

# Development of Silicon Carbide Reinforced Jute Epoxy Composites: Physical, Mechanical and Thermo-mechanical Characterizations

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**Abstract** The aim of the present study was to investigate the physical and thermo-mechanical characterization of silicon carbide filled needle punch nonwoven jute fiber reinforced epoxy composites. The composite materials were prepared by mixing different weight percentages (0–15 wt.%) of silicon carbide in needle punch nonwoven jute fiber reinforced epoxy composites by hand-lay-up techniques. The physical and mechanical tests have been performed to find the void content, water absorption, hardness, tensile strength, impact strength, fracture toughness and thermo-mechanical properties of the silicon carbide filled jute epoxy composites. The results indicated that increase in silicon carbide filler from 0 to 15 wt.% in the jute epoxy composites increased the void content by 1.49 %, water absorption by 1.83 %, hardness by 39.47 %, tensile strength by 52.5 %, flexural strength by 48.5 %, and impact strength by 14.5 % but on the other hand, decreased the thermal conductivity by 11.62 %. The result also indicated that jute epoxy composites reinforced with 15 wt.% silicon carbide particulate filler presented the highest storage modulus and loss modulus as compared with the unfilled jute epoxy composite.

**Keywords** Needle punch nonwoven jute · Silicon carbide · Mechanical characterization

## 1 Introduction

The development of light weight fiber reinforced polymer composites has been more focused on superior physical, mechanical, thermal, and thermo-mechanical properties by means of adding filler particles into fiber-reinforced composites [1, 2]. Polymeric laminated composites exhibit higher strength-to-weight and stiffness-to-weight ratios as compared to metallic materials. However, these laminated composites do not have strong interfaces between the plies and hence are susceptible to damage loads. This characteristic explains the importance of improving the material properties by modification of the resin/fiber/filler system using adequate modifiers [3, 4]. The properties of jute such as considerable tensile strength, low extensibility and better breathability make it long lasting and reusable as compared to synthetic materials [5]. Jute fiber has been shown to be a potential natural source in a number of durable goods. Studies have been carried out on mechanical properties of untreated (as received) jute fabric reinforced polyester composites [6]. The authors concluded that the optimum ratio of fiber to matrix very much influences the improvement in properties. The physical and mechanical properties can further be modified by incorporation of solid filler material into the matrix body during the composite fabrication. Jawaid et al. [7] proposed that the tensile properties of jute oil palm fiber hybrid composites were increased substantially with increasing the jute fiber content of oil palm epoxy composites. Reinforcing glass fiber and sisal fiber in polypropylene composites enhanced tensile and flexural properties, thermal properties and wear resistance properties of the hybrid composites [8]. Research has been conducted to find the influence of SiC filler addition on physical and mechanical properties and specific wear rate of the glass fiber—SiC filler reinforced epoxy matrix composites [9, 10]. Pawar

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et al. [11] investigated the mechanical and viscoelastic performance of jute fiber reinforced epoxy composites using stone waste (granite powder) as filler material. It was found that tensile and flexural properties are marginally decreased whereas properties such as hardness, impact strength and fracture toughness positively improved due to incorporation of stone waste in the polymer composite. Viscoelastic analysis showed that 16 wt.% of stone waste is the optimum filler content in jute epoxy composites. Varatharajan [12] conducted extensive tensile, flexure and interlaminar tests on glass/polypropylene and glass/polyester composites. Hitchen et al. [13] studied the effect of the fiber length on the fatigue of a short carbon/epoxy composite. They showed that fatigue life is independent of fiber length at any peak strain. Dhieb et al. [14] studied the surface and sub-surface degradation of unidirectional carbon fiber and have given many conclusions concerning sliding in demineralized water, the simplest degradation was detected on sliding in an anti-parallel direction. Shankar et al. [15] have reported that the ultimate tensile strength value maximum is at 15 % and then decreases with increase in fiber starting from 15 to 20 %. They also reported that the flexural strength value decreased from 5 to 10 % fiber (87.31 MPa) and after that the value increased.

The major purpose of this research was to fabricate silicon carbide particulate filled needle punch nonwoven jute fiber reinforced epoxy composites using a hand-lay-up technique and to characterize their various physical, mechanical, and thermal properties, and to study thermo-mechanical behavior via the void content test, water absorption test, tensile strength test, hardness test, impact strength test, fracture toughness test, thermal conductivity test, dynamic mechanical analysis, thermo-gravimetric analysis etc.

## 2 Experimental Materials and Methods

### 2.1 Preparation of Needle Punch Nonwoven Jute Fiber Reinforced Epoxy Composites

The composite was prepared by reinforcing needle punch nonwoven jute fabric mats (areal density 200 gsm) in epoxy

resin (Grade epoxy LY 556, chemical name Bisphenol-A-Diglycidyl-Ether). The epoxy resin was mixed with the corresponding hardener (HY951) in a ratio of 10:1 by weight as recommended by the manufacturer (Huntsman International (India) Pvt. Ltd). Silicon carbide was mixed in the composite in four different weight percentages (0 wt.%, 5 wt.%, 10 wt.%, 15 wt.%) as filler material. The composite materials were fabricated by a simple hand-lay-up technique and compressed under a load of about 50 kg for 24 hrs in the compression molding machine to get proper curing as well as to avoid any void formation in composites.

The designation and composition of the particulate filled jute epoxy composites are presented in Table 1.

### 2.2 Density and Void Fraction

The theoretical density of the particulate filled jute epoxy composite in terms of weight fraction was obtained by using Eq. 1 [16].

$$\rho_{ct} = \frac{1}{(W_f/\rho_f) + (W_m/\rho_m) + (W_p/\rho_p)} \quad (1)$$

Where, the suffix 'ct' indicates composite, 'm' indicates matrix, 'f' indicates fiber' and 'p' indicates particulate filler materials.

The actual density ( $\rho_{ce}$ ) of the composite, however, was determined experimentally by a simple water immersion technique. The volume fraction of voids ( $V_v$ ) in the particulate filled composites was calculated using Eq. 2.

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \quad (2)$$

### 2.3 Hardness

Micro-hardness tests were performed on a Leitz micro-hardness tester (Dublin, Ireland). A diamond indenter, in the form of a right pyramid with a square base and an angle of 136° between opposite faces, was forced into the material under a load F. The two diagonals X and Y of the indentation left on the surface of the material after removal of the load were measured and their arithmetic mean  $L$  was calcu-

**Table 1** Designation and composition of composites

Sl. No.	Composition	Designation
1.	60 wt.% Epoxy+200 gsm jute fiber (40 wt.%)+0 wt% Silicon Carbide	EJS-0
2.	55 wt.% Epoxy+200 gsm jute fiber (40 wt.%)+5 wt% Silicon carbide	EJS-5
3.	50 wt.% Epoxy+200 gsm jute fiber (40 wt.%)+10 wt% Silicon carbide	EJS-10
4.	45 wt.% Epoxy+200 gsm jute fiber (40 wt.%)+15 wt% Silicon carbide	EJS-15

lated. In the present study, the load considering  $F = 24.54$  N and Vickers hardness number were calculated using Eq. 3.

$$H_V = 0.1889 \frac{F}{L^2} \quad (3)$$

and,  $L = \frac{X+Y}{2}$ .

## 2.4 Mechanical Properties

The tensile strength test and three point bending test of silicon carbide filled jute epoxy composites were performed on a Universal Testing Machine (Instron) as per ASTM standard D-3039 method and ASTM D-2344 respectively. The impact strength test was performed according to ASTM D256. Fracture toughness ( $K_{IC}$ ) was evaluated in crack opening mode (mode-I) using the same UTM (Instron) in single end notch bend (SENB) configuration conforming to ASTM D5045. The three different crack lengths to specimen width ratio ( $a/W$ ) were considered as 0.1, 0.3 and 0.5.

## 2.5 Dynamic Mechanical Analysis (DMA)

Dynamic Mechanical Analysis tests (DMA) were performed in compression mode on a Perkin Elmer Pyris-7 Dynamic Mechanical Analyzer (DMA) in three point bending mode conforming to ASTM D4065. DMA was conducted in atmospheric environment at a fixed frequency of 1 Hz, heating rate of 2 °C/min and within a temperature range of 25–120 °C.

## 2.6 Thermo-Gravimetric Analysis (TGA)

The thermal decomposition of the polymer and natural fiber was evaluated by thermo-gravimetric analysis (TGA) conforming to ASTM E1131 using a Perkin Elmer Simultaneous Thermal Analyzer (STA6000). In this study, 11 mg of sample was heated in a high purity nitrogen atmosphere (20 ml/min) from room temperature to 500 °C at a heating rate of 20 °C/min to get the onset temperature of decomposition, mass loss and maximum decomposition peak respectively.

## 2.7 Thermal Conductivity

Thermal conductivity of the silicon carbide filled jute epoxy composites was evaluated by the hot disk method (using Hot Disk TPS 500) according to ISO 22007-2. The conductivity of the composites was calculated by putting a plane hot disk sensor between two pieces of samples. The known electrical power through the samples with flow and temperature increase was measured to evaluate the effective thermal conductivity of the composites.

## 3 Results and Discussion

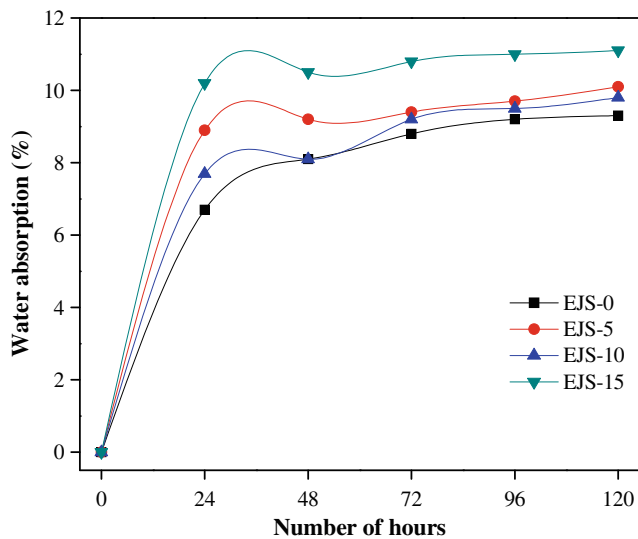
### 3.1 Effect of Voids Content and Water Absorption on Nonwoven Needle Punch Jute Fiber Epoxy Composites

The presence of void content can strongly influence the mechanical and physical properties of fiber reinforced polymer composites. Air or other volatile matter exist in the composite during impregnation of reinforcement into the matrix or during manufacturing of composites [17, 18]. The theoretical and experimentally measured densities along with the corresponding volume fraction of voids are presented in Table 2. Clearly, the increase in silicon carbide filler resulted in an increase in void content. The formation of voids was attributed to the incomplete wetting out of the filler and fibers in the matrix and the presence of entrapped air at the filler matrix interface [19].

Figure 1 shows the water absorption curve of the silicon carbide filled nonwoven needle punch jute epoxy composites. From Fig. 1 it was observed that the unfilled composite indicated a lower water absorption percentage as compared with the silicon carbide filled needle punch jute epoxy composites. Increase in silicon carbide filler content increased the water absorption of the jute epoxy composite. The increase in water absorption was directly related to the void content of the composites [20]. Another reason for the increase of water percentage in particulate filled jute epoxy composites was the presence of a hydroxyl group in the raw jute fiber which absorbed moisture from the environment. The water absorption phenomenon in the case of natural

**Table 2** Effect of void content on nonwoven needle punch jute fiber epoxy composites

S. No.	Composition	Theoretical density	Apparent density	Void content (%)
1	EJS-0	1.253	1.234	1.57
2	EJS-5	1.299	1.271	2.17
3	EJS-10	1.348	1.312	2.68
4	EJS-15	1.401	1.346	3.92



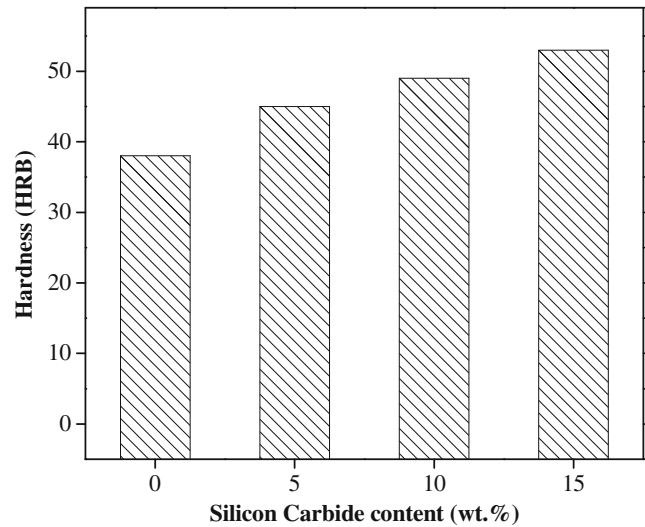
**Fig. 1** Water absorption characteristics curve of unfilled and particulate filled jute fiber epoxy composites

fiber reinforced polymer composites when exposed to moisture environment the hydrophilic nature of any natural fiber causes the fiber to absorb water and then swell. Therefore, automatically micro-cracks are generated and with high cellulose content any natural fiber that absorbed more water creates swelling and finally composite failure occurs [21]. Hence, water molecules actively flow in to the fiber-matrix interface and result in de-bonding of the particulates, fibers and matrix materials [22].

### 3.2 Effect of Hardness on Nonwoven Needle Punch Jute Fiber Epoxy Composites

Figure 2 indicates the hardness of nonwoven needle punch jute fiber reinforced epoxy composites filled with different weight percentages of silicon carbide. The hardness of jute epoxy composites filled with 5, 10 and 15 wt.% of silicon was found to be 45, 49 and 53 Hv respectively whereas the hardness of the unfilled jute epoxy composite was 38 Hv. Hence, it can be concluded that incorporation of silicon carbide increased the hardness of the jute epoxy composite. This increase in hardness was due to the increase in the hard and brittle silicon carbide compared to the flexible epoxy resin.

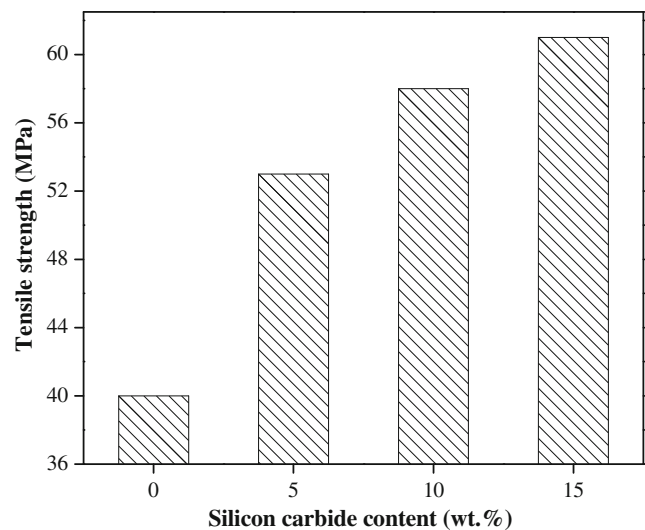
Also, during hardness testing the applied compressive force transferred to the ceramic load. This trend of increase in hardness with the addition of silicon carbide filler in the jute epoxy composite is in agreement with Mantri et al. [23]. Pawar et al. [24] showed similar improvement in the hardness of FRP with inclusion of granite powder as filler material.



**Fig. 2** Effect of hardness for unfilled and particulate filled jute epoxy composites

### 3.3 Effect of Filler Contents on Tensile Strength of Nonwoven Needle Punch Jute Fiber Epoxy Composites

Figure 3 shows the tensile strength graphs of silicon carbide filled nonwoven needle punch jute fiber epoxy composites. It is seen that the tensile strength of the filled composites increased with increase in filler content. The increase in tensile strength with the increase in silicon carbide filler content is due to better and uniform dispersion of fillers in the matrix. The presence of more volume of the resin matrix allowed more filler to disperse uniformly and led to formation of a strong interface between filler and matrix. Similar

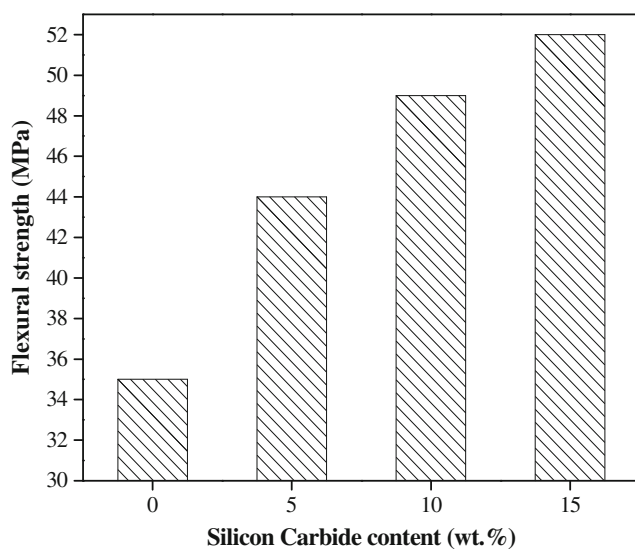


**Fig. 3** Effect of tensile strength for unfilled and particulate filled jute epoxy composites

trends have been reported by Bigg [25] and Fuad et al. [26] for other particulate filled epoxy composites. However, Fan et al. [27] reported the reverse trend of decrease in tensile strength of epoxy composites with increase in clay content. A similar observation has been reported by Aziz et al. [28]. The decrease in tensile strength with the increase in filler content was attributed to the presence of a weak interface between matrix and filler content in the composites.

### 3.4 Effect of Filler Contents on Flexural Strength of Nonwoven Needle Punch Jute Fiber Epoxy Composites

The flexural strength of the silicon carbide filled nonwoven needle punch jute epoxy composites are shown in Fig. 4. It can be seen that the flexural strength of the silicon carbide filled jute epoxy composites increased gradually with the increase in silicon carbide content. The flexural strength of jute epoxy composites filled with 5, 10 and 15 wt.% silicon carbide was 44, 49 and 52 MPa respectively. However, the flexural strength of unfilled jute epoxy composites was 35 MPa. This study is in agreement with the work done by Shinji [29] who concluded that reinforcement of 45 wt.% of kenaf fiber increased the flexural strength of polyester composites by 62 %. The increase in flexural strength was attributed to an extrinsic toughening mechanism due to the presence of long fibers and an intrinsic toughening mechanism due to the presence of silicon carbide filler particles along the path of damage. The increase in flexural strength is due to the jute fiber and hard silicon carbide being able to resist the bending force and the bending stress transfer of particulate filled matrix material resulting in enhancement of bending strength.



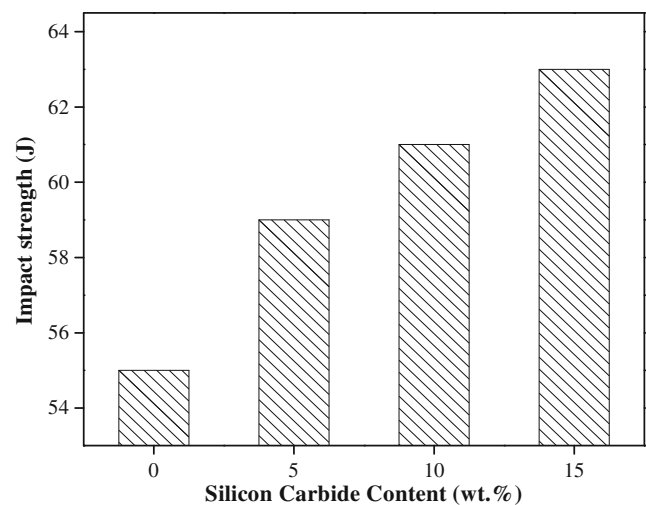
**Fig. 4** Effect of flexural strength for unfilled and particulate filled jute epoxy composites

### 3.5 Effect of Filler Contents on Impact Strength of Nonwoven Needle Punch Jute Fiber Epoxy Composites

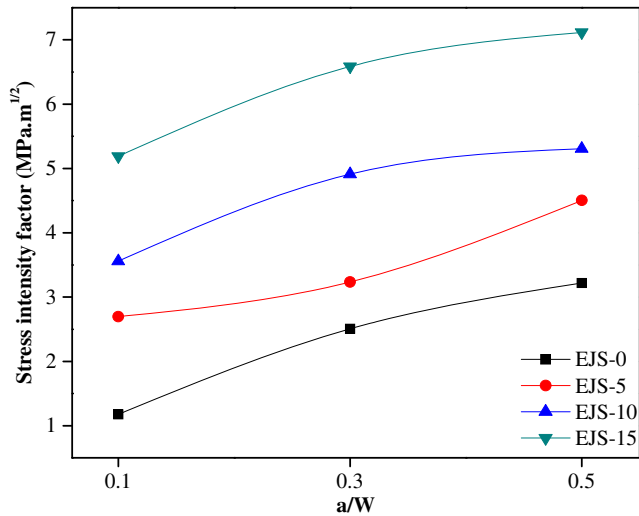
Figure 5 shows the impact strength of jute epoxy composites filled with different weight percentages of silicon carbide filler. The impact strength of jute epoxy composites filled with 5, 10 and 15 wt.% silicon carbide was 59 J, 61 J and 63 J respectively. However, the impact strength of unfilled jute epoxy composites was 55 J. It can be concluded that mixing of silicon carbide as filler in needle punch jute epoxy composites gives better impact energy than an unfilled one. The increase in impact strength with the increase in silicon carbide filler was attributed to energy absorbed by silicon carbide particles present on the plane of fracture resulting in resisting the fracture. The specimen failure starts with the crack propagation due to loss of adhesion between fibers and matrix and then initiates fiber breakage and fiber pullout.

### 3.6 Effect of Filler Contents on Fracture Toughness of Nonwoven Needle Punch Jute Fiber Epoxy Composites

Figure 6 shows the stress intensity factor of silicon carbide filled nonwoven needle punch jute epoxy composites under three different crack lengths evaluated using the initial notch depth method. It can be observed that with the increase in crack length the fracture toughness also increases up to a 0.3 a/W ratio. However, on increase of the a/W ratio further from 0.3 to 0.5 the fracture toughness is marginally decreased. Addition of silicon carbide has a positive effect on the stress intensity factor of needle punch jute epoxy composites. The value of  $K_{IC}$  at a/W ratio of



**Fig. 5** Effect of impact strength for unfilled and particulate filled jute epoxy composites



**Fig. 6** Effect of stress intensity factor for unfilled and particulate filled jute epoxy composites

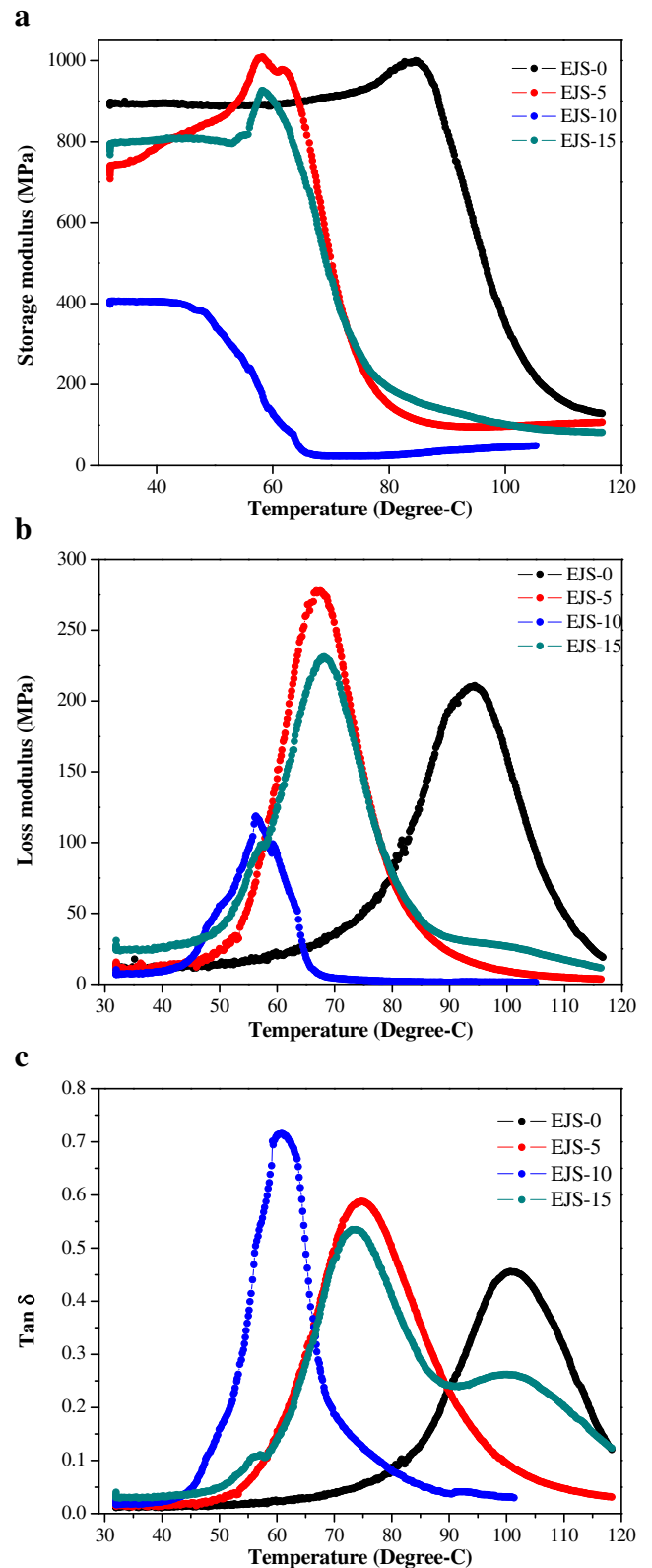
0.5 for the unfilled needle punch jute epoxy composite was  $3.21 \text{ MPa.m}^{1/2}$  which further improved to 4.50, 5.30 and  $7.11 \text{ MPa.m}^{1/2}$  for incorporation of 5, 10 and 15 wt.% silicon carbide respectively. The increase in fracture toughness with the increase in filler loading is due to fiber pull-out, fiber fracture and fiber-bridging respectively. The fracture toughness depends on the number of layers as well as the reinforcing particulate filled materials which arrest the crack propagation direction. The crack propagates through the thickness of the specimen from one fabric layer to the next. Such crack arresting, bridging mechanisms and crack deflection are responsible for the significant enhancement in fracture toughness.

### 3.7 Effect of Filler Contents on Thermo-Mechanical Characterizations of Nonwoven Needle Punch Jute Fiber Epoxy Composites

#### 3.7.1 Dynamic Mechanical Analysis of Nonwoven Needle Punch Jute Fiber Epoxy Composites

Dynamic mechanical analysis is a technique to measure storage modulus, loss modulus and damping factor of the specimen under oscillating load. The storage modulus and loss modulus are monitored against time, temperature and frequency of oscillation. The storage modulus indicates the elastic modulus of the composite in dynamic loading condition and the loss modulus indicates the amount of energy lost due to the friction of polymer chain movement and the damping factor is very sensitive to the structural transformation.

Figure 7a shows the storage modulus of silicon carbide filled nonwoven needle punch jute epoxy composites. The silicon carbide filled jute epoxy composites have



**Fig. 7** a Variation of the storage modulus with the temperature for unfilled and particulate filled jute epoxy composites, b Variation of the loss modulus with the temperature for unfilled and particulate filled jute epoxy composites, c Variation of the Tan delta with the temperature for unfilled and particulate filled jute epoxy composites

lower storage modulus as compared with the unfilled jute epoxy composites. However, among the silicon carbide filled jute epoxy composites, the 15 wt.% silicon carbide filled jute epoxy composite has maximum storage modulus. The increase in storage modulus in the case of particulate filled composites is due to the strong interaction between the hydroxyl group of the epoxy resin, hydroxyl groups of the jute fiber surface and the reinforcing material. As with the increase in filler content in the jute epoxy composites the mobility and deformation of the matrix material reduces and the stress can be transferred from the matrix material to the fiber and filler reinforcement [30]. The storage modulus increases with temperature for all composites due to the increase in molecular mobility in the resin matrix.

Figure 7b shows the loss modulus of the particulate filled and unfilled jute epoxy composites as a function of temperature. Irrespective of filler content the loss modulus increases from a minimum value at room temperature to a maximum value in the vicinity of the glass transition temperature. With further increase in temperature the loss modulus again declines to its lowest value. The loss modulus of the particulate filled jute epoxy composites increased with an increase in silicon carbide content in the composites. The unfilled jute epoxy composites show quite low modulus as compared with the filled composites. The highest loss modulus was observed in the case of 15 wt.% silicon carbide filled jute epoxy composites and the minimum loss modulus was observed in the case of the unfilled jute epoxy composite. The increase in loss modulus of almost all the silicon carbide filled jute epoxy composites is due to the energy losses caused by the rearrangement of molecules and internal friction between the reinforcing hard particulates, fiber and the matrix materials.

Figure 7c shows the damping factor of the particulate filled jute epoxy composites as a function of temperature. In the transition region the damping factor increases with increase in temperature. It reaches a maximum value and further in the rubbery region it falls. It is also observed for all composites that the damping factor value below the  $T_g$  is quite low due to polymeric chains frozen in this temperature range. The highest peak is observed for jute epoxy composites filled with 10 wt. % of silicon carbide whereas for the unfilled of jute epoxy composite the damping factor gives the lowest peak. It can be seen that the peak values of the damping curves increase from unfilled to 10 wt.% silicon carbide filled jute epoxy composite but on increasing up to 15 wt.%, its damping factor decreased. This is attributed to the fact that the enhancement of the storage modulus of the composite could limit the degree of freedom of the polymeric network at the atomic level [31]. The peak value of the damping factor represents non-elastic behavior whereas the lower value of  $\tan \delta$  indicates that the material is elastic in nature. Figure 8c shows that as the

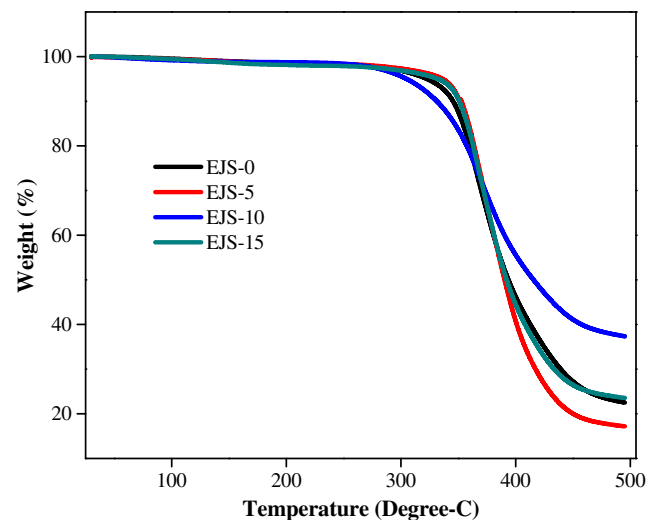
weight percentage of silicon carbide increased from 0 to 10 wt.%, elastic properties decreased with increase in temperature but with further increase in silicon carbide filler content, elastic properties improved.

### 3.7.2 Thermo-Gravimetric Analysis of Nonwoven Needle Punch Jute Fiber Epoxy Composites

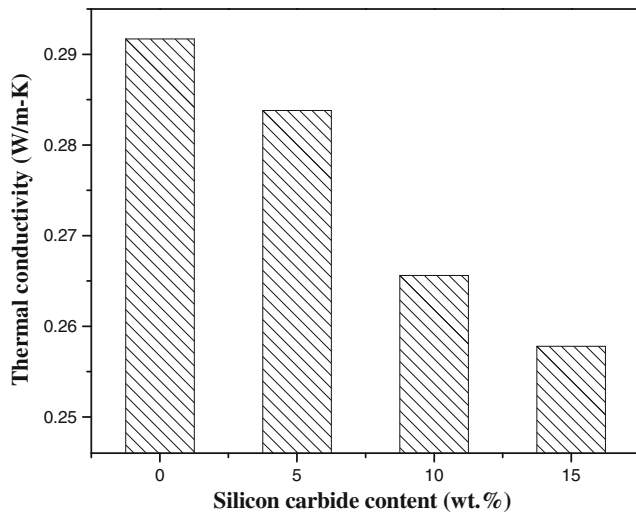
Thermo-gravimetric analysis is used to monitor weight loss of a substance as a function of temperature under a controlled temperature program in a controlled atmosphere. Figure 8 shows the TGA curves of the jute epoxy composites filled with different weight percentage of silicon carbide. The TGA curves indicate that mass loss took place in two different steps. Initially removal of moisture occurred at 30–40 °C. In the range of 30–250 °C, jute fiber epoxy composites indicated similar thermal behavior for all filler content (0–15 wt.%). The second mass loss around 350 °C was due to thermal degradation of the cellulose of the jute fiber and the epoxy [32]. The thermal stability of the jute fiber epoxy composite filled with maximum silicon carbide filler content (15 wt.%) gave the maximum value due to the increase in silicon carbide filler content which exhibited a higher degradation temperature than the epoxy resin and jute fiber.

### 3.8 Effect of Filler Contents on Thermal Conductivity of Nonwoven Needle Punch Jute Fiber Epoxy Composites

Figure 9 shows the thermal conductivities of silicon carbide filled jute epoxy composites. It can be seen that incorporation of silicon carbide decreased the thermal conductivity. As silicon carbide content increased, the thermal



**Fig. 8** Thermo-gravimetric analysis of unfilled and particulate filled jute epoxy composites



**Fig. 9** Thermal conductivity analysis of unfilled and particulate filled jute epoxy composites

conductivity decreased. Hence, the unfilled jute epoxy composite has highest thermal conductivity as compared with the filled composites. The decrease in thermal conductivity with the increase in filler content is attributed to the lower thermal conductivity of the silicon carbide filler as compared to the epoxy resin.

#### 4 Conclusions

The following conclusions are drawn from this study for the unfilled and silicon carbide filled jute epoxy composites:

1. A new set of needle punch nonwoven jute epoxy composites filled with different weight percentages of silicon carbide filler has been fabricated using hand-lay-up techniques.
2. The physical properties such as void content and water absorption were increased by 1.49 and 1.83 % respectively on addition of 15 wt.% silicon carbide particulate filler.
3. The physical properties such as hardness, tensile strength, bending strength and impact strength of jute epoxy composites were increased by 39.47, 52.5, 48.5 and 14.5 % respectively on addition of 15 wt.% silicon carbide particulate filler. However, the thermal conductivity of the jute epoxy composite indicated the reverse trend.
4. Stress intensity factors of the silicon carbide filled composites were also increase with increase in filler content at different ratios of crack lengths and specimen width ( $a/w$ ).
5. Jute epoxy composites filled with 15 wt.% silicon carbide indicated maximum storage modulus, and loss

modulus. The highest damping peak was observed for jute epoxy composites filled with 10 wt.% of silicon carbide.

6. Thermal stability and degradation temperature improved with increase in silicon carbide content in jute epoxy composites.

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