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Effect of Treatments on Thermo-mechanical Properties of Epoxy based Sisal Biocomposites

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ABSTRACT: Researchers around the world are working for the development of natural fibre reinforced composites because natural fibres are not harmful to the environment. But, the poor bonding between the polar natural fibres and non-polar polymer matrix is the major challenge in the development of their composites. The aim of the present work is to improve the interfacila bonding between sisal fibres and epoxy resin using alkali treatment, and carbon nanotube (CNT) treatment and blending. The effect of these treatments on flexural and dynamic mechanical properties of sisal/epoxy composites has been studied. All the treated and untretaed samples were fabricated using a hand lay-up method. Both the flexural (bend) test and dynamic mechanical analysis (DMA) tests were executed in a point load simply supported mode. For the DMA tests, the variable temperature in the range 30°C to 120°C was used while the frequency was kept constant at 1 Hz. The significant improvement in the bonding between sisal fibres and epoxy resin was observed after treatments. There was 27.49%, 36.22%, and 74.45 % improvements in flexural strength for alkali-treated, CNT treated, and CNT blended composites respectively over untreated composite. At the low temperature, CNT treatment of sisal-fibres was more effective than chemical treatment, while at higher temperatures, chemical treatment was better than CNT treatment. The composites prepared in the present work by treatment of fibres can be proposed to be used for applications such as wind turbine blades, where high bending strength with sufficient thermal resistance is required.

Keywords: Carbon Nano Tube, Sisal Fibre, Chemical Treatment, Flexural strength, Dynamic Mechanical Properties.

I. INTRODUCTION

Metals, Ceramics, Polymers, and Composites are the various classes of materials [1]. Metals are suitable for low-temperature applications, at high-temperature metals lose their strength [2-4]. Polymer materials lose their strength at even lower temperatures [5]. Ceramic materials are good at high-temperature applications but are very brittle in nature [6]. Brittleness restricts their use in structural applications. Further, a broad spectrum of demands is needed for present industrial applications, which cannot be achieved by these traditional materials. Therefore, researchers are working on the development of the composites materials which can satisfy these demands. A composite is an amalgamation of two or more insoluble micro constituent materials (i.e., matrix and reinforcement) having different form or composition [7]. Matrix material can be a metal, ceramic, or polymer. Reinforcement material can be natural fibres, synthetic fibres or particulates.

Synthetic fibre and glass fibre reinforced polymer composites have been very popular and are being used in automobile and aerospace industries. These composites are not environmentally friendly, which leads to the development of the material with no environmental impact. Such material may be developed by using natural fibres as reinforcement because of the various advantages offered (i.e., low asking price, high specific strength, low density, environmentally friendly, eco-friendliness, and abundant availability) offered by natural fibres [8-13]. Instead, there are some disadvantages also with natural-fibre composites such as low strength, poor thermal stability, poor surface finish, low moisture resistance, and poor compatibility with the polymer matrix [14-15]. The natural-fibres have polar groups due to which these fibres form poor bonding with the non-polar epoxy matrix. The compatibility of polar natural-fibres with non-polar polymer matrixes can be increased by the treatment of natural-fibres to change their polar nature [16]. There are various fibre treatment techniques such as chemical treatment, physical treatment, thermal treatment, and nano-powders treatment to improve interfacial bonding, thereby increased mechanical performance [16]. In the literature, CNT treatment is not attempted so far to improve the bonding of polar sisal fibres with non-polar epoxy matrix. This fact was the motivation to investigate the effect of alkali (chemical) treatment and CNT modification of the sisal fibres and CNT reinforcement on the viscoelastic and bending properties of epoxy matrix bio-composites.

II. MATERIALS AND METHODS

A. Materials

Epoxy AY105 was used as a matrix material, and HY905 was used as a hardening agent for initiating the polymerization. Epoxy AY105 and hardener HY905 were purchased from the Universal Enterprises, Kanpur. Sisal-fibres (un-treated, chemically treated, and CNT treated) and Multiwall Carbon Nano Tubes (MWCNTs) were used as reinforcement materials. The properties and chemical composition of sisal fibres purchased from the local resources are given in Table 1 [17]. MWCNTs were purchased from Nano Research Lab, Jharkhand, India. The diameter, average length, and purity of MWCNTs were 20 nm, 20 µm and 98%, respectively.

Table 1: Properties of Sisal fibre.

Cellulose (%)	Lignin (%)	Hemicellulose (%)	Tensile strength (MPa)	Density (g/m^3)	Young's Modulus (GPa)
70	12-16	10-14	511-700	1.5	9.4-22

B. Surface treatment of fibres

Purchased long sisal-fibres were dried at 70°C for 24 hrs. Then these were cut into small pieces of around 5mm using scissors. Then the ball milling of small pieces of fibres was done at 200 rpm for 80 hours in the planetary ball milling machine to produce the micron-size sisal fibres (all less than 75 µm).

Alkali treatment of the fibres was done to increase the cellulose content in fibres because cellulose is more crystalline and stable in nature than lignin and hemicellulose. Higher is the cellulose content in the fibres; more is compatibility with the non-polar polymer matrix. Lignin and hemicellulose are less stable and increase the water absorption capabilities of natural fibres. The obtained micron size sisal fibres were first submerged in 12% solution of sodium hydro-oxide at 80°C for 12 hrs. Then these fibres were counterbalanced by soaking in one molar HCl solution at 80°C for 1 hr. Then these fibres were cleaned with water and kept for drying at 60°C. Fig. 1 shows the flowchart for the alkali treatment of sisal fibres.

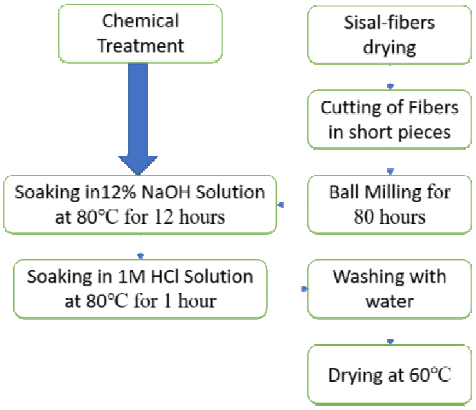

Fig. 1. Flowchart of alkali treatment of sisal fibre.

CNT treatment of sisal-fibres was done to increase the compatibility of polar sisal-fibres with a non-polar polymer matrix. CNT treatment changes the polar nature of sisal-fibres into non-polar nature because functionalized MWCNTs form the physical bonding between the hydro-oxyl groups present on sisal-fibres.

Fig. 2 shows the diagrammatic depiction of the functionalization of MWCNTs and the formation of physical bonding between Sisal-fibres and MWCNTs. For the functionalization of CNTs, CNTs were soaked in three-molar nitric acid solution and stirred using a magnetic stirrer at 60°C for 15 minutes. Then these were placed in an oven at 60°C for 24 hrs. Then 1 gram of sisal fibres obtained after ball milling was mixed with 1 mg/ml solution of functionalized CNTs and stirred using a magnetic stirrer at room temperature for 24 hrs. Then obtained CNT treated sisal fibres were washed and dried at 60°C for 24 hrs. Fig. 2 shows the flowchart for the CNT treatment of Sisal-fibres. The one wt.% of CNT was added into the epoxy matrix, and uniform dispersion was assured using Magnetic stirrer.

C. Synthesis of composites

All the samples were developed by the hand-lay-up method. Epoxy AY105 was used as a matrix material, and sisal fibres (un-treated, chemically treated, and CNTs treated) and CNTs were used as reinforcement materials. Table 2 shows the composition and code of the prepared specimens. Matrix and reinforcement materials were mixed well using a magnetic stirrer for 30 minutes. After mixing, hardening agent HY905 (1/10th of the weight of Epoxy AY105) was added and mixed well before pouring into the mould. Fabricated samples were then kept under pressure at normal room temperature for curing for 24 hours.

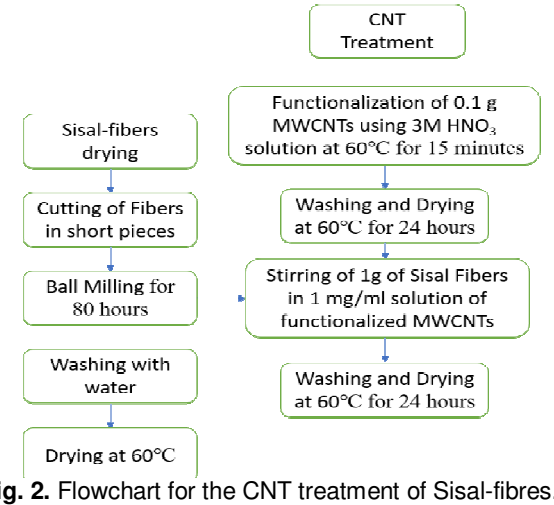

Fig. 2. Flowchart for the CNT treatment of Sisal-fibres.

Table 2: Specimens composition and code.

S. No.	Specimens	Sample Code
1.	Neat Epoxy	S1
2.	Epoxy + 1 wt.% untreated sisal fibres	S2
3.	Epoxy + 1% alkali-treated sisal fibres	S3
4.	Epoxy + 1% CNT treated sisal fibres	S4
5.	Epoxy + 1% CNT blending	S5

D. Characterization

Flexural tests of the composites were performed on Tinius Olsen three-point bend testing machine as per ASTM D790. Bend tests were carried out in open atmosphere at normal room temperature (30°C). The flexural stress and flexural modulus for all the samples were calculated on the basis of results obtained during a three-point bend test using the mathematical formulas given below.

Flexural stress = $3PL/2bt^2$

Flexural modulus = $mL^3/4bt^3$

Where P = applied breaking force (N); b, t and L = width, thickness and span length of sample (mm) respectively, and m = slope of the load v/s displacement plot.

The viscoelastic properties of fabricated samples were investigated using Dynamic Mechanical Analyzer 6100. For the DMA tests, the temperature was varied from 30 to 120℃. Rate of heating was kept constant at 10℃/min, while the frequency was kept constant at 1 Hz.

III. RESULTS AND DISCUSSION

A. Flexural Test

The flexural stress v/s deflection curves plotted using the data obtained from the three-point bending tests of fabricated samples are shown below in Fig. 3.

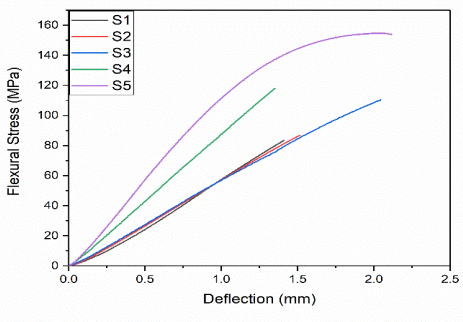

Fig. 3. Flexural stress v/s deflection curve of neat epoxy and sisal composites.

The computed values of flexural modulus and stress are given in Table 3. It can be observed from the graph that flexural stress of neat epoxy was increased on the addition of sisal-fibres, and also found to further increased due to treatments (alkali and CNT) and blending of CNT. It shows that treatments of sisal fibres and blending of CNTs with epoxy matrix have a constructive outcome on the properties. An increase in the stress value is due to effective load transfer from the epoxy matrix to reinforced sisal fibres credited to treatments and blending. The alkali-treated sisal composite S3 has 27.49% more flexural strength than that of untretaed sisal composite. It was due to enhanced interfacial bonding after alkali (chemical) treatment. The irregularities at surface of the fibres increases after the chemical treatment. It increases the interfacial bonding and causes an effective stress transfer [15, 16]. The increase in flexural strength after alkali treatment has already been reported in past literature [15, 16, 18]. Further, CNT treated composite S4 has 41.51% enhancement in flexural strength and 111.23% enhancement in flexural modulus than neat epoxy. It was due to lowering in polar behavior of sisal fibres by CNTs treatment, thereby improvement in bonding and compatibility between fibres and matrix. The flexural stress enhancement after CNT treatment has been described already by Rahmanian *et al.*, [19]. Furthermore, on comparing with neat epoxy, there was 85.43% improvement in flexural strength, and 30.54 % improvement in flexural modulus for CNTs blended composite S5 due to uniform stress transfer.

Table 3: Flexural stress and modulus of sisal composites.

Sample	Flexural Stress (MPa)	Flexural Modulus (GPa)
S1	83.36	3.47
S2	86.62	3.85
S3	110.44	3.32
S4	118.00	7.33
S5	154.58	4.53

B. DMA Test

Storage Modulus (E'): The stiffness or elastic behavior of the material is measured in terms of E'. Fig. 4 shows the storage modulus v/s temperature curves of the prepared composites. In the glassy region, it can be seen that the neat epoxy sample has the lowest value of E', whereas its highest value was obtained for the CNT blended composite S5. While, intermediate values were offered by the chemically and CNT treated composites. It can be attributed to improvement in the stiffness of fibres after treatments. On increasing the temperature, the storage modulus was observed to be reduced for all the composites.. It is due to a decrease in stiffness of fibres and softening of the matrix [14, 18, 20, 21]. In the transformation region, a sudden descend in storage modulus values for all the composites was seen. At higher temperatures, a better performance was shown by the untreated composite S2, followed by alkali-treated composite S3. In the case of CNT treated composites, a lower value of storage modulus was due to weakening in hydrogen bond between sisal fibres and functionalized MWCNTs at increased temperature. It can also be observed that the start of the rubbery region of the composites follows the order: S3 > S2 > S1> S5 > S4. Finally, this can be concluded that chemically-treated composite performed better than other composites in terms of storage modulus. Improvement in the storage modulus of jute fibre reinforced composite after chemical treatment has been described by Gupta *et al.*, already [18].

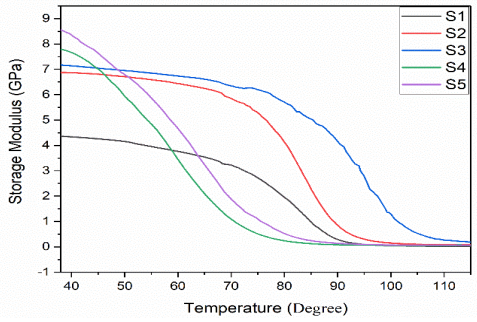

Fig. 4. Storage Modulus vs. Temperature curves for Prepared Specimens.

Loss Modulus (E"): Loss modulus or viscous modulus is the measure of energy loss in internal friction during deformation in the specimen. It describes the adhesive response of the composites [20, 21]. Fig. 5 shows the loss modulus v/s temperature plots for fabricated composites specimens. It can be seen in the figure that when temperature increases, loss modulus first increase and reach to its highest value and then starts to decline. The lowest peak was seen for neat epoxy as expected. Whereas its highest peak was obtained for composite S5 followed by composites, S2, S4 & S3. T_g of each sample was obtained from the plot based on the temperature associated with the highest peak of each curve. The crest values of the loss modulus curve and T_g values of each sample are given below in Table 4. The value of T_g was highest for sample S3 credited to alkali treatment. The increase in loss modulus value after alkali treatment has already been reported in the literature [18, 21]. The T_g obtained from the loss modulus plot was observed to be less than the T_g obtained from Tan δ plot. This result was in accordance with results reported by [18, 20, 21].

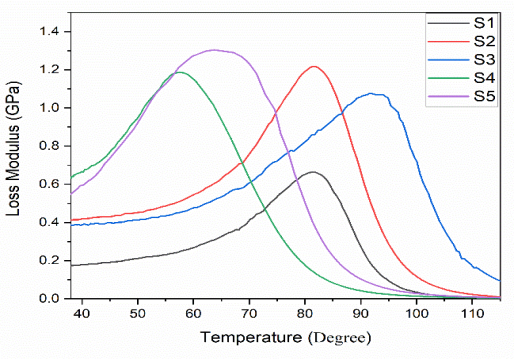

Fig. 5. Loss modulus v/s temperature curves for neat epoxy and sisal composites.

Tan δ (Damping): Tan δ is computed by dividing loss modulus by storage modulus. It is the measure the viscous behaviour and the load carrying capability of the composites specimen [14, 20, 21]. Fig. 6 reveals the Tan δ values v/s temperature plot of the composites. The temperature associated with the at the crest value of the Tan δ plot gives the value of T_g. It can be observed that Tan δ peaks were found to increase up to T_g of each composite and then decreased on increasing the temperature. It can also be observed from the plot that the pure epoxy sample has the highest Tan δ value. It is due to rise in the movement of the polymeric chain on raising the temperature. It describes its higher damping and low load carrying capability. The lowest value of Tan δ was seen for alkali-treated composite S3, followed by composites S4 & S5. The lowest value of Tan δ for composite S3 shows its excellent load carrying capability and low damping. Similar results of alkali treatment of jute fibres were shown by Gupta *et al.,* [18]. The values of T_g from the Tan δ curve was calculated and illustrated in Table 4. The highest value of T_g was obtained for composite S3 due to powerful interfacial bonding offered by alkali treatment, which resists the molecular movement. In the case of CNT treated composite S4, lower glass transition temperature might be due to declining of the hydrogen bond between fibres and matrix.

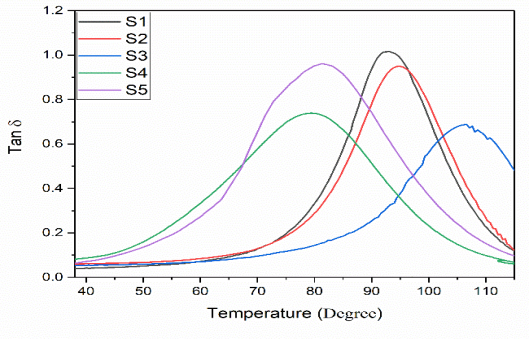

Fig. 6. Tan δ v/s temperature curve for neat epoxy and sisal composites.

Table 4: DMA test results of samples.

Sample	The crest value of E" (GPa)	The crest value of Tan δ	T_α (℃) from E"	T_α (℃) from Tan δ
S1	0.66	1.01	81.78	92.05
S2	1.22	0.94	81.99	95.48
S3	1.08	0.68	91.63	106.66
S4	1.19	0.73	57.53	79.95
S5	1.30	0.96	63.13	81.29

IV. CONCLUSIONS

Following conclusions are drawn after the experimental study:

– The addition of sisal-fibres enhance both strength and T_g of the neat epoxy.

– A better dynamic mechanical performance in terms of loss modulus, storage modulus, and glass transition temperature was observed for the chemically-treated sisal fibres reinforced composite as compared to other samples.

– The highest flexural strength was obtained for CNT blended composites followed by CNT treated composite.

–The CNT treatment is good at room temperature applications only. Chemical treatment of sisal-fibres is more effective at higher temperatures in comparison to the CNT treatment.

V. FUTURE SCOPE

CNT treatment and blending were found an effective method to enhance the performance of the Biocomposites. In future, effect of these treatments could be studied on thermal, rest mechanical properties, wear and insulating properties of the biocomposites.

Conflict of Interest. There is not any type of conflict of interest related to the current research.

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