2°C/min. The samples were tested at 10 Hz frequency and 30 μm amplitude. Figure 6 & figure 7 show the DMA specimens and test setup. DMA is widely used technique to characterize material properties with respect to temperature, frequency, amplitude, air or nitrogen atmosphere [12]. The temperature dependence of storage modulus which is an indication of elastic behaviour; loss modulus which is an indicator of viscous behaviour and tan δ (Mathematically, tan δ is
the ratio of loss modulus, $E''$ to storage modulus, $E'$. ) which represents the damping of the material is obtained for jute-polyester composites.

![Figure 4. Tensile test specimens.](image1)

![Figure 5. Specimen mounted on UTM](image2)

![Figure 6. DMA specimen.](image3)

![Figure 7. Specimen mounted on DMA.](image4)

3. Results and discussion

Present study deals with the effect of different functionalities present on the fibre surface due to various chemical treatment on the jute-polyester composites. These interactions between the matrix and treated fibre play a vital role in deciding the thermo-mechanical properties of the composites.

3.1 Static Tensile Testing

The stress-strain diagram for polyester, untreated jute-polyester composite, NaOH treated jute-polyester composite and benzoyl treated jute-polyester composite is shown in figure 8. The tensile strength of the composite depends on the jute fibre strength, the matrix strength and the bond strength at the fibre/matrix interface which is dependent on the chemical treatment carried out on jute which influences the transfer of stress across the interface.

The jute-polyester composite exhibit a linear behaviour in the initial portion at low strains, the tensile modulus of the composite is obtained as the slope of this linear region. At higher strains it assumes non-linear behaviour indicating either matrix cracking or local fibre breakages. Upon introduction of the jute fabric in the polyester, the modulus and strength of the composites is enhanced although the strain to failure of the composite is reduced. Mechanical properties for all the specimens are shown in Table 1 and their comparison in figure 9 & figure 10.
Figure 8. Stress-strain behaviour of jute-polyester composites.

Table 1. Mechanical properties of jute-polyester composite

| Sample                                | Tensile modulus of elasticity (GPa) | Tensile strength (MPa) | Strain to failure (%) |
|----------------------------------------|-------------------------------------|------------------------|-----------------------|
| Polyester                              | 1.47                                | 24                     | 1.98                  |
| Untreated jute-polyester composites    | 3.36                                | 30.5                   | 1.4                   |
| NaOH treated jute-polyester composites | 3.53                                | 32.5                   | 1.31                  |
| Benzoyl treated jute-polyester composites | 2.68                              | 26.4                   | 1.56                  |

Figure 9. Tensile strength of jute-polyester composites.
3.2 Dynamic Mechanical Analysis

The important dynamic properties namely storage modulus (E’), loss modulus (E”) and damping parameter (tan δ) are measured with respect to temperature. The glass transition temperature (Tg) of the specimen can be ascertained from either the (i) onset of slope change in Storage modulus or (ii) Loss modulus peak or (iii) tan δ peak. The three curves for polyester resin are shown in figure 11. For the purpose of this study the peak of loss modulus curve is considered for interpretation of Tg. Thus the Tg for polyester resin is 61°C, which is close to the slope change in storage modulus plot i.e. 58°C.

![Figure 10. Tensile modulus of jute-polyester composites.](image)

![Figure 11. Glass transition temperature for polyester.](image)
Figure 12 shows storage modulus plots. At 30°C the storage modulus for polyester is 2223 MPa. The storage modulus shows a decreasing trend as the temperature increases. At 100°C the storage modulus drops to 68.49 MPa, a drop of 97%. Similar behaviour is observed for jute-polyester composites i.e a drop of the order of approximately 100 times to that of at 30°C storage modulus value. It is observed that the storage modulus of NaOH treated jute-polyester shows the highest value of storage modulus of 2613 MPa among other composites, this can be attributed to better fibre-matrix adhesion. The benzoyl treated composite shows higher value than untreated jute composite but the variation of storage modulus over the temperature range (i.e. 30 to 150°C) is not appreciable.

![Figure 12. Plot of storage modulus, E' at 10 Hz frequency.](image)

Figure 13 shows the loss modulus plots for jute-polyester composites. The glass transition temperature as read from loss modulus peak for polyester is observed at 60.94 °C. For untreated jute fibres the Tg shifts to 62.25 °C, while for Benzoyl treated jute-polyester composite the Tg is 66.58 °C while that of alkali treated jute-polyester composite it is 71.44°C. This increase can be attributed to restricted mobility of polyester chain due to the introduction of treated jute fabric. Table 2 shows viscoelastic properties of all tested samples.

| Sample                        | Storage modulus @ 30°C (MPa) | Tan δ height | Loss modulus peak (°C) |
|-------------------------------|------------------------------|--------------|------------------------|
| Polyester                     | 2223                         | 0.599        | 61                     |
| Untreated jute-polyester composites | 2305                         | 0.321        | 62                     |
| NaOH treated jute-polyester composites | 2613                         | 0.386        | 71                     |
| Benzoyl treated jute-polyester composites | 2438                         | 0.385        | 67                     |
Figure 13. Plot of loss modulus, $E''$ at $f=10$ Hz.

Figure 14 for tan $\delta$ plot it is observed that the peak height and width decreases for jute-polyester composites in comparison with the polyester resin. This is owing to the reduction of damping capacity in composites which means relative less area under the tan $\delta$ peaks indicates less ability to dampen impact which is clearly seen for untreated jute-polyester composite.

Figure 14. Plot of tan $\delta$ at $f=10$ Hz.

4. Conclusion
Improved properties in terms of higher tensile strength, modulus of elasticity and dynamic mechanical properties (storage modulus and damping) were observed in case of NaOH treated jute-polyester composites relative to the untreated jute-polyester composite. In case of benzoyl treated jute-polyester composite the tensile strength and modulus were lower than NaOH-treated jute composite, this is due to the fact that the adhesion properties associated with similar ester functional groups in the benzoyl treated jute fabric and polyester resin were not obtained. This is mainly because the ester groups are
not terminal in nature. There is further scope of comparing effects on the properties of composites with respect to the type and positions of functional groups present in the treated jute fibre and polyester matrix.

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