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Research Article



Influence of Concentrations of Alkali Treatment on Mechanical and Dynamic Mechanical Properties of Hemp/Polyester Composite

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Abstract

In present work, hemp fibres were subjected to alkali treatments at various concentrations (5, 10 & 15 wt.%) to prepare its polyester based composites. Hemp fibre reinforced polyester composites were prepared by hand lay-up technique followed by static compression keeping constant 25 wt.% of fibres content in order to study influence of alkali treatment concentrations on its mechanical and dynamic mechanical properties. Mechanical properties of prepared composites were studied in terms of tensile strength and modulus, and flexural strength and modulus whereas dynamic mechanical properties were studied in terms of storage modulus (E'), damping ($Tan \delta$), glass transition temperature (T_g) and effectiveness constant of reinforcement (\in). The results indicated that mechanical and dynamic mechanical properties were found to increase due to alkali treatment up to 5% concentration and then decrease.

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1. Introduction

In recent past decades, natural fibres have been a suitable replacement of synthetic fibres for polymer based composites because of its extraordinary advantages such as low density, recyclability, high specific strength and modulus and less wear and tear [1-3]. These fibres also offer the benefits of low cost associated with processing as compared synthetic fibres such as glass, carbon, aramid and nylon [4-5]. In addition, these fibres have some other dominating properties over the synthetic fibres like huge availability and biodegradability [6-8]. However, these fibres are troubled by some disadvantages also i.e. poor resistance to water absorption, low impact strength and poor compatibility [9-10]. Another main disadvantage of composite of these fibres is poor mechanical performance over the synthetic fibres reinforced polymer composite. Hence, it can be concluded that composite of natural fibres cannot be better option for structural applications. The disadvantages of these fibres can be overcome by surface modifications using various types of chemical treatments [11-13].

Many researchers had used various types of chemical treatments to improve the interfacial adhesion between natural fibres and matrix in order to improve mechanical properties. Shanmugam and Thiruchitrambalam [14] studied the effect of alkali treatment on mechanical and dynamic mechanical properties of palmyra palm leaf stalk/jute fibre reinforced polyester hybrid composite and obtained improved mechanical and dynamic mechanical properties. Gupta and Srivastava [15] investigated the tribological and dynamic mechanical properties of hybrid jute/sisal epoxy composite and reported the positive effect of alkali treatments. Tensile and flexural properties of roystonea regia fibre reinforced epoxy composite was found to improved due to alkali treatments [16] and interface between fibres and polymer matrix was improved by incorporating modified coir fibres as resulted increase in mechanical properties [17]. EI-Abbassi et al. [18] obtained conclusions stated that the mechanical properties of Alfa fibre reinforced polypropylene composite was found to increase after alkali treatments.

Different natural fibres such as jute, sisal, banana, hemp, coir, palm pine apple etc had already attracted the attention of researchers due to its excellent properties and being used in various applications such as constructions, packaging and automobiles. Hemp is a widely used natural fibre had high strength and stiffness which make him useful reinforcing materials to be used in composite materials. Currently, there has been an increasing improvement in use of this fibre for various engineering applications. According to FAO, the production of hemp fibre has continuously increased and its production is 0.5% of total production of natural fibre in the world.

The potential of hemp fibre and a significant effect of alkali treatment on properties of various natural fibres reinforced polymer composites have motivated to investigate the effect of alkali treatment concentration on mechanical and dynamic mechanical properties of hemp fibre reinforced polyester composite. In present investigation, effect of concentration of alkali treatments on mechanical and dynamic mechanical properties of prepared hemp fibre reinforced polyester composite is studied.

2. Materials and methods

2.1. Materials

Hemp fibres were purchased from Uttarakhand Bamboo and Fibre Development Board, Dehradun, India. Unsaturated polyester resin with ketone peroxide as catalyst and cobalt naphthalate as accelerator were purchased from Local resource. The physical and mechanical properties, and chemical composition of hemp fibre is given in Table 1 [19].

Table 1 Physical and mechanical properties, and chemical composition of hemp fibre

Properties	Values
Density (g/m ³)	1.4
Diameter (µm)	17-23
Aspect ratio	549
Tensile strength (MPa)	210-510
Young's modulus (GPa)	20-41
Cellulose (%)	67
Lignin (%)	3.3
Microfibrillar angle	6.2^{0}
Pectin (%)	0.8
Hemi- cellulose (%)	14-20

2.2. Alkali treatment

Alkaline treatment or mercerization is a widely used chemical treatment of natural fibres. It modified the disturbance of hydrogen bonds in the network structure cause increase in surface roughness as resulted improved adhesion between fibres and matrix. Reactions of NaOH with natural fibre are given as follows:

$$Fibre - OH + NaOH \longrightarrow Fibre - O - Na + H_2O$$
(1)

Alkali treatments were carried out using NaOH concentrations of 5, 10 and 15 wt.%. The fibres were immersed in various NaOH concentrations for 30 minutes as recommended [14]. The fibres were then cleaned several times with distilled water followed by immersion of fibres in very dilute HCl in order to remove the NaOH adhering to the surface of the fibres. Finally the fibres were again washed several times with distilled water and then dried in an oven maintained at 70 °C for 24 hours.

2.3. Fabrication method

Alkali treated hemp fibres were reinforced into unsaturated polyester resin to make the composites. The hemp/polyester composites were fabricated by hand lay-up technique followed by static compression subjected to study the effect of varying alkali treatment concentrations, keeping constant 25 wt.% of total fibres content in each composite. Polyester resin was mixed with 2 wt.% catalyst and accelerator to prepare the matrix. The mixture was stirred thoroughly to ensure consistent mixing. A stainless steel mould having dimensions of 300 mm \times 200 mm \times 3 mm was used to make the 3 mm thickness of laminate. Silicon spray was used to facilitate easy removal of the composite from the mould after curing. The cast of each composite was cured under a load of 50 kg for 24 hours before it was removed from the mould. Specimens were cut in proper dimensions as per ASTM standard using a diamond cutter subjected to analysis of mechanical and dynamic mechanical properties. The composites manufactured with varying alkali treatment concentrations are designated as shown in Table 2.

Notations	Composites
HC	Hemp fibre reinforced polyester composite
TC5	5% NaOH treated hemp fibre reinforced polyester composite
TC10	10% NaOH treated hemp fibre reinforced polyester composite
TC15	15% NaOH treated hemp fibre reinforced polyester composite

 Table 2 Designations of hemp/polyester composites

3. Characterization

3.1. Tensile test

Tensile test of prepared hemp/polyester composite samples were performed on Tinius Olsen H 10 K-L (Bi-axial testing machine) with a crosshead speed of 2 mm/min. Tests were conducted as per ASTM D638 with dimension of 165 mm \times 20 mm \times 3 mm. Five specimens of each composite were tested and their average values and standard deviation are reported.

3.2. 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 the test of dimensions 80 mm \times 12.7 mm \times 3 mm with a recommended span to depth ratio of 16:1 as per ASTM D790. The flexural test was carried out at room temperature with the crosshead speed of 2 mm/min. Calculation of flexural strength and flexural modulus are done using following equation.

Flexural strength =
$$\frac{3FL}{2bd^2}$$
 and Flexural modulus = $\frac{mL^3}{4bd^3}$ (2)

where F is ultimate failure load (N), L is span length (mm), b and d are width and thickness of specimen in (mm) respectively and m is slope of the tangent to initial line portion of the load-displacement curve. Five specimens of each composite were tested and their average values and standard deviation are reported.

3.3. Dynamic mechanical analysis

The dynamic mechanical properties of polyester and hemp/polyester 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. The laminates were cut into samples having dimensions of 50 mm \times 13 mm \times 3 mm as per ASTM D 5023. Experiments were carried out at 1Hz frequency and within temperature range of 30 –200 °C.

4. Results and discussion

4.1 Tensile properties

The tensile properties of prepared hemp/polyester composites in terms of tensile strength and tensile modulus are given in Table 3 and corresponding data are plotted in Figure 1. A positive effect of alkali treatment is observed as increase in tensile properties of untreated hemp/polyester composite. Alkali treated hemp/polyester composite TC5 has higher values of tensile strength (74.01 MPa) and tensile modulus (2.205 GPa) which is 26 % and 16% more than those of untreated hemp/polyester composite HC. This observation, effect of alkali treatment, can be explained as alkali treatment increases the surface roughness of fibres resulting in better mechanical interlocking and hence increases in mechanical properties [20]. It is also observed that on increasing the concentration of alkali treatment tensile properties are found to decrease as compared to TC5. Alkali treated hemp/polyester composite TC10 has lower tensile properties than TC5 due to further increase in roughness of fibre cause poor adhesion between fibres and matrix and hence decrease in tensile properties. Tensile properties are further found to decrease for composite TC15 as compared to TC5 because of damages of fibre's surface on increasing the concentration of alkali treatment for the fibre's surface on increasing the concentration of alkali treatment for the fibre's surface on increase for composite TC15 as compared to TC5 because of damages of fibre's surface on increasing the concentration of alkali treatment over 10 wt.%.

Composites	Tensile strength	Tensile modulus	Flexural strength	Flexural modulus
	(MPa)	(GPa)	(MPa)	(GPa)
Polyester	35.16± 2.19	1.712±0.10	45.73±4.49	6.781±0.50
HC	58.93±3.18	1.905 ± 0.12	65.99±4.99	8.84 ± 0.62
TC5	$74.01{\pm}5.40$	2.205±0.16	76.65±5.19	10.74±0.73
TC10	61.50 ± 4.59	2.120±0.15	72.75±6.25	9.44±0.61
TC15	59.96 ± 4.94	$1.980.\pm0.18$	68.39±5.11	8.87±0.56

Table 3 Tensile and flexural properties of polyester and hemp/polyester composite



Figure 1 Variation in tensile strength and tensile modulus of hemp/polyester composite with variation in alkali concentrations

4.2. Flexural properties

The flexural properties of prepared hemp/polyester composites in terms of flexural strength and flexural modulus are given in Table 3 and corresponding data are plotted in Figure 2. Flexural properties show similar trends as that of tensile properties. Alkali treated hemp/polyester composite TC5 has higher values of flexural strength (76.65MPa) and flexural modulus (10.74 GPa) which is 16 % and 21% more than those of untreated hemp/polyester composite. On increasing the concentration of alkali treatment over 5wt.% flexural properties are also found to decrease. This fact can be explained as treatment of fibres with higher concentration of NaOH makes the fibres more brittle and stiffer as results decease in flexural properties. The increase in flexural strength and flexural modulus follows the order : TC5 > TC10 > TC15 > HC > polyester.



Figure 2 Variation in flexural strength and flexural modulus of hemp/polyester composite with variation in alkali concentrations



Figure 3 Variation in storage modulus with temperature for polyester and hemp/polyester composites

4.3. Storage modulus

The variation in storage modulus of polyester, and untreated and treated hemp/composites as a function of temperature at 1 Hz frequency is shown in Fig. 3. Glassy, transition and rubbery regions are the mainly three significant region of storage modulus versus temperature curve. In the glassy region, it is observed that alkali treated hemp/polyester composite TC5 has higher value of E' than those of other composites due to better interfacial adhesion between fibres and matrix which is further cause of uniform transfer of stress. In glassy region, the values of E' for polyester and hemp/polyester composite follow the order: TC5 > TC10 > TC15 > HC > polyester. In transition region, it is observed that all composites have a gradual fall in value of E' with increase in temperature whereas polyester resin has a sudden fall. It is due to increase in molecular mobility as temperature reached above to T_g [21]. In rubbery region, alkali treated hemp/polyester composite TC5 has higher value of E' whereas its lower value is shown by polyester. Polyester resin has the lowest value of E' due to increase in molecular mobility at higher temperature [22]. The values of \in is calculated using following equation [22]:

$$\in = \frac{\left(\frac{E'_{g}}{E'_{r}}\right)_{Composite}}{\left(\frac{E'_{g}}{E'_{r}}\right)_{Epoxy}}$$
(3)

where E'_g and E'_r are the storage modulus in glassy region and rubbery region respectively. The higher value of \in shows the lower efficiency of the reinforcement and its lower value show the higher efficiency of reinforcement. The calculated values of \in for hemp/polyester composites are given in Table 4. The composite TC5 has the lowest value (0.232) of \in whereas the composite TC15 has its highest value (0.452). The lowest value of \in for composite TC5 shows the high efficiency of reinforcement than other all composites.

Composites	E	Peak height of $Tan\delta$ curve	T_{g} from $Tan\delta$ curve $\begin{pmatrix} 0 \\ C \end{pmatrix}$
Polyester	-	0.350	73.20
HC	0.346	0.313	72.35
TC5	0.232	0.231	81.54
TC10	0.302	0.246	65.00
TC15	0.452	0.321	73.90

Table 4 Values of \in and T_g for polyester and hemp/polyester composites

4.4. Damping

During one cycle of oscillation, the ratio of loss modulus and storage modulus is damping or $Tan\delta$. The variation in $Tan\delta$ for polyester, and untreated and treated hemp/polyester composites as a function of temperature at 1 Hz frequency is shown in Fig. 4. The peak values of $Tan\delta$ curve for polyester and hemp/polyester composites are given in Table 4. The peak of $Tan\delta$ curve follows the order: Polyester > TC15 > HC > TC10 > TC5 as shown in Table 4. Polyester resin has the highest value of $Tan\delta$ curve as expected due to very poor restriction of molecular motion at higher temperature. The higher value of $Tan\delta$ for polyester shows better damping than all composites. The lowest value of $Tan \delta$ is found for composite TC5 due to strong interface between fibres and matrix which reflects good load bearing capacity. The value of T_g obtained from peak of $Tan\delta$ curve are given in Table 4. Shifting of T_g towards higher temperature is found for composite TC5 which shows the better thermal stability than polyester and other composites.



Figure 4 Variation in Tan δ with temperature for polyester and hemp/polyester composites

5. Conclusions

Tensile, flexural and dynamic mechanical properties of polyester, and untreated and treated hemp/composites are studied and following conclusions are drawn.

- Alkali treatment has significantly affected the tensile, flexural and dynamic mechanical properties of hemp/ polyester composite.
- Mechanical and dynamic mechanical properties are found to increase at 5 % NaOH treatment but decease on further increase in concentration of NaOH.
- Alkali treated hemp/ polyester composite TC5 has the higher values of tensile and flexural properties, storage modulus, and glass transition temperature as compared to those of polyester and other composites.
- The lower value of $Tan\delta$ and ϵ is shown by alkali treated hemp/polyester composite TC5 which shows good load bearing capacity and higher efficiency of reinforcement.

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