

Influence of SiC Particulates on Dynamic Mechanical Response of Treated *Luffa Cylindrica* Epoxy Composites

S. Chethan^{1*}, S. Suresha¹ and Govardhan Goud²

¹Department of Mechanical Engineering, ATME College of Engineering, Mysuru – 570028, Karnataka, India; chethans.nie@gmail.com, surs.mar69@yahoo.com

²Department of Mechanical Engineering, Bahubali College of Engineering, Sravanabelagola, Hassan – 573135, Karnataka, India; pgovardhan0@yahoo.com

Abstract

Objective: To characterize dynamic response of Epoxy composites reinforced with optimally treated *Luffa Cylindrica* fibres and Silicon Carbide particulates. **Methods:** Hand Lay-up technique is used for composite fabrication. Two layer of *Luffa Cylindrica* fibres and 0% to 7% by weight, as decided by Central Composite Design of Experimental technique, of Silicon Carbide particulates are reinforced. The composites are subjected to Dynamic Mechanical Analysis with temperature ramped from 26°C to 160°C and 1Hz frequency. **Findings:** About 2300 MPa high storage modulus is noticed within temperature range of 30°C to 70°C. Low loss modulus of 282.35 MPa is observed for composites with 3.5wt% Silicon Carbide at glass transition temperature of 80°C. **Improvement:** Inclusion of Silicon Carbide particulates has yielded lower damping ratio and higher thermal stability of 95.62°C.

Keywords: Design of Experiments, Dynamic Mechanical Analysis, Epoxy, *Luffa Cylindrica*, Silicon Carbide

1. Introduction

Over past few decades, polymer composites are preferred over conventional materials. These composites are used in many applications due to ease of processing, productivity, decrease in cost etc. Presently the researchers are concentrating or emphasising on the synthesis of new green materials with environment as the key concern. Some of the natural fibres used to prepare polymer composites are sisal, hemp, jute, flax, banana, palm, coir, kenaf, *Luffa Cylindrica* etc. These fibres are available

in abundance, biodegradable, recyclable and possess appreciable mechanical strength.

Epoxy is chosen as matrix material with natural fibres as reinforcement due to its diverse and unique characteristics. Basically Epoxy is a resin material with at least one epoxide unit in the group or molecule. Epoxy exhibits comparatively many advantages over other resins like thermoset and thermoplastics namely minimum cheaper and abundant, shrinkage during curing, good moisture and chemical resistant, long shelf life, good mechanical strength etc. The Epoxy modifications are

*Author for correspondence

done to improve its toughness, brittleness, wear etc. hence they are reinforced with natural fibres. Fillers can also be used for further enhancing its properties and manufacture high performance engineering materials¹.

The introduction of *Luffa Cylindrica* as reinforcement greatly increases the mechanical properties of the composite due to the nature of reinforcement. Increase in the mechanical stiffness of about 48% is noticed by reinforcing *Luffa Cylindrica* in Epoxy matrix material². It has been noticed that significant improvement in the mechanical properties of chemically treated *Luffa Cylindrica* fibres is achieved compared to that of fibres without treatment. Because treatment results in rough surface produced on the fibre which results in good adhesion between matrix and fibre³. An astonishing modification of mechanical properties of Epoxy composite is noticed because of chemical treatment of kenaf fibres with NaOH solution ensuring the fibre-matrix compatibility⁴.

The effect of hybridization, by introducing glass fibre to *Luffa Cylindrica* reinforced Epoxy hybrid composites, has resulted in increased tensile and flexural strength by taking characteristic advantages of both the fibres⁵.

Addition of micro fillers such as CaCO_3 , Al_2O_3 and TiO_2 has enhanced the tensile, bending and impact strength of *Luffa Cylindrica* reinforced polyester composites compared to that of unfilled composites⁶. Incorporation of Silicon Carbide filler has resulted in enhanced strength of Epoxy/Glass fibre composites. Silicon Carbide reinforcement up to 10% by weight has led to better thermo-mechanical properties of composites above which there exists a barrier leading to decrease in

overall performance of the composite⁷.

Enhancement in the thermo-mechanical properties such as storage modulus and loss modulus is observed because of nanofiller in kenaf Epoxy composites. Nanofillers have resulted in decrease of damping factor compared to that of kenaf/Epoxy composites without filler. Hybridization has improved dynamic behaviour of composites⁸. Enhanced dynamic properties of Epoxy composites are noticed because of hybridization and bonding of fibres⁹.

Dynamic mechanical behaviour of neat resorcinol-formaldehyde, treated *Luffa Cylindrica* and untreated *Luffa Cylindrica* fibre composites are studied. It has been observed that storage modulus of composites has increased with increase in fibre loading. Low loss factor or damping factor is found for the composites with good bonding between *Luffa Cylindrica* fibre and resorcinol-formaldehyde matrix. Addition of fibre has resulted not only in increase of glass transition temperature (T_g) but also its shift on the region of higher temperature for all the different tested composites¹⁰.

Epoxy composites with reinforcement of treated *Luffa Cylindrica* fibres and Silicon Carbide particulates are investigated for mechanical strength and Dynamic Mechanical Analysis (DMA).

2. Materials and Methods

2.1 Materials

Luffa Cylindrica obtained locally are dried under sunlight. After complete drying, the outer covering of the *Luffa Cylindrica* is detached by squashing along the length. The

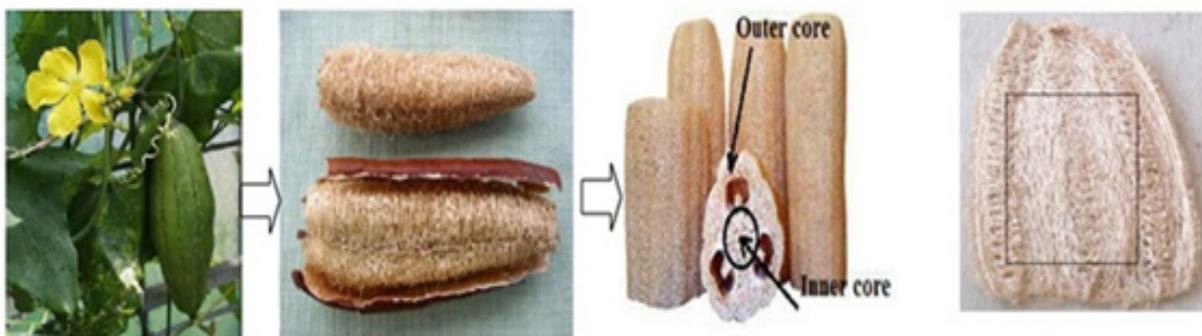


Figure 1. *Luffa Cylindrica* fibre extraction.

ends are cut open with the purpose of removing the seeds and the *Luffa Cylindrica* thus obtained is cut along the length in order to form rectangular shape fibre as shown in the Figure 1.

Silicon Carbide particulates are used as the secondary reinforcement to form *Luffa Cylindrica*/Silicon Carbide reinforced Epoxy hybrid composites. Silicon Carbide particulate of 25µm particle size is obtained from a Private Chemical Ltd, Chennai. The matrix material consists of Epoxy resin LY556 and HY951 hardener.

2.2 Fibre Treatment

Luffa Cylindrica fibre is ligno cellulose with 8 - 22% hemicelluloses, 10 - 23% lignin, 55-90% cellulose and around 3.2% extractives⁵. The presence of unwanted matrix and oil on the surface of the fibre results in poor mechanical strength. The fibres are subjected to alkali treatment to remove unwanted materials and improve interfacial bonding between fibre and matrix through enhanced surface roughness. Treated fibres are normally tested as reinforcement in the composites^{3,11}. This does not indicate influence of treatment on the strength of the fibre. Therefore, testing of treated fibres before reinforcement with the matrix is essential.

Time duration and % NaOH, as alkali treatment parameters, are optimized by Central Composite Design (CCD) of Experimental techniques. Optimal alkali treatment of *Luffa Cylindrica* fibre for 6 hour at 10% NaOH has yielded the highest tensile strength and the fibres are treated accordingly. Temperature of 80°C is used to dry treated fibres for duration of 2 hour in an electrical oven.

2.3 Compositions of Epoxy Hybrid Composites

Highest strength is noticed in Epoxy composites for 2 *Luffa Cylindrica* fibre layers¹². Tensile and flexural strengths of composites reinforced with single and double layer of fibres are provided in Table 1. As can be noticed, two layers of fibres yield the maximum values for both the strengths¹³. Thus, two layers of optimally treated *Luffa Cylindrica* fibres reinforced Epoxy composites are considered for investigation of Dynamic Mechanical Analysis.

Silicon Carbide is added as second reinforcement to study mechanical, visco-elastic and wear behaviour of Epoxy hybrid composites. Range of Silicon Carbide wt% is similar to that used for wear studies where the wt% is according to Central Composite Design (CCD) as it is

Table 1. Tension and flexural test results

	Single fibre layer	Double fibre layer
Tensile strength, (MPa)	20.67	29.90
Flexural strength, (MPa)	51.50	72.17

Table 2. Range of parameters considered for treatment

Parameter	Minimum value	Maximum value
Silicon Carbide, % (A)	0	7
Load, kg (B)	1	3
Distance, m (C)	500	1000

Table 3. Values of parameters at five levels

Parameter	- α	-1	0	+1	+ α
Silicon Carbide, % (A)	0	1.5	3.5	5.5	7
Load, kg (B)	1	1.4	2	2.6	3
Distance, m (C)	500	600	750	900	1000

Table 4. Detailed composition of hybrid epoxy composites

Sl. No	Composition
1	0% Sic filled <i>Luffa Cylindrica</i> Epoxy composites
2	1.5% Sic filled <i>Luffa Cylindrica</i> Epoxy composites
3	3.5% Sic filled <i>Luffa Cylindrica</i> Epoxy composites
4	5.5% Sic filled <i>Luffa Cylindrica</i> Epoxy composites
5	7% Sic filled <i>Luffa Cylindrica</i> Epoxy composites

also a parameter in addition to load and distance. The range of parameters for experimental plan is given in Table 2.

Regular experiments require more number of trials where one parameter at a time is varied. Three parameters (k) of Table 2, each varied at five levels (L) yield total experiments equal to $L^k = 5^3 = 125$. On the other hand, total experiments get reduced by use of Design of Experimental techniques (DOE). Central Composite Design (CCD) which is one of the DOE techniques in Response Surface Methodology (RSM) is used in the present investigation.

Details of values corresponding to five levels of each parameter considered for treatment and composition of Hybrid Epoxy composites are indicated respectively in Table 3 and Table 4.

2.4 Fabrication of Composites

Conventional Hand layup technique is used to produce the *Luffa Cylindrica* Epoxy and *Luffa Cylindrica*/Silicon

Carbide Epoxy hybrid composites. Different compositions of composites are fabricated by adding Silicon Carbide content according to Table 4. Silicon Carbide is added to the Epoxy resin and stirred carefully resulting homogeneous mixture as in Figure 2. This is added to the hardener with 10:1 ratio and is then impregnated with treated *Luffa Cylindrica* fibres. A mould with dimensions of 150mm x 100mm x 5mm with release coat being applied for easy removal of laminate is used. Pre-impregnated fibres are placed in the mould after applying a thin layer of resin. This is repeated for each layer of laminate up to two layers of treated fibres with care being taken in order to avoid formation of voids. Figure 3 represents complete impregnation of fibres which is ensured by applying resin after placing the final layer of fibre. Resin is evenly distributed and air pockets are removed using a roller. A slab of adequate weight is placed to take out excess resin and to apply required pressure as shown in Figure 4. Then the laminate is cured at room temperature for one day. Post curing of laminates is carried out in an oven at 100°C



Figure 2. Mechanical stirring of resin mixture.



Figure 4. Placing pressure weight.



Figure 3. Hand lay-up technique.



Figure 5. Curing of laminates in oven.

and is shown in Figure 5. Samples as per ASTM standards are prepared.

2.5 Dynamic Mechanical Analysis

DMA, which is essentially thermo-mechanical behaviour and is carried out with DMA Q800 instrument for both Epoxy composites and Epoxy hybrid composites.

The tests are carried out at 1Hz frequency with temperature ramped from 26°C to 160°C and heating rate of 1°C/min. The samples are cut according to ASTM D5023 with dimensions of 55x13x5mm³.

3. Results and Discussion

3.1 Storage Modulus

Storage modulus is an indication of stiffness of polymer composites. Figure 6 is the plot of storage modulus of composites with temperature. Composites exhibit higher values of storage modulus in the region of 30°C to 70°C which reveals strong interfacial adhesion relationship between the fibre and matrix representing glassy region. Gradual fall in glassy region followed by severe drop of storage modulus in the range of 70°C to 100°C which

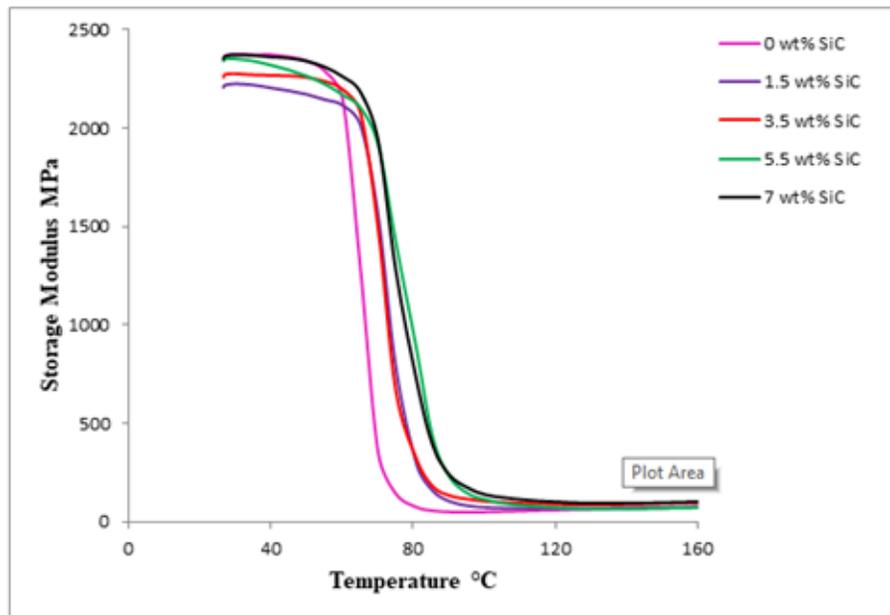


Figure 6. Effect of SiC on storage modulus with respect to temperature at 1Hz.

represents the transition region are noticed. The sudden decline in the storage modulus is due to molecular mobility occurred by softening of composites. The transition will lead to changeover from glassy region to rubbery region.

In rubbery region, the storage modulus of both the composites show degradation leading to nearly zero modulus as a result of lower contribution of fibre to the stiffness at higher temperature. Also increased molecular mobility due to considerable flexibility in molecular chains of the polymer will lead to flatter storage modulus in the rubbery region.

Natural fibres other than *Luffa Cylindrica* have resulted in lower value of storage modulus compared to present investigation. Storage modulus of around 550MPa is noted in Epoxy composites reinforced through 30% by weight of Jute fibres which is more than other three variations of Jute fibre Epoxy composites¹².

3.2 Loss Modulus

Loss modulus signifies the viscid response of polymer composites. Figure 7 represents the variation of loss modulus of composites in regard to temperature at

frequency of 1 Hz. Its value increases up to transition temperature and subsequent gradual decrease with temperature. Beyond 120°C, it is found that the curve tends to zero modulus. From Figure 7, it can be perceived that the loss modulus curve for *Luffa Cylindrica*/Silicon Carbide reinforced Epoxy hybrid composites spreads along a wide range with increase in peak width compared to that of *Luffa Cylindrica* reinforced Epoxy composites. This is due to the fact that incorporation of Silicon Carbide increases the area under the curve. The highest value of peak of loss modulus is obtained for *Luffa Cylindrica* reinforced Epoxy composites without Silicon Carbide particles whereas the lowest value of peak of loss modulus is obtained for 3.5% Silicon Carbide reinforced *Luffa Cylindrica* Epoxy hybrid composites.

Epoxy composites reinforced with *Luffa Cylindrica* and Silicon Carbide particulates have yielded better loss modulus compared to composites prepared with other natural fibres. The highest loss modulus of 70.5 MPa for Epoxy composites with 20% by weight Jute fibres is less than the highest value noticed in the present investigation¹⁴.

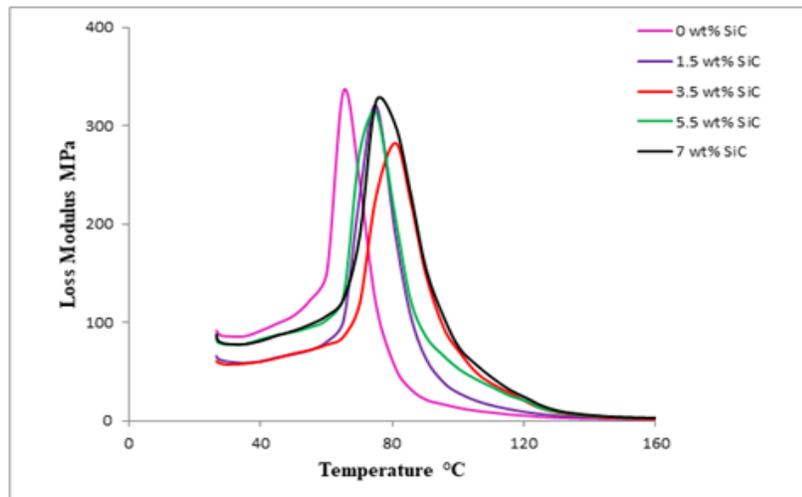


Figure 7. Effect of SiC on loss modulus with respect to temperature at 1Hz.

3.3 Damping Factor ($\tan \delta$)

Figure 8 reveals the variation of $\tan \delta$, the ratio of loss and storage modulus, relating to temperature at frequency of 1 Hz. The change of pattern at the peak represents the transition temperature (T_g) which distinguishes the glassy and rubbery regime. Higher value of damping factor represents the viscous behaviour i.e. non elastic

behaviour whereas lower value of $\tan \delta$ represents elastic behaviour. Figure 8 depicts that the damping ratio for *Luffa Cylindrica* Epoxy composite without Silicon Carbide particles shows a higher value compared to that of *Luffa Cylindrica*/Silicon Carbide reinforced Hybrid Epoxy composites. Presence of Silicon Carbide particles restricts the molecular mobility of polymer segments and hence provide lower damping ratio, whereas the absence

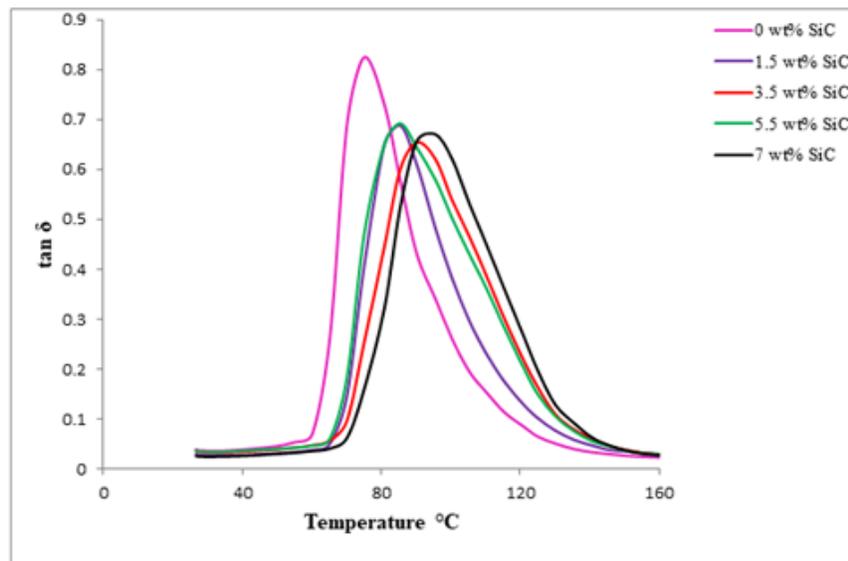


Figure 8. Effect SiC on damping ratio $\tan \delta$ with respect to temperature at 1H.

Table 5. Peak value and transition temperature of damping ratio $\tan \delta$

Composition	Damping ratio $\tan \delta$	Transition temperature (Tg) °C
0% Sic filled <i>Luffa Cylindrica</i> Epoxy composites	0.8257	75.82
1.5% Sic filled <i>Luffa Cylindrica</i> Epoxy composites	0.6952	84.26
3.5% Sic filled <i>Luffa Cylindrica</i> Epoxy composites	0.6542	90.20
5.5% Sic filled <i>Luffa Cylindrica</i> Epoxy composites	0.6925	85.47
7% Sic filled <i>Luffa Cylindrica</i> Epoxy composites	0.6785	95.62

of Silicon Carbide particles lead to free movement of polymer segments resulting in higher damping ratio. The Table 5 represents the peak value of $\tan \delta$ and transition temperature (Tg) for *Luffa Cylindrica* Epoxy composite and *Luffa Cylindrica*/Silicon Carbide reinforced Epoxy hybrid composites. The transition temperature (Tg) represents the thermal stability of polymer composites. It is observed from Figure 8 that 3.5wt% Silicon Carbide reinforced *Luffa Cylindrica* Epoxy hybrid composites has high thermal stability with a Tg value of 93.58°C compared to other variations of *Luffa Cylindrica* Epoxy hybrid composites.

Natural fibres other than *Luffa Cylindrica* and also *Luffa Cylindrica* reinforced with other matrix material than Epoxy have exhibited lower thermal stability compared to present investigation. Glass transition temperature (Tg) of 65.8°C is noticed in Epoxy composites reinforced with 23 vol. % of Coir fibre¹⁵. *Luffa Cylindrica* reinforced with resorcinol formaldehyde has revealed glass transition temperature (Tg) of 40.1°C¹⁰.

4. Conclusions

1. *Luffa Cylindrica* fibres are treated for optimality and then are used as reinforcements with Epoxy to determine mechanical strength.
2. Epoxy composites with double layers of treated *Luffa Cylindrica* fibres have exhibited more values

of tensile and bending strength of 29.90 MPa and 72.17 MPa respectively.

3. Hybrid Epoxy composites with 0% to 7% of SiC are studied for Dynamic Mechanical Analysis (DMA).
4. High storage modulus of around 2300MPa is obtained for Epoxy composites reinforced with *Luffa Cylindrica*/Silicon Carbide.
5. 3.5wt% Silicon Carbide reinforced *Luffa Cylindrica* Epoxy hybrid composites has exhibited better loss modulus compared to other variations of Epoxy hybrid composites.
6. Presence of Silicon Carbide particles has resulted in lower damping ratio and higher thermal stability of 95.62°C.

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