Effect of Various Chemical Treatments on the Damping Property of Jute Fibre Reinforced Composite

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Abstract

In this paper the effect of various chemical treatments on the damping property of jute fiber reinforced green composite is discussed. The surface of the jute fiber was modified by alkali, permanganate, benzoyl chloride, malic anhydride and silane treatments. Several samples of unidirectional composites were prepared with treated jute fibers as well as untreated jute fibers. Matrix used is epoxy and material is made by hot pressing. Surface treatments results in enhancement of bonding between fiber and matrix. Dynamic mechanical analysis (DMA) results show that, surface treated jute fiber reinforced polymer composites have higher storage modulus and lower tan δ with respect to untreated composite.

1. Introduction

Now a day’s Industrial technology is growing at a very rapid rate and consequently there is an increasing demand and need for new materials. So particulate reinforced composites constitute a large portion of these new advanced materials. Composite materials find in a wide range of applications in all cutting-edge ranges of advanced materials such as automotive, renewable energy industries, aerospace and medical devices. The use of renewable materials has gathered much momentum throughout the nineties. One of the major reasons for this renewed growth is an increased awareness for our environment, reflected in phrases such as ‘protection of resources’, and ‘reduction of CO₂ emissions’. The use of plant fibers as insulating or damping materials or as fillers or reinforcement in polymeric materials plays an important role. But low strength and water absorption of natural fiber polymer composites is a serious concern mainly for their potential outdoor applications. Lot of studies have been performed on developing biocomposite materials with natural fibers and polymers
such as starch, polylactide (PLA), polycaprolactone (PCL) and poly (3-hydroxybutyrate-co-3- hydroxyvalerate) (PHBV) [1–4]. The natural fibers such as jute, hemp, flax, bamboo, kenaf, abaca, curaua, coir and cordenka [5–13] were generally used as reinforcements in these studies. Jute fiber is a promising reinforcement for use in composites on account of its low cost, low density, high specific strength and modulus, no health risk, easy availability, renewability and much lower energy requirement for processing. In this work, jute fibers used as reinforcement are subjected to different chemical treatments to make them hydrophobic in nature and to improve the bonding between fiber and matrix, increase adhesion between fiber and matrix. The successful use of natural fibers is dependent on their well-defined structural and mechanical properties. These parameters determine the characteristics of the jute fiber. Due to jute’s low density combined with relatively stiff and strong behavior, the specific properties of jute fiber can compare well with glass fibers. Strength and mechanical properties of few selected fiber materials are shown in Table 1.1.

Table 1.1: Natural and artificial fiber properties.

<table>
<thead>
<tr>
<th>Fibre</th>
<th>Density (g/cm3)</th>
<th>Tensile strength (MPa)</th>
<th>Young’s Modulus (GPa)</th>
<th>Elongation At break (%)</th>
<th>Specific Strength (GPa/g.cm-3)</th>
<th>Specific Young’s Modulus (GPa/g.cm-3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jute</td>
<td>1.3-1.45</td>
<td>393–773</td>
<td>13-26.5</td>
<td>1.16-1.5</td>
<td>0.3–0.5</td>
<td>10–18.3</td>
</tr>
<tr>
<td>Flax</td>
<td>1.5</td>
<td>345–1100</td>
<td>27.6</td>
<td>2.7-3.2</td>
<td>0.2–0.7</td>
<td>18.4</td>
</tr>
<tr>
<td>Hemp</td>
<td>1.45</td>
<td>690</td>
<td>9.4–22.0</td>
<td>3 - 7</td>
<td>0.3–0.4</td>
<td>26.3 - 52.6</td>
</tr>
<tr>
<td>Sisal</td>
<td>1.5–1.6</td>
<td>400–938</td>
<td>5.5–12.6</td>
<td>7.0–8.0</td>
<td>0.2–0.5</td>
<td>6.4–15.2</td>
</tr>
<tr>
<td>Cotton</td>
<td>2.5</td>
<td>287–800</td>
<td>70</td>
<td>2.5</td>
<td>0.8–1.4</td>
<td>28</td>
</tr>
<tr>
<td>E-glass</td>
<td>2.5</td>
<td>2000 - 3500</td>
<td>86</td>
<td>2.8</td>
<td>1.8</td>
<td>34.4</td>
</tr>
<tr>
<td>S-glass</td>
<td></td>
<td>4570</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The mechanical properties of natural fiber-reinforced composite depend extremely on the degree of adhesion between the natural fiber and the matrix [14]. There by, poor fiber/matrix interface leads to a weaker material with low strength and life span. In the last two decades, many researchers have focused on improving the interfacial adhesion by modifying the fiber surfaces via physical and chemical treatments to make them more compatible with matrix [15]. These surface treatments also enhance environmental durability (moisture and temperature) and wear resistance of the composite.

Numerous surface treatments methods have been reported in literature such as electric discharge, alkalization, acetylation, peroxide treatment, permanganate treatment, benzylation, silane treatment and cyanoethylation. Ray Dipa et al.[16] investigated the effect of untreated and alkali treated jute fiber and vinylester-resin composite on dynamic mechanical behavior. They reported that storage modulus
increases with the increase in fiber loading and treatment. On the other hand tanδ value decreases. Mohanty S et al.[17] investigated the effect of MAPE treated jute fiber and jute loading on flexural, tan δ, impact and tensile strength of jute HDPE composite. They found that the tanδ decreased in comparison to virgin HDPE and untreated composite. Flexural, impact and tensile strength increased with 30% fiber loading and thermal property increased due to treatment. Vilaseca F et al.[18] investigated the effect of fiber content and alkali treatment on tensile, flexural, and impact strength of starch polymer jute fiber composite. They found that both tensile and flexural properties increased with increase in fiber loading of treated fiber, but impact strength decreased with same fiber loading. Jawaid Met al.[19] investigated the effect of woven hybrid composites on tensile and flexural properties of epoxy woven jute fiber composite. They found that flexural and tensile properties of hybrid composite were higher but less than woven jute composite. Thiruchitrambalam M et al.[20] investigated the influence of pre-treatments (alkali treatment, benzoxylation, permanganate treatment) on tensile, flexural, and impact strength and water absorption of leaf stalk polyester resin composite. They found that tensile, flexural, and impact strength increased due to fiber treatment and water absorption tendency decreased due to treatment. Sever K Sarikanat et al.[21] investigated the effect of surface treatment of jute fabric polymer composite on tensile and flexural strength. They found that the tensile strength and flexural strength increased due to fiber treatment. Goriparthi B K et al.[22] investigated the effect of fiber surface treatments on tan δ, glass transition temperature (Tg), flexural, impact and tensile strength of polylactide jute fiber composite. They found that the Tg, flexural, impact and tensile strength increased due to treatment and tan δ decreased due to treatment. Hossain M K et al.[23] investigated the effect of surface modified jute fiber with addition of nanoclay on dynamic mechanical properties and tensile property of Poly (3-hydroxybutyrate-co-3-hydroxyvalerate-12%)-Biopolymer granule (Biopol) and Jute fiber + nanoclay composite. They found that the dynamic mechanical properties and tensile strength was improved with the addition of nanoclay and treated jute fiber. Rahman M M et al.[24] investigated the effect of surface modification of jute fiber and fiber loading on tensile, flexural, impact strength and water absorption property of polypropylene jute fiber composite. They found that the tensile strength of the composites decreased with an increase in the jute fiber loading, the Young’s modulus, flexural strength, flexural modulus, impact strength and hardness of the composites increased with an increase in the fiber loading. Post-treatment improved the hydrophilic nature of the jute fiber and the interfacial bonding and wettability between the fiber and matrix. D. Ray et al.[26] investigated the effect of alkali-treatment on flexural strength of jute fibers and vinyl ester resin composite and they found that the flexural strength increases due to treatment.

After extensive study of various research papers, it was observed that the effect of jute fiber as reinforcement were investigated majorly for the mechanical properties of polymer composite; such as tensile, flexural, impact, compression properties etc. The research work to identify the effect on damping properties if jute fiber used as
reinforcement in polymer composite is very few. No comparative analysis was found in research papers which show the effect of different types of chemical treatment of jute fibers on the damping properties of the composite. Accordingly the objectives of the present work are to study the effect of different chemical treatments on dynamic properties of jute fiber reinforced composite.

2. Materials and Methods

2.1. Materials

In this study Araldite GY-257 (Bisphenol-A, mono-functional reactive diluents) epoxy resin was used as structural matrix. It was selected as the matrix material due to its low viscosity; which lies in range of 500-650 MPa-s with a density of 1.150 g/cm³. The flash point of GY-257 epoxy is greater than 138°C. Aradur HY-951, a low viscosity aliphatic amine, was used as a compatible hardener for polymerizing the epoxy resin. Its viscosity and density is 10-20 MPa-s and 0.980 g/cm³ respectively at room temperature. Tossa jute fiber supplied by jute board Kolkata was selected as the reinforcement. It is a low cost fiber containing cellulose (61-71.5%), hemicellulose (13.6-20.4%), pectin (0.2%), lignin (12-13%), moisture content (12.6%) and wax 0.5% by weight. Sodium hydroxide, potassium permanganate, benzoyl chloride, maleic anhydride, silane coupling agents (3-methacryloxy-propyl-trimethoxy-silane), acetone and ethanol was used for chemical treatment of fiber.

2.2. Fiber surface treatments

Jute fibers were subjected to the following surface treatments, in order to improve interfacial adhesion between jute fiber and epoxy matrix.

In alkalization, fibers were cut to 45 cm of length and were soaked in a 5% NaOH solution at room temperature maintaining a liquor ratio of 15:1 for 4 hr. The fibers were then washed several times with fresh water to remove any NaOH sticking to the fiber surface. Fibers were neutralized with dilute acetic acid and finally washed again with distilled water. Final pH maintained was 7 and then dried for 6 h followed by oven drying at 80°C.

In permanganate treatment, alkaline treated fibers were dipped in 0.125% permanganate acetone solution for 1 min with liquor ratio 15:1. Fibers were dried at 80°C for 5 h to remove excessive solvent and moisture in hot air oven.

In Benzoylation, the fiber was initially treated by alkaline in order to activate the hydroxyl groups of the cellulose and lignin in the fiber; then the fiber was suspended for 15 min in benzoyl chloride solution. The isolated fibers were then soaked in ethanol for 1 h to remove the benzoyl chloride and finally, the fibers were washed with distilled water several times and dried in an oven maintained at 80°C.

In silane treatment, 3-methacryloxy-propyl-trimethoxy-silane was used to modify the jute fiber surface. Jute fibers were soaked in a solution of acetone/water (50/50 by volume) with a 1% silane concentration for 2 h and then cleaned with acetone and dried in the hot air oven at 80°C for 5 h.
In maleic anhydride treatment, jute fibers were surface treated for 5 min with 1% solution of maleic anhydride dissolved in toluene solvent at 55°C. After treatment, fibers were washed in toluene to remove extra MA. The fibers were then dried for 6 h followed by oven drying at 60°C.

2.3. Composite fabrication
In order to remove the absorbed moisture and thereby to prevent the void formation, jute fiber and epoxy matrix were dried in the vacuum at 80 °C for 6 hr before specimen fabrication. Polymer composite sample sheets with jute fiber concentration of 30 wt% were manufactured by using “compression molding process”. Resin and hardener were mixed together in the ratio of 10:1 by weight. The mixture was then stirred very slowly to forefend any bubbles formation chances for a prolonged duration. Then jute fiber, 30% by weight was spread in the mould cavity. Mixture of hardener and resin was placed inside the mould impression in which fiber was already present in the filament form and the other half of mould plate was placed over it to complete the assembly. Thereafter, the mould assembly was placed in the hot press table and hydraulic actuator lever was activated to raise the hot press table and apply the force on the mould plate assembly. The force of 1.5 ton per square inch was applied on the mould assembly. This force squeezes the resin-fiber mixture, due to which this mixture takes the shape of mould impression. The mould was kept at the same pressure for 10 hours at 90°C for complete cross linking of monomers of epoxy resin to form a stable composite sheet. Then the mould was allowed to cool gradually for 8-9 hours at room temperature so that no hardening effect takes place on the composite sheet due to sudden cooling. After the whole process, mould was opened and sample was taken out in the form of consolidated composite sheet.

2.4. Dynamic mechanical analysis
Storage modulus, loss modulus and loss factor (tanδ) of the composite specimens (50 mm x 6mm x 2 mm) according to ASTM-D790 were measured under the frequency of 1 Hz with strain rate of 5 micron and ramp rate of 5 °C/min. Three point bending mode was selected for analyzing the damping capability of jute reinforced composites. For the DMA analysis, the following types of samples were used for study:
3. Results and Discussions

3.1 Effect of Temperature on Damping Properties of Polymer Sheet

A general DMA plot for pure epoxy sample showing the relation between the storage modulus and tan δ with respect to varying temperature is illustrated in Figure 1.1. Storage modulus (E’) is closely related to the load bearing capacity of a material and is analogous to the flexural modulus (E). The tan δ indicates the vibration absorbing capacity (i.e. damping capacity) of the material. Mathematically, tan δ is represented by the ratio of loss modulus, E’’ to storage modulus,

\[
\tan \delta = \frac{E''}{E'}
\]

It can be seen from Figure 1.1 that with the increase in temperature, storage modulus reduces. There is a marked decrease in storage modulus from 3.12 GPa at 28 °C to 0.301 GPa at 60 °C, i.e. 90.4% decrease in storage modulus. This decrease is attributed to the softening of the polymer due to the increase in the chain mobility of the polymer matrix at high temperatures. This means that the segmental mobility of polymer chains increases with temperature rise and at glass transition temperature, the state changes from glassy to rubbery state. Therefore the load bearing capacity is drastically reduced at elevated temperature.

From Figure 1.1, it can be also seen that, on contrary to storage modulus, the tan δ value increases with the rise in temperature. The tan δ values rises from 0.063 at 28 °C to 0.49 at 65 °C, but beyond this temperature tan δ starts decaying. This increase is attributed to the increase in viscoelastic property of the polymer material at higher temperature. The contribution of viscous part to the dynamic response is relatively high in higher temperature region.

This response is high up to a specific temperature, which is called as glass transition temperature (T_g). Beyond T_g, the tan δ value decreases, and the decay in modulus levels off, indicating that a more elastic region is reached, the so called rubber plateau. In this rubber elastic region, the polymer chains have full mobility and properties are determined by the entangled network. This rubber elastic region is found till the minimum of tan δ (Harold G H and Melick V [25]). Upon further heating the polymer chain starts to disentangle and the dynamic modulus decays further out of the measurable range and the material become the liquid like.

The above discussion explains how storage modulus and tan δ varies with respect to the temperature. Now when jute fiber is incorporated as reinforcement in the polymer composite, there is certain variation in composite properties. The variation in storage modulus is shown in the Figure 1.2 and variation in tan δ shown in Figure 1.3.

It is clearly seen from the Figure 1.2 that storage modulus increases due to addition of jute fiber in epoxy (from 3.13 GPa to 4.33 GPa) at room temperature. The tan δ value decreases from 0.53 for unreinforced epoxy sample to 0.193 for jute fiber reinforced composite. This is due to wetting behavior of fiber with resin as a result of which storage modulus is increased and tan δ (loss factor) decreases (Ray D et al.[26]).
3.2 Effect of Chemical Treatment of Jute Fiber on Damping Properties of Composites

Figure 1.4 shows the vibration in $E$ (storage modulus) with respect to temperature for untreated jute fiber and jute fiber subjected to different chemical treatments. From figure 4.4 it is observed that storage modulus of chemically treated jute fiber reinforced composite are higher than that of untreated jute fiber composites. At room temperature from table 1.1 it is seen that the chemically treated fiber reinforced composite have higher storage modulus then that of untreated jute fiber reinforced composite.

![Fig. 1.1: Storage modulus and tan δ variation with epoxy.](image1)

![Fig. 1.2: Variations in storage modulus due to jute fiber reinforcement in.](image2)
Malic anhydride treated jute fiber/epoxy composite has highest storage modulus (13.2 GPa), when compared to rest of the composites sample. The highest storage modulus of malic anhydride treated composites may be attributed the strong fiber matrix interface between the fiber and matrix, which leads to efficient stress transfer from the matrix to fiber and enhancement in storage modulus. Silane treated and benzoyl chloride treated fibre reinforced composite also gives higher values of storage moduli (10.11 GPa, 10.95 GPa) in comparison to untreated fiber composite (4.33 GPa). respect to temperature of pure epoxy sample.

Therefore, with the obtained DMA test result, it will be appropriate to say that the addition of chemically treated fiber increases the storage modulus which indicates that the load bearing capacity of jute reinforced composite is increased. Therefore, it can be said that, treated jute fiber reinforcement helps in increasing the storage modulus of polymer composite. The chemical treatment improves the wetting behavior of fibers with resin as result of which storage modulus increases.

**Fig. 1.3**: Variations in tan δ value due to jute fibre.

**Fig. 1.4**: Effect of chemical treatment on storage modulus.reinforcement in epoxy.
Table 1.1: Effect of chemical treatment on storage modulus.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Temp. (°C)</th>
<th>Storagemodulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UJF composite</td>
<td>28</td>
<td>4.33</td>
</tr>
<tr>
<td>AJF composite</td>
<td>28</td>
<td>5.3</td>
</tr>
<tr>
<td>PJF composite</td>
<td>28</td>
<td>7.63</td>
</tr>
<tr>
<td>BJF composite</td>
<td>28</td>
<td>10.11</td>
</tr>
<tr>
<td>SJF composite</td>
<td>28</td>
<td>10.95</td>
</tr>
<tr>
<td>MJF composite</td>
<td>28</td>
<td>13.2</td>
</tr>
</tbody>
</table>

Fig. 1.5 shows the variation in loss factor with respect to temperature for untreated jute fiber as well as jute subjected to various chemical treatments. The untreated jute fiber shows loss modulus peak at 65.7°C, which is attributed to the increase mobility of resin molecular. This peak also referred as the glass transition temperature (Tg). The surface modification of fiber due to various chemical treatment result in shifting of the peak to higher temperature (65.7, 66, 69.7, 73.2, 79.9, 83.7°C for UJF, MJF, AJF, PJF, BJF, SJF composite). This could be due to the immobility of polymer molecules near the surface jute fibers due to various molecular interactions, which increases the Tg of the composite (Ray et al. [16]).

Fig. 1.5: Effect of chemical treatment on loss modulus.

Variation of the loss factor (tan δ) of jute fiber/epoxy composites with respect to temperature are compared in Fig. 1.6. Untreated composites exhibit a higher tan δ peak value as compared to treated composites. Higher tan δ peak value for untreated composite may be attributed to more energy dissipation by internal friction at the weaker interface between untreated fiber and matrix. Among the treated composites, silane treated composites showed a lower tan δ peak values. Since the tan δ peak value is related to fiber matrix adhesion, lower tan δ peak value of silane treated composite
corresponds to better adhesion and compatibility between silane treated jute fibers and resin than that of the other treatments. Benzoyl chloride treated fibre reinforced composite also exhibits similar tan δ value as compared to silane treated composite.

Fig. 1.6: Effect of chemical treatment on tan δ.

Table 1.2: Effect of chemical treatment on tan δ.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Loss Factor (tan δ)</th>
<th>Percent change in tan δ</th>
</tr>
</thead>
<tbody>
<tr>
<td>UJF Composite</td>
<td>0.193</td>
<td>-</td>
</tr>
<tr>
<td>AJF Composite</td>
<td>0.142</td>
<td>26.4%</td>
</tr>
<tr>
<td>PJF Composite</td>
<td>0.112</td>
<td>41.9%</td>
</tr>
<tr>
<td>BJF Composite</td>
<td>0.086</td>
<td>55.4%</td>
</tr>
<tr>
<td>SJF Composite</td>
<td>0.080</td>
<td>58.5%</td>
</tr>
<tr>
<td>MJF Composite</td>
<td>0.135</td>
<td>28.5%</td>
</tr>
</tbody>
</table>

In case of malic anhydride treated fiber the storage modulus is highest but on the contrary it also has the higher value of tan δ. This is because of the reason that malic anhydride treatment increases the bonding between fiber and matrix as a result of which storage modulus increases but on the other side it weakens the fiber that increases the loss modulus under dynamic loading. Fig. 1.6 and Table 1.2 shows the variation in tan δ with respect to temperature for untreated jute fibre as well as jute subjected to various chemical treatments. The untreated composite exhibits a higher tan δ peak value (0.193) as compared to the treated composites. Higher tan δ peak value for untreated composite may be attributed to more energy dissipation by internal friction at the weak interface between the matrix and untreated fibre. Among treated
composite, silane treated shows the lowest tan \( \delta \) peak value (0.08). Since the tan \( \delta \) peak value is related to fibre matrix adhesion, low tan \( \delta \) peak value of silane treated composite correspond to better adhesion and compatibility between silane treated jute fibre and epoxy resin matrix than that of other treatments.

4. Conclusions

The influences of chemical pre-treatments of the jute fibers on the damping properties of the jute/epoxy polyester composites have been experimentally evaluated and the following conclusions are drawn. The performance of the jute/epoxy composites is very much influenced by the surface properties of the fibers. Alkali, permanganate pre-treatments, benzoyl chloride, malic anhydride and silane have improved the mechanical properties, i.e. damping properties of the composites in comparison to composites reinforced with untreated fibers due to improved interfacial bonding. The DMA test result shows that silanized and benzoyl chloride treated jute fiber reinforced in polymer composite improves storage modulus and the thermal stability in comparison to untreated fiber reinforced composite. This is attributed to the improved interfacial bonding due to silanization which resulted in the reduction of frictional damping aspects. Silanized and benzoyl chloride treated jute fiber reinforced composite shows better strength when compared to untreated jute fiber reinforced composite at same concentrations.

References


