

Properties of sisal fibre reinforced epoxy composite

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Sisal fibre reinforced epoxy composites have been prepared by hand lay-up technique followed by static compression, using various fibre weight fractions (15, 20, 25 and 30%). Mechanical properties, thermal properties, water absorption properties and dynamic mechanical analysis of sisal composites are investigated. The results show that the addition of sisal fibres in epoxy matrix up to 30 wt. % increases the mechanical, thermal and water absorption properties. The values of storage modulus and loss modulus increase with the increase in fibre content up to 25 wt. % and then decrease. The glass transition temperature (T_g) is obtained from loss modulus and tan delta curve. The value of T_g obtained from loss modulus curve is found to be lower than that obtained from tan delta curve.

Keywords: Dynamic mechanical analysis, Mechanical properties, Sisal fibre, Thermal properties, Water absorption properties

1 Introduction

There is a rising interest of researchers in the use of natural fibres as reinforcement for polymer composite. Natural fibres have many advantages, such as low cost, low density, availability in abundance, eco-friendliness, non-toxicity, high flexibility, renewability, biodegradability, relative non-abrasiveness, and high specific strength and modulus¹⁻⁴. However, natural fibres suffer from some disadvantages also, like low impact strength, high brittleness and higher moisture absorption properties⁵. Nowadays, natural fibre reinforced polymer composites are being used in automotive parts, aerospace and constructions industries^{5,6}.

Kaewkuk *et al*⁷. studied the physical properties of sisal fibre reinforced polypropylene composite and found that on increasing the sisal fibre content, the tensile strength, tensile modulus and water absorption properties of sisal polypropylene composite increase but impact strength and elongation-at-break decrease. Mohanthy *et al*⁸. studied the mechanical and viscoelastic behavior of jute fibre reinforced high density polyethylene composites and observed that the tensile, flexural and impact strength are found to be increased with the increase in fibre loading up to 30 %. Storage modulus was increased on increasing fibre loading, whereas damping parameters are decreased as compared to epoxy. Cheng *et al*⁹.

presented studies on mechanical and thermal properties of chicken feather reinforced PLA composites. They observed that the addition of chicken feather as reinforcement into PLA, enhanced the tensile moduli and thermal stability of composites as compared to pure PLA. Girisha *et al*¹⁰. reported study on the mechanical properties and water absorption behavior of sisal and coconut coir fibre reinforced epoxy composite. They highlighted that as a result of hybridization of sisal fibre with coconut coir epoxy composite, the mechanical properties were improved and water absorption property was reduced. Venkateshwaran *et al*¹¹. investigated the effect of sisal fibre loading on mechanical and water absorption properties of banana fibre reinforced epoxy composite and reported that the addition of sisal fibre results in increased mechanical properties and decreased water absorption properties of banana fibre reinforced epoxy composite.

The present study aims at investigating the mechanical properties, thermal property, water absorption properties and dynamic mechanical analysis of sisal fibre reinforced epoxy composite. The proposed sisal composites found are suitable in light weight automotive parts application.

2 Materials and Methods

2.1 Materials

Sisal fibres were used as reinforcement and epoxy AY 105 as a matrix in this study. Sisal fibres and epoxy matrix were purchased from local resource.

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Epoxy is a thermosetting polymer and hence requires a hardener for curing; HY951 hardener was used. Density and dynamic viscosity of epoxy resin are 1.108 g/cm³ and 11.789 Pa.s respectively. The matrix material was prepared by using epoxy and hardener in the ratio 10:1, as recommended¹¹.

Sisal fibre (*Agave sisalana*) was extracted by decortication, wherein leaves were crushed and beaten by a rotating wheel set with blunt knives. The properties of sisal fibre have been reported earlier^{12,13}.

2.2 Fabrication of Composites

Hand lay-up technique was used to fabricate the composites by reinforcing sisal fibres into epoxy matrix. Composites were prepared using various fibre weight fractions (15, 20, 25 and 30 wt. %) with unidirectional alignment of sisal fibres. A stainless steel mould having dimensions of 500 × 300 × 3 mm³ was used for casting of composites. 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 h before its removal from the mould. Dimension of specimens were cut as per ASTM standard using a diamond cutter. The composites manufactured with varying wt. % of fibres are designated as S15 (15 wt. % of sisal fibre), S20 (20 wt. % of sisal fibre), S25 (25 wt. % of sisal fibre) and S30 (30 wt. % of sisal fibre).

2.3 Testing and Characterizations of Composites

The fabricated sisal fibre reinforced epoxy composites were tested for mechanical, thermal, water absorption properties and dynamic mechanical analysis.

2.3.1 Tensile Test

Tensile properties tests of the composite samples were performed on Tinius Olsen H 10 K-L (Bi-axial testing machine) with a crosshead speed of 2 mm/min and temperature of 30 °C. Tests were conducted as per ASTM D638, using sample of dimension 165 mm × 13 mm × 3 mm. Five specimens of each composite were tested and average values are reported.

2.3.2 Flexural Test

Flexural properties of the composite were determined using a three point bending test on Tinius Olsen H10 K-L (Bi-axial testing machine). The dimension of the sample used for the flexural test was taken as 80 mm × 12.7 mm × 3 mm as per ASTM D790. The flexural test was carried out at 30 °C temperature and 2 mm/min crosshead speed. Flexural

strength and flexural modulus were calculated using the following equations¹⁴:

$$\text{Flexural strength} = \frac{3FL}{2bd^2} \text{ and} \quad \dots (1)$$

$$\text{Flexural modulus} = \frac{mL^3}{4bd^3} \quad \dots (2)$$

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

2.3.3 Impact Test

Impact test of composite was performed on Tinius Olsen Impact 104 machine. The dimension of the sample used for the impact test was taken as 65 mm × 12.7 mm × 3 mm, with notch thickness of 2.5 mm, as per ASTM D 256. Five specimens of each composite were tested and average values are reported.

2.4 Water Absorption Behavior

Behavior of water absorption by sisal fibre reinforced epoxy composites has been studied. The water absorption causes the degradation of fibre-matrix interface region, resulting in reduction of mechanical properties along with the change in dimensions of composites. Water absorption by natural fibre reinforced polymer composite is very similar to Fickian diffusion process¹⁵. Diffusion is defined as the mass flow process by which molecules change their position under the influence of thermal energy and gradient (concentration, electrical, magnetic and stress). Under the steady state condition and unidirectional flow of matter, Fick's first law states¹⁵:

$$\phi = -D \frac{\partial C}{\partial x} \quad \dots (3)$$

where ϕ is the flux (flow of matter per unit area and per unit time); D , the diffusion coefficient; and $\frac{\delta c}{\delta x}$, the concentration gradient¹⁵.

Under the non steady state condition and unidirectional flow of matter, Fick's second law states¹⁶.

$$\frac{\partial c}{\partial x} = D \frac{\partial^2 c}{\partial x^2} \quad \dots (4)$$

Water absorption behavior of sisal fibre reinforced polymer composite was investigated as per ASTM D 570. The specimens were submerged in water at 30 °C temperature to study the kinetics of water absorption. The samples were taken out periodically and weighed immediately after wiping out the water from the surface of samples. Water absorption by the sample was measured using a precise 4-digit balance. The percentage of water absorption was calculated using the following equation¹⁶:

$$\text{Water absorption (\%)} = \frac{W_2 - W_1}{W_1} \times 100 \quad \dots (5)$$

where W_1 is the weight before soaking into water (g); and W_2 , the weight after soaking into water (g). The higher diffusivity of one substance with respect to another shows that they diffuse into each other faster. The kinetic parameter, diffusion coefficient (mm^2/s), was calculated using the following equation¹⁶:

$$\text{Diffusion coefficient (D)} = \pi \left(\frac{t^2 m^2}{16M_s^2} \right) \quad \dots (6)$$

where m is the slope of linear portion of the sorption curve; and t , the initial sample thickness in (mm).

The permeability of water molecules through the composite sample depends on the sorption of water by the fibres. Therefore, the sorption coefficients (related to the saturation sorption) was calculated using the following equation¹⁶:

$$\text{Sorption coefficient } S = M_s / M_t \quad \dots (7)$$

where M_s and M_t are the percentage of water uptake at saturation time and at a specific time t respectively.

The permeability coefficient P (mm^2/s), which implies the net effect of sorption and diffusion

coefficient was calculated using the following equation¹⁶:

$$\text{Permeability coefficient } P = D \times S \quad \dots (8)$$

2.5 Thermogravimetric Analysis

Thermal stability of the composites was assessed by thermogravimetric Perkin Elmer TGA 4000 apparatus. TGA measurements were carried out on 15-25 mg sample placed in a platinum pan, heated from 30 °C to 800 °C at a heating rate of 10 °C/min in a nitrogen atmosphere with a flow rate of 20 mL/min to avoid unwanted oxidation.

2.6 Dynamic Mechanical Analysis

Viscoelastic properties of fibre reinforced polymer composites depend on the nature of the matrix, reinforcement and fibre–matrix interfaces. The viscoelastic properties of epoxy and sisal composites were studied by using the dynamic mechanical analyzer (Seiko instruments DMA 6100). The viscoelastic properties were determined in 3 point bending test at 1 Hz frequency as a function of temperature. The composites were cut into samples of dimensions 50 mm ×13 mm × 3 mm according to ASTM D 5023. Experiments were carried out in the temperature range 30°–200°C at a heating rate of 10°C/min. The viscoelastic properties such as storage modulus, loss modulus and damping parameter of the specimens were measured.

3 Results and Discussion

3.1 Mechanical Properties

The tensile, flexural and impact properties of epoxy and sisal fibre reinforced epoxy composite are given in Table 1.

3.1.1 Tensile Properties

Table 1 shows the tensile strength and tensile modulus of epoxy and sisal fibre reinforced epoxy composites. The tensile strength and tensile modulus of sisal composites are found to be increased with increasing sisal fibre content up to 30 wt. %. The maximum values of tensile strength and tensile

Table 1— Mechanical properties of epoxy and sisal composites

Composite	Tensile strength MPa	Tensile modulus GPa	Flexural strength MPa	Flexural modulus GPa	Impact strength kJ/m ²	Impact energy J
Epoxy	33.86±2.59	0.712±0.01	118.73±10.49	5.781±0.60	5.67±0.35	0.14±0.01
S15	44.93±3.80	0.905±0.07	155.99±12.99	7.584±0.83	16.84±1.46	0.55±0.07
S20	47.01± 2.40	1.205±0.10	169.99±12.19	9.174±1.08	18.01±1.83	0.65±0.12
S25	61.46± 4.59	1.222±0.10	180.57±17.25	9.445±1.14	19.96±2.86	0.87±0.17
S30	83.96± 6.94	1.58±0.08	252.39±12.11	11.316±1.02	22.03±1.74	1.09±0.10

modulus are found for the composite S30 due to strong adhesion between sisal fibres and epoxy matrix which allows a uniform transfer of stress from matrix to fibres. Tensile strength and tensile modulus of the composite S30 are found 83.96 MPa and 1.580 GPa, which are 148% and 122% more than that of epoxy. It is observed that tensile strength of composite S30 is 87, 79 and 37% more than those of composites S15, S20 and S25 respectively, and tensile modulus is 75, 31 and 29% more than those of composites S15, S20 and S25 respectively.

3.1.2 Flexural Properties

The flexural strength and flexural modulus of epoxy and sisal fibre reinforced epoxy composites are shown in Table 1. The flexural strength and flexural modulus of sisal composite are also found to be increased with increasing sisal fibre content up to 30 wt.%. The composite S30 offers the maximum value of flexural strength and flexural modulus due to strong fibre-matrix adhesion. Flexural strength and flexural modulus are found maximum for composite S30 such as 252.39 MPa and 11.316 GPa respectively, which are 113% and 96% more than that of epoxy. It is observed that the flexural strength of composite S30 is 62, 48 and 40% more than those of composites S15, S20 and S25 respectively, and flexural modulus is 49, 23 and 20% more than those of composites S15, S20 and S25 respectively.

3.1.3 Impact Properties

Table 1 shows the Impact strength and impact energy of epoxy and sisal fibre reinforced epoxy composites. The similar trend of tensile and flexural properties is seen for impact properties of sisal fibre reinforced epoxy composites. Impact properties are found to be increased with increasing sisal fibre content up to 30 wt. % in epoxy matrix. Impact strength and impact energy are found to be maximum for composite S30 such as 22.03 kJ/m² and 1.0909 J respectively, which are very high than those of epoxy. The impact strength is found 31, 22 and 10% higher than those of composites S15, S20 and S25 respectively, and impact energy is 99, 68 and 26 % higher than those of composites S15, S20 and S25 respectively.

3.2 Water Absorption Behaviour

The percentage water absorption of sisal fibre reinforced epoxy composites is plotted against the square root of time (Fig. 1). It is found that the initial rate of water absorption and maximum water uptake

increase with the increase in fibre content up to 30 wt.%. This may be due to the presence of micro-voids in matrix; water molecules start diffusing into micro-voids till saturation state. The composite S30 shows the higher water absorption, which is 13, 10 and 4% more than those of composites S15, S20 and S25 respectively. The increase in water absorption is due to the hydrophilic nature of sisal fibre and greater interfacial area between fibre and matrix¹⁶. Sisal fibre shows the hydrophilic nature due to presence of cellulose. On increasing the sisal fibre content, weight fraction of fibres is increased which causes increase in amount of cellulose, micro voids and interface surface area. The water absorption by epoxy is almost negligible due to its hydrophobic nature. The sorption, diffusion and permeability coefficient of sisal fibre reinforced epoxy composites are given in Table 2. The composite S15 shows the higher values of diffusion and permeability coefficient than all other composites, due to lower fibre loading.

3.3 Thermogravimetric Analysis (TGA)

Figure 2 shows the variation in percentage weight loss of epoxy and sisal composites with temperature. It is observed that there are three significant regions

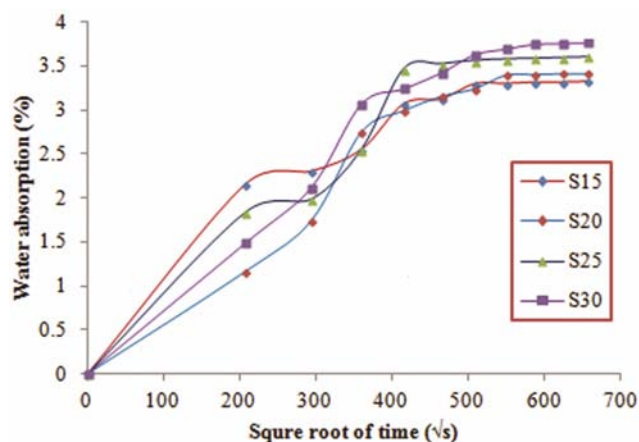


Fig. 1—Water absorption of sisal composites as a function of square root of time

Table 2—Sorption, diffusion and permeability coefficient of sisal composites

Composites	% water uptake at saturation time (M_{∞})	Sorption coefficient (S)	Diffusion coefficient (D) $\text{mm}^2/\text{s} \times 10^{-5}$	Permeability coefficient (P) $\text{mm}^2/\text{s} \times 10^{-5}$
S15	3.32	1.54	1.71	2.63
S20	3.41	2.93	0.48	1.41
S25	3.60	1.96	1.06	2.07
S30	3.76	2.53	0.64	1.62

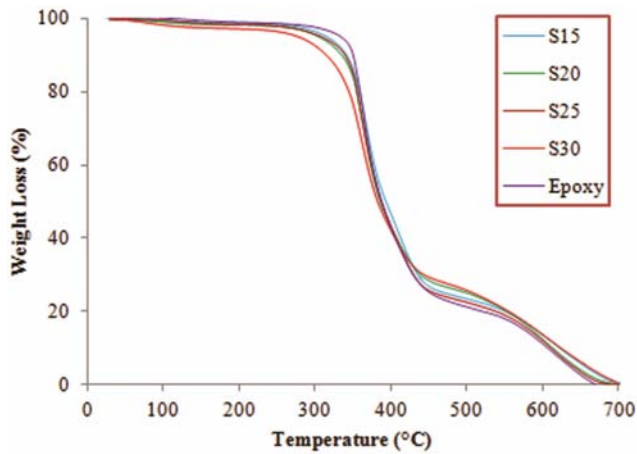


Fig. 2— Variation in weight loss of epoxy and sisal composites with temperature

of weight loss due to rise in temperature. The initial weight losses ($\sim 5\%$) of epoxy and sisal composites S15, S20, S25 and S30 are obtained at 330° , 317° , 306° , 310° and 279°C respectively. The initial low temperature weight losses of composites are due to the removal of solvent from composites¹⁷. The major weight losses ($\sim 75\%$) of epoxy and sisal composites S15, S20, S25 and S30 are obtained at 450° , 470° , 502° , 460° and 508°C respectively. The major weight loss is due to degradation and volatilization of epoxy along with the fibres present in composites¹⁷. The residue formed after degradation requires higher temperature for subsequent degradation. The final weight losses of epoxy and sisal composites S15, S20, S25 and S30 are obtained at 679° and, 688° , 680° , 672° and 694°C respectively. The major weight loss of the composite S30 occurs at 508°C . Here, degradation is shifted towards higher temperature which shows increased thermal stability of the composite S30 due to stronger interface between fibres and matrix as compared to the all other composites.

3.4 Dynamic Mechanical Analysis

The dynamic mechanical analysis (DMA) of composite samples was performed to study their viscoelastic properties. shows the variation in E' , E'' and $\text{Tan}\delta$ of the epoxy and sisal composites as a function of temperature at a frequency of 1 Hz.

3.4.1 Storage Modulus

Storage modulus (E') is amount of energy stored by materials during one cycle of oscillation. Figure 3(a) shows the variation in storage modulus of epoxy and sisal composites as a function of

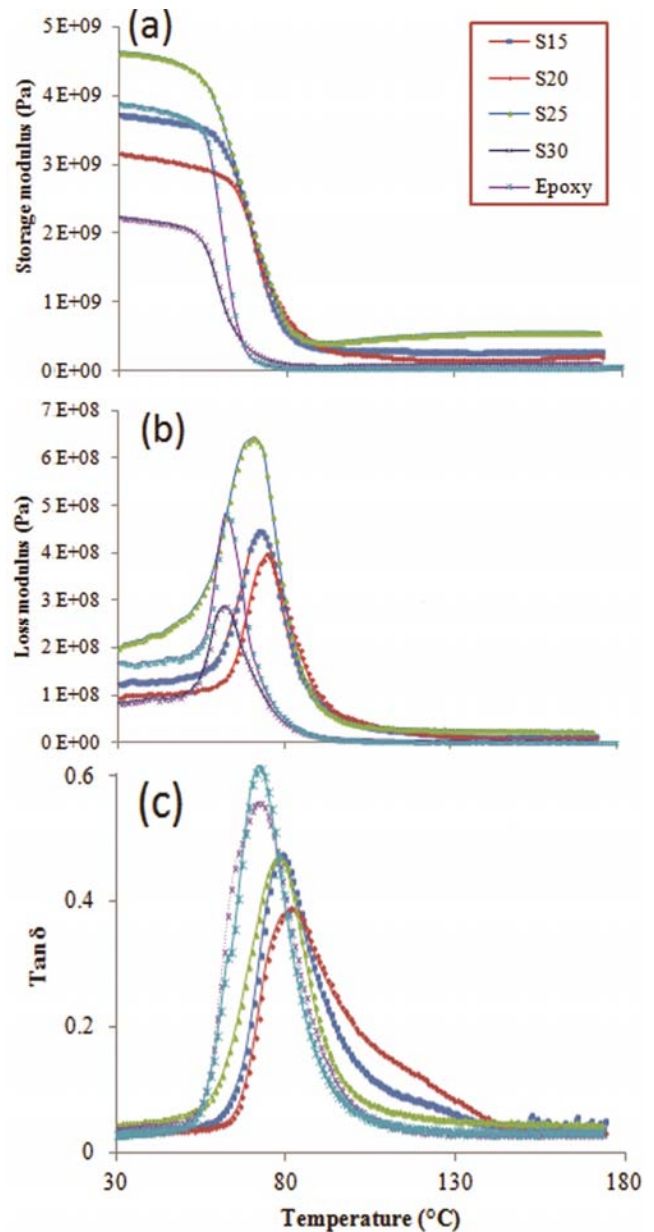


Fig. 3— Variation in (a) storage modulus, (b) loss modulus and (c) $\text{Tan}\delta$ with temperature of epoxy and sisal composites

temperature at a frequency of 1 Hz. On comparing the different composites, it is found that the value of E' increases with an increase in weight fraction of sisal fibres up to 25 wt. %. The value of E' is found 3.6532 GPa for epoxy in the glassy region, but this value reaches up to 4.4172 GPa for the composite S25 due to reinforcement of sisal fibres in epoxy matrix. The storage modulus of the epoxy and sisal composites decrease as temperature is increased due to the loss in stiffness of fibres. Epoxy has a very sudden fall in the value of E' , whereas sisal

composites have a gradual fall in the value of E' due to the incorporation of high modulus sisal fibres in the epoxy matrix¹⁸. In the rubbery region, storage modulus of epoxy is found much lower than those of sisal composites due to increase in molecular mobility at higher temperature. The value of E' of epoxy is 0.22 GPa, whereas with the incorporation of sisal fibre this value is increased to 0.868 GPa for composite S25. This may be credited to the reinforcement of sisal fibres which allow proper stress transfer from the epoxy to the sisal fibres. Following equation is used to calculate the effectiveness of reinforcement (ϵ) of the composites¹⁸:

$$\epsilon = \frac{\left(\frac{E'_g}{E'_r}\right)_{Composite}}{\left(\frac{E'_g}{E'_r}\right)_{Epoxy}} \dots (9)$$

where E'_g and E'_r are the storage modules in the glassy and rubbery region respectively. The higher value of ϵ reflects lower efficiency of the reinforcement and vice-versa. The effectiveness constant of sisal fibre reinforced epoxy composites is given in Table 3. The composite S25 shows the lowest value of effectiveness constant, whereas composite S30 shows the highest value (0.632) of effectiveness constant. The lower value of effectiveness constant (0.334) for sisal composite S25 shows higher efficiency of reinforcement than all other composites.

3.4.2 Loss Modulus

Loss modulus (E'') is defined as loss of energy in the form of heat from materials per cycle of oscillation¹⁹. The glass transition temperature (T_g) can be obtained from the peak of either E'' or $\text{Tan}\delta$ curve. The variation in loss modulus as a function of temperature at 1 Hz frequency is shown in the Fig. 3 (b). On increasing the temperature the value of E'' is found to increase up to glass transition temperature and then it decreases. In glassy region, the increase in values of E'' follows the order: S25 > Epoxy > S15 > S20 > S30. The higher value of T_g is found to be maximum for the composite S20 due to incorporation of sisal fibres, causing decrease in mobility of epoxy. The value of glass transition temperature for epoxy

Table 3—Effectiveness constant of reinforcement (ϵ) for sisal composites

Composite	E'_g of composites GPa	E'_r of composites GPa	E'_g of epoxy GPa	E'_r of epoxy GPa	ϵ
S15	3.5521	0.6024	3.6532	0.2263	0.365
S20	2.8456	0.4607	3.6532	0.2263	0.382
S25	4.4172	0.8183	3.6532	0.2263	0.334
S30	2.0903	0.2047	3.6532	0.2263	0.632

Table 4—Peak height and glass transition temperatures (T_g) of epoxy and sisal composites

Composite	Peak height of loss modulus curve, GPa	Peak height of $\text{Tan}\delta$ curve	T_g , °C	
			Loss modulus curve	$\text{Tan}\delta$ curve
Epoxy	0.482	0.6170	62.17	71.77
S15	0.446	0.4726	73.29	78.48
S20	0.394	0.3884	74.34	81.23
S25	0.642	0.4698	70.88	77.52
S30	0.286	0.5558	62.61	71.63

and sisal composites obtained from loss modulus curve is given in Table 4.

3.4.3 Damping

Damping ($\text{Tan}\delta$) can be determined by the ratio of loss modulus and storage modulus. $\text{Tan}\delta$ shows the damping properties of material. The composite having strong interface between fibres and matrix shows lower value of $\text{Tan}\delta$ and higher damping property. The variation in $\text{Tan}\delta$ of epoxy and sisal composites as a function of temperature at a frequency of 1 Hz is shown in Fig. 3(c). The maximum value of $\text{Tan}\delta$ is found 0.6170 for epoxy as expected, showing better damping properties. The values of $\text{Tan}\delta$ peak and T_g of epoxy and sisal composites is given in Table 4. The value of T_g increases with the increase in fibre loading as compared to epoxy (Table 4). The value of T_g obtained from $\text{Tan}\delta$ curve is higher than that obtained from loss modulus curve (Table 4).

4 Conclusion

4.1 Mechanical properties of prepared sisal composites are found to be increased on increasing sisal fibre content in epoxy matrix. The composite S30 shows the maximum value of mechanical properties (tensile strength and modulus, flexural strength and modulus, and impact strength and energy) as compared to epoxy and other sisal composites.

4.2 Thermal stability is found the maximum for the sisal composite S30 as compared to epoxy and other sisal composites.

4.3 The water uptake by sisal composites is found to be increased on increasing the fibres loading in epoxy matrix. The composite S30 shows the maximum water uptake as compared to all other composites.

4.4 Storage modulus of epoxy and sisal composite is found to decrease at higher temperature due to loss in stiffness. Storage modulus as well as loss modulus is found to be high for the sisal composite S25.

4.5 On increasing the sisal fibre content, the value of T_g increases as compared to epoxy. The value of T_g obtained from loss modulus curve is lower than that obtained from $\text{Tan}\delta$ curve for epoxy and all composites.

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