

Research Article

Role of Silicon Dioxide Filler on Mechanical and Dry Sliding Wear Behaviour of Glass-Epoxy Composites

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The mechanical properties and dry sliding wear behaviour of glass fabric reinforced epoxy (G-E) composite with varying weight percentage of silicon dioxide (SiO_2) filler have been studied in the present work. The influence of sliding distance, velocity, and applied normal load on dry sliding wear behaviour has been considered using Taguchi's L_9 orthogonal array. Addition of SiO_2 increased the density, hardness, flexural, and impact strengths of G-E composite. Results of dry sliding wear tests showed increasing wear volume with increase in sliding distance, load, and sliding velocity for G-E and SiO_2 filled G-E composites. Taguchi's results indicate that the sliding distance played a significant role followed by applied load, sliding velocity, and SiO_2 loading. Scanning electron micrographs of the worn surfaces of composite samples at different test parameters show smooth surface, microploaching, and fine grooves under low load and velocity. However, severe damage of matrix with debonding and fiber breakage was seen at high load and velocity especially in unfilled G-E composite.

1. Introduction

Present day industries are experiencing an escalating trend in the applications of particulate and fiber reinforced polymer matrix composites. Some of these applications related to mechanical engineering experience surface interactions with the surroundings as well as with the pairing element. Such applications call for better understanding of the tribological behaviour of the material under study.

Functional fillers are added to the thermoset matrix for improving its physical, mechanical and tribological properties. The modification of the mechanical, and tribological behavior of various polymers by the addition of filler materials has shown a great promise, and hence, it has been a subject of considerable interest. The filler materials include organic, inorganic, and metallic particulates in both macro- and nanolevels. Inclusion of solid lubricants such as graphite, molybdenum disulphide (MoS_2), and polytetrafluoroethylene (PTFE) into polymers has proven effective in reducing the coefficient of friction but their influence on wear resistance is not distinctly clear [1].

Wear rate was reduced with the addition of PTFE into polymers such as polyphenylene sulfide (PPS), polyvinylchloride (PVC), polyarylate (PA), polyoxymethylene (POM), and polyamide (PA) [2]. Bolvari et al. [3] reported that the PTFE filled PPS reduced the wear rate of polymer remarkably. The role of PTFE filler in modifying the tribological behavior of fiber-reinforced composites has been studied and also reported that there is a considerable reduction in wear and the coefficient of friction of aramid fiber reinforced polyamide composites.

Suresha et al. [4] investigated the influence of inorganic fillers like silicon carbide (SiC) and graphite on the wear of the glass fabric reinforced epoxy (G-E) composites under dry sliding conditions. Higher wear volume loss has been recorded with increase in sliding velocity and the coefficients of friction showed an increasing trend with increase in load and sliding velocity. Siddhartha et al. [5] have investigated titanium dioxide (TiO_2) reinforced epoxy functionally graded composites. They concluded that addition of TiO_2 particles into epoxy has a dramatic effect on the flexural strength tensile modulus, and impact strength in comparison

TABLE 1: Properties of epoxy, E-glass fiber, and silicon dioxide.

Property	Epoxy	E-glass fibers	Silicon dioxide
Density (g/cm ³)	1.15	2.54	2.19
Tensile strength (MPa)	110	3400	55
Young's modulus (GPa)	4.1	72.3	70

TABLE 2: Fabricated composites.

Sample code	Matrix	wt.%	Fiber	wt.%	Filler	wt.%
G-E	Epoxy	40	E-glass	60	—	0
5 SiO ₂ -G-E	Epoxy	35	E-glass	60	SiO ₂	5
7.5 SiO ₂ -G-E	Epoxy	32.5	E-glass	60	SiO ₂	7.5

to homogeneous composites. Shi et al. [6] studied the effects of filler crystal structure and shape on the friction and wear properties of potassium titanate whisker ($K_2Ti_4O_9$ whisker, $K_2Ti_6O_{13}$ whisker) and TiO_2 particles filled PTFE composites. They reported that the friction coefficients of various PTFE based composites are weakly dependent on filler shape, but they are more strongly dependent on filler crystal structure. Basavarajappa et al. [7] investigated the tribological behavior of G-E composites with SiC and graphite particles as secondary fillers. A plan of experiments based on Taguchi's design of experiments was adopted to acquire data in a controlled manner. An orthogonal array and analysis of variance (ANOVA) were employed to investigate the influence of process parameters on the wear behaviour of these composites. They concluded that the inclusion of SiC and graphite as secondary fillers increased the wear resistance of G-E composite. Akram et al. [8] reported the mechanical properties of G-E with fillers, namely, aluminum oxide (Al_2O_3), calcium carbonate ($CaCO_3$), silicon dioxide (SiO_2), and lead oxide (PbO), incorporated to the epoxy matrix to improve the mechanical properties. They concluded that the PbO as a filler material into G-E (52% epoxy + 13% PbO + 35% glass fiber) has better tensile properties as compared to composites. However, SiO_2 filler addition (52% epoxy + 13% SiO_2 + 35% glass fiber) showed better torsion and hardness properties. Further, unfilled G-E (65% epoxy + 35% glass fiber) composite showed good energy absorption capability as compared to filler filled G-E composites.

The whole phenomenon of adhesive wear is a complicated matter influenced by different factors such as the properties of the materials coming in contact with each other, and the service conditions also play their part in sliding wear. A comprehensive, overall understanding of adhesion is therefore called for, and there are indeed many studies on the subject [9]. However, most research has investigated only a single dimension of relationship between the different factors involved in wear loss. Such research is valuable, accurate, and detailed but fails to allow any overall evaluation of the cumulative effect of all the factors and their interactions to be extrapolated from them. In view of the above situation, a number of statistical methods have recently been implemented in wear studies [10]. A majority of research studied detailed experimental work, that is, the effect of one factor by

keeping all other factors fixed; this approach is not advisable because in actual environment there will be combined effects of interacting factors influencing the adhesive wear. Hence, in this study an attempt is being made to study main factors effect.

To achieve this, a design of experiments based on the Taguchi method is adopted. This method is advocated by Taguchi and Konishi [11]. Taguchi's technique uses special design of orthogonal arrays to study the entire parameter space with only a small number of experiments. Taguchi's technique also helps in optimizing the critical parameters [12].

2. Materials and Methods

2.1. Materials. Matrix material selected for the present work is epoxy resin (LAPOX L-12) supplied by ATUL India Ltd. Woven glass fabrics made of 360 g/m² E-glass fibers of diameter of about 12 μm have been used as the reinforcing material in all the composites. SiO_2 particles having average particle size of about 10 μm are taken as filler material. The details of the properties of constituent materials and composition of different composite samples are shown in Tables 1 and 2, respectively. The amount of fillers in the composite was kept below 7.5 percentage by weight because an excessive amount of filler makes the molded composite specimen fragile and poor in interfacial adhesion between the laminae.

2.2. Specimen Preparation. The composite slabs were made by conventional hand-lay-up technique followed by compression molding technique as shown in Figure 1. The fabrication was done by mixing the measured amount of neat epoxy resin to a known amount of K6 hardener (Aromatic amines), a low viscosity room temperature curing liquid hardener was used in the present investigation. The epoxy to hardener ratio of 100:12 was maintained. Before layup, a polyester film was used as releasing agent to facilitate easy removal of the composite slab from the mould after curing. Plain weave woven fabrics were cut according to the required size (500 mm \times 500 mm) as shown in Figure 1(a). Then, the woven fabric is dipped into the resin mix. A roller was used to smear the resin mix to impregnate the fabrics (Figure 1(b)). Sufficient

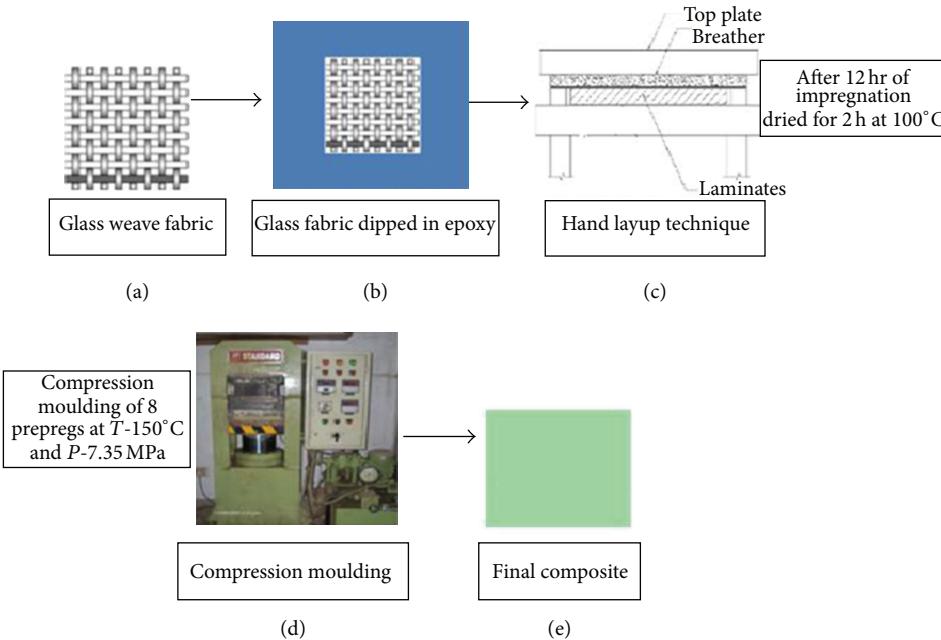


FIGURE 1: Schematic representation of composite fabrication process.

TABLE 3: Experimental parameters.

Test parameter	I	II	III
Filler content (%)	0	5	7.5
Load (N)	20	40	60
Sliding velocity (m/s)	1.047	2.094	3.141
Sliding distance (m)	1500	3000	4500

care was taken during layup to ensure a uniform sample since fabrics have a tendency to clump and tangle together. Eight layers of fabrics were used to obtain laminates of thickness of about 3 mm. Figure 1(c) shows the basic process of hand layup technique. The process involves 12 h of impregnation and drying for 2 h at 100°C followed by compression moulding as shown in Figure 1(d) at a temperature of 120°C and pressure of 7.35 MPa. All the composite samples with and without particulate fillers were fabricated by the same technique. The fillers were mixed thoroughly in the epoxy resin before the glass fiber mats were stacked during layup for particulate filled composites. Specimens of suitable dimensions as per ASTM standards were cut for physical, mechanical, and tribological characterization.

2.3. Mechanical Characterization. Densities of the composites were determined by using a high precision Mettler, Toledo machine Model AX 205 by using Archimedean's principle. Shore hardness of composite samples was also measured using a Shore-D hardness tester (Time Group, Shore-D Durometer TH210). Four readings at different points were taken, and average values were reported.

Tensile testing of polymeric materials is often performed on flat, dog bone-shaped specimens with geometry according

to ASTM D638-10. The mechanical properties of the fabricated microcomposites were studied using the universal testing machine (manufactured by Shimadzu and capacity 50 kN) with wedge grips operated by pneumatic nonshift wedge grips. The tensile test was performed at a cross-head speed of 5 mm/min, and strain (mm/mm) was calculated by dividing cross-head displacement (mm) by the gage length (mm). Five samples were tested for each composition of the composites. A three-point bending technique was adopted for flexural test as per ASTM-D790 standard for all composites. The impact strength was determined using izod impact tester pendulum type (PSI make, India) as per ASTM-D256 specification.

2.4. Tribological Characterization. Dry sliding wear tests were performed using pin-on-disk equipment, conforming to ASTM G99 standards with electronic data acquisition system. Sliding tests were performed at different operating parameters as shown in Table 3. Sliding tests were interrupted at regular intervals for measuring mass loss with an accuracy of 0.1 mg.

Mass loss was converted into volume loss for plotting the wear data using the computed density values based on the composition. Friction force was measured using strain gages mounted on the specimen-loading arm. A minimum of three

TABLE 4: $L_9 (3^4)$ orthogonal arrays.

L_9	Filler content (%)	Load (N)	Sliding velocity (m/s)	Sliding distance (m)
1	0	20	1.047	1500
2	0	40	2.094	3000
3	0	60	3.141	4500
4	5	20	2.094	4500
5	5	40	3.141	1500
6	5	60	1.047	3000
7	7.5	20	3.141	3000
8	7.5	40	1.047	4500
9	7.5	60	2.094	1500

tests were conducted for each set of operating conditions, in order to ensure consistent test results, within 8% variation.

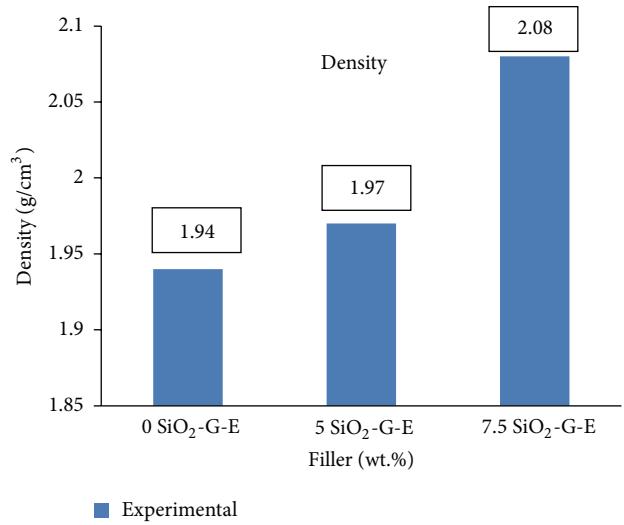
2.5. Design of Experiments by the Taguchi Technique. The present study consists of 3 levels of 4 operating parameters as shown in Table 4. Based on these experimental conditions, Taguchi's $L_9 (3^4)$ orthogonal array is applied in the present study. Table 4 shows the combination of operating parameters selected as per L_9 orthogonal array. With four parameters and three levels for each, a full factorial experiment would require $3^4 = 81$ runs, whereas Taguchi's factorial experiment approach reduces the requirement to nine trials.

3. Results and Discussion

3.1. Influence of Fillers on Mechanical Properties

3.1.1. Density. The experimental values of density are illustrated in Figure 2. It can be observed that addition of SiO_2 filler has increased the density of the G-E composites as compared to unfilled ones. This is due to the fact that addition of high density SiO_2 into G-E increased the density of G-E composite.

3.1.2. Hardness. The hardness values of unfilled and SiO_2 filled G-E composites are shown in Figure 3. It can be seen that Shore-D hardness is higher for SiO_2 filled G-E composites as compared to that of unfilled G-E samples. Further, the hardness is found to increase with the increase in filler loading. The increase in SiO_2 loading results in an increase in brittleness of the composite and hence the hardness. Particulate filled G-E composites with sufficient surface hardness are resistant to in-service scratches that can compromise fatigue strength and lead to premature failure. Therefore, under an indentation loading, microparticles would undergo elastic rather than plastic deformation, as compared to unfilled G-E composites. The improvement in hardness with incorporation of filler can be explained as follows: under the action of a compressive force, the thermoset matrix phase and the brittle fiber and filler phase will be pressed together, touch each other, and offer resistance. Thus the interface can transfer load more effectively although the interfacial bond may be poor. This results in enhancement of hardness of SiO_2 filled G-E composites.

FIGURE 2: Density of SiO_2 filled G-E composites.

3.1.3. Tensile Strength. Tensile test data are illustrated in Figures 4 and 5. The test data indicate that there is marginal decrease in tensile strength of composites, with increase in SiO_2 filler loading. Generally, the particulate fillers increase the stiffness and decrease the tensile strength depending on the type of filler used in the fabrication of composites. Some of the fillers showed improved tensile strength as well as tensile modulus of G-E composites. However, in the present case, the SiO_2 filler reduced the tensile strength and increased the stiffness of the G-E composites.

3.1.4. Flexural Strength. The flexural test data are illustrated in Figure 6. It can be observed that the flexural strength values are increased with increase in SiO_2 filler content in G-E composite. This can be attributed to improved adhesion among the various phases like glass fiber, SiO_2 filler and epoxy matrix.

3.1.5. Impact Strength. The impact strength of unfilled and SiO_2 filled G-E composites is shown in Figure 7. The test results indicate that increasing trend in the impact strength SiO_2 loading. This is due to uniform dispersion of SiO_2 filler loading in G-E composite.

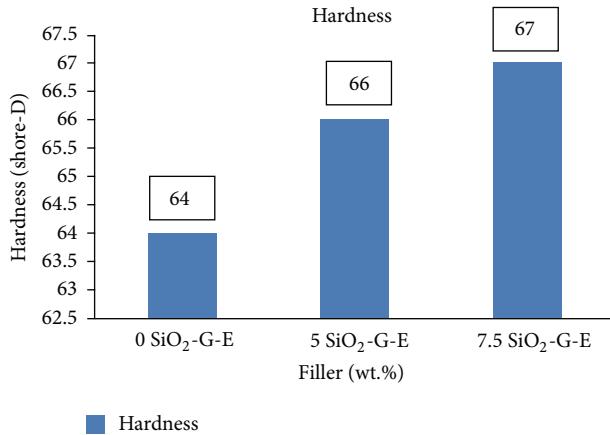
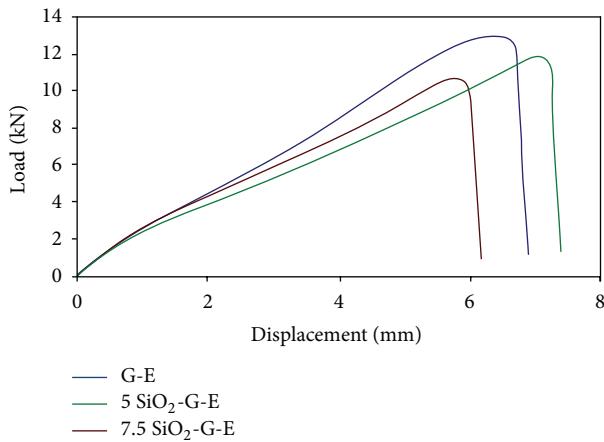
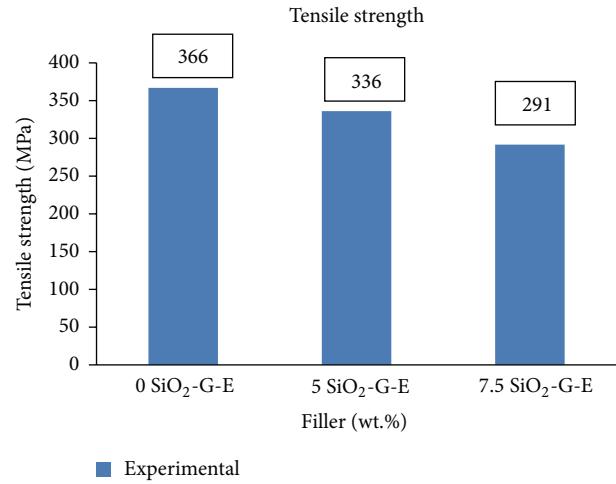
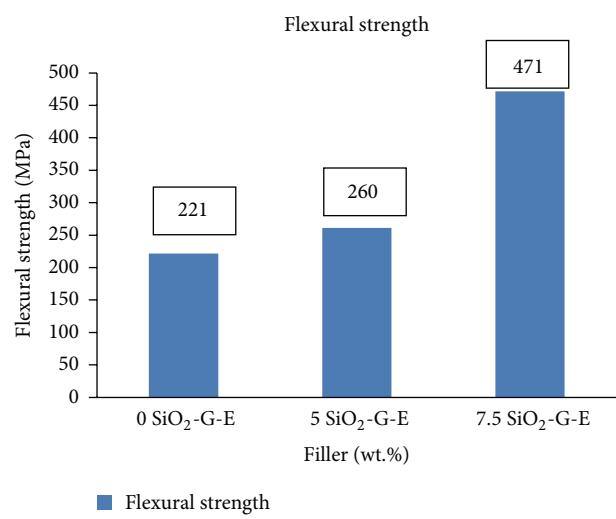
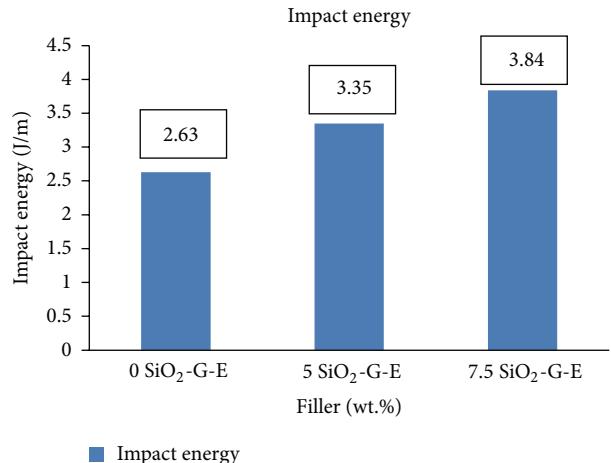
FIGURE 3: Hardness of SiO₂ filled G-E composites.

FIGURE 4: Typical load displacement curves.

3.2. Fractographic Analysis. Figures 8(a), 8(b), 9(a), and 9(b) show the scanning electron micrographs of the fractured surfaces of G-E and SiO₂ filled G-E composites, respectively.

The photomicrographs reveal that the fracture is due to delamination between the layers of the composite samples as well as due to fiber-pullout (Figure 8(a)). For G-E sample, the fracture is ductile-brittle and this can be due to the plastic deformation of the matrix after fiber-matrix debonding (Figure 8(b)). It is also evident from the clean fibers on fractured surfaces. Other important failure mechanisms of composites such as fiber-matrix debonding (marked "a"), fiber fracture (marked "b"), and cohesive resin fracture (marked "c") can also be observed from the microphotograph (Figure 8(b)). Generally, matrix fracture is found to initiate at the surface of the fibers as indicated by the direction of river lines (marked by an arrow) and propagates into the resin on either side, where cracks extend from the surfaces of adjacent fibers simultaneously.

SEM characterization of the SiO₂-G-E fractured surface shows (Figures 9(a) and 9(b)) better adherence of the fibers as well as SiO₂ particles to the matrix (Figure 9(b)), which is a qualitative indication of greater interfacial strength. Disorientation of transverse fibers, fiber bridging, fibers pull

FIGURE 5: Tensile strength of SiO₂ filled G-E composites.FIGURE 6: Flexural strength of SiO₂ filled G-E composites.FIGURE 7: Impact strength of SiO₂ filled G-E composites.

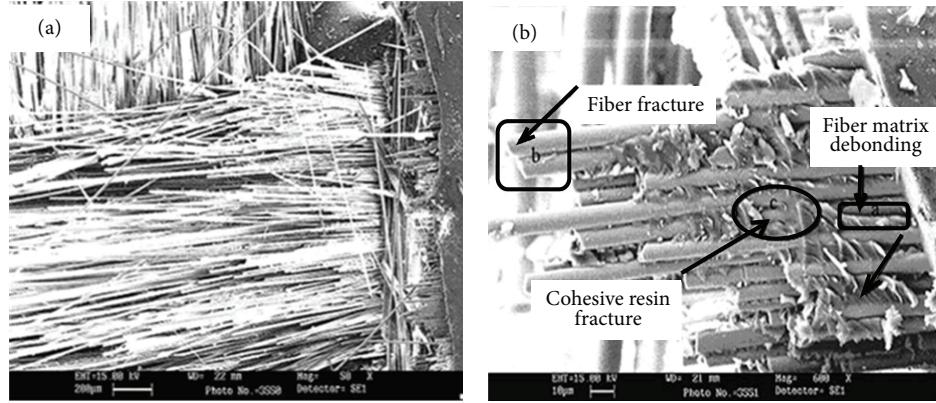
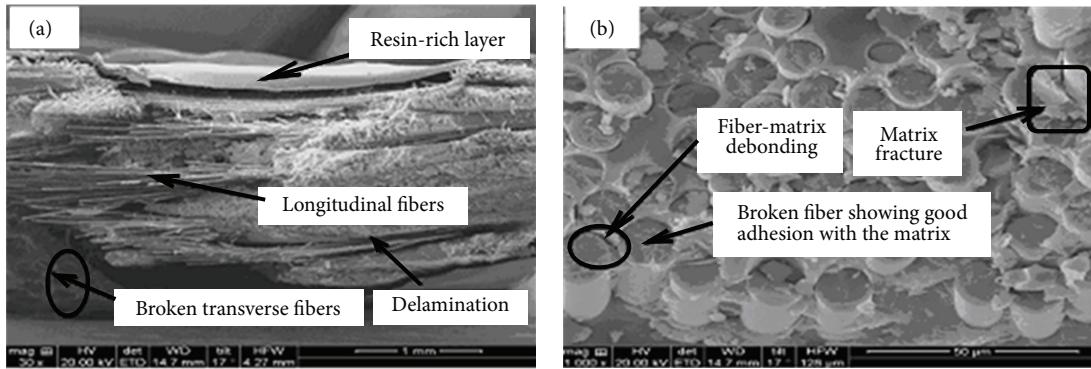


FIGURE 8: SEM pictures of tensile fractured surfaces of unfilled G-E composites.

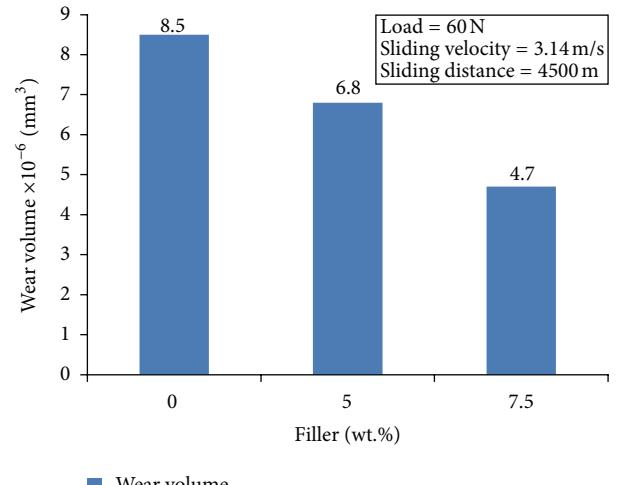
FIGURE 9: SEM pictures of tensile fractured surfaces of 7.5% SiO₂ filled G-E, (a) longitudinal direction, (b) transverse direction.

out (Figure 9(a)), and normal fracture of longitudinal fibers, matrix debris, and matrix cracking (Figure 9(b)) can also be observed from the micrographs. The improvement reported in terms of mechanical properties of the composites tested is mainly due to the enhancement of adhesion or interfacial interactions among the fibers, matrix, and SiO₂ filler.

3.3. Tribological Behaviour

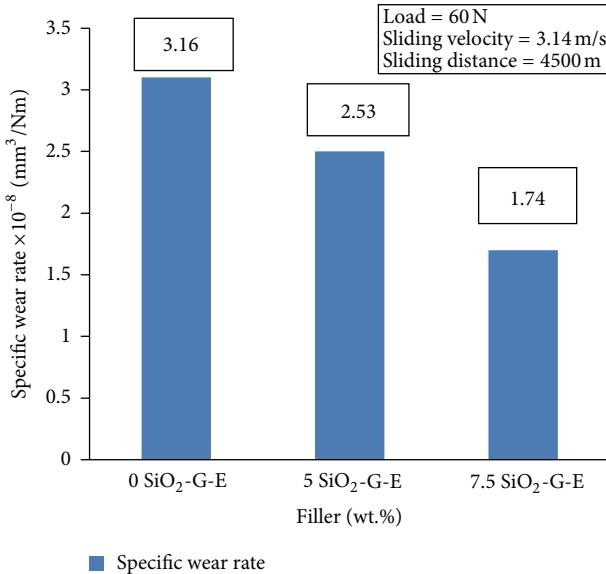
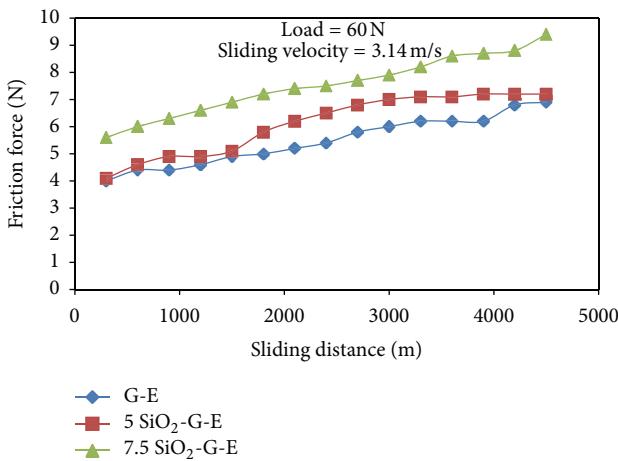
3.3.1. Influence of Filler Content on Tribological Characteristics. G-E composites were prepared with SiO₂ contents of 5 wt.% and 7.5 wt.%, and tribological behavior of the G-E composite was studied to assess the influence of SiO₂ and compare with the unfilled G-E composites. Figure 10 shows the dry sliding wear volume loss of different composites under the test conditions used in the present study. It can be observed from the test results that the wear volume loss of the samples decreases with increase in filler loading.

Specific wear rate of SiO₂ filled G-E composite is studied with respect to SiO₂ loading (Figure 11). At the normal load of 60 N and for sliding velocity of 3.14 m/s, the specific wear rate of G-E is $3.16 \times 10^{-8} \text{ mm}^3/\text{Nm}$. The specific wear rate decreases on addition of SiO₂. It decreases to the values $2.53 \times 10^{-8} \text{ mm}^3/\text{Nm}$ and $1.74 \times 10^{-8} \text{ mm}^3/\text{Nm}$ for the SiO₂ loading of 5 and 7.5 wt.%, respectively. The wear loss decreases

FIGURE 10: Wear volume of SiO₂ filled G-E composites.

significantly with addition of SiO₂ as compared to unfilled G-E composites. The incorporation of hard particles like TiO₂, SiO₂, ZrO, SiC, and Al₂O₃ to polymer matrix has led to better enhancement in wear resistance [13–15].

The improvement in wear resistance is due to the presence of fine SiO₂ particles (10 μm) uniformly dispersed in epoxy matrix. The SiO₂ particles dispersed in the epoxy acts as a

FIGURE 11: Specific wear rate of SiO_2 filled G-E composites.FIGURE 12: Frictional force of SiO_2 filled G-E composites.

barrier and also prevent large scale of fragmentation of epoxy matrix.

This behavior is clearly observed from worn surface pictures (Figures 13 and 14). The wear of unfilled G-E seems to be slightly high with some material pullout. The asperities in the steel counter face easily remove the material from the soft polymer matrix by cutting action. The wear surface of SiO_2 filled G-E is smooth and fine and no material pullout is noticed. The SiO_2 particles also act as secondary reinforcement, bear the load, and reduces wear rate. Hardness contribution also plays a vital role in wear property improvement as reported elsewhere [13–15].

The frictional force of SiO_2 filled G-E composites is studied and compared with unfilled G-E, and the data are shown in Figure 12. The frictional force of unfilled G-E is 4 N and increases with increasing sliding distance. However, it increases on addition of SiO_2 to the value of 4.2 N and 5.8 N for the SiO_2 loading of 5 and 7.5 wt.%, respectively.

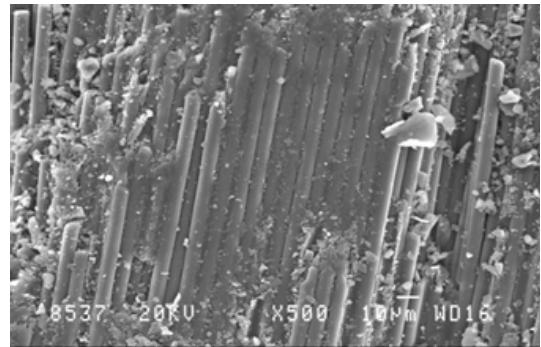
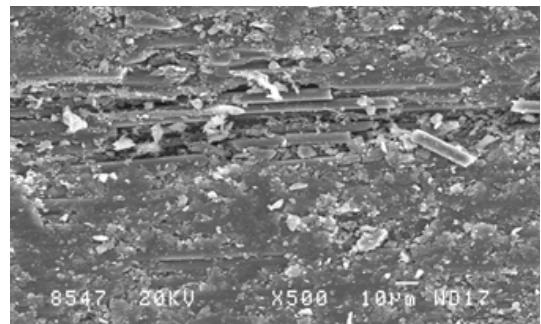


FIGURE 13: SEM of worn surface of G-E material.

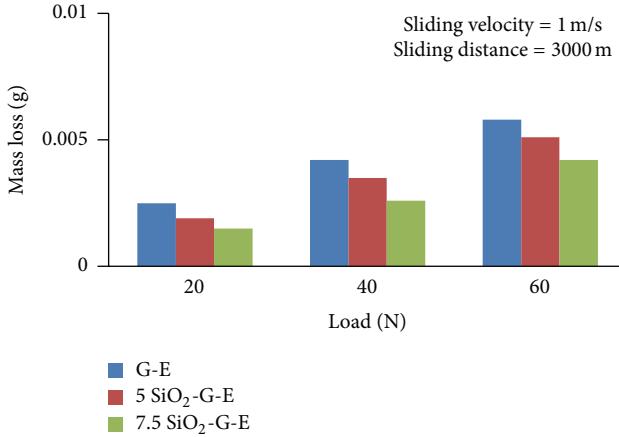
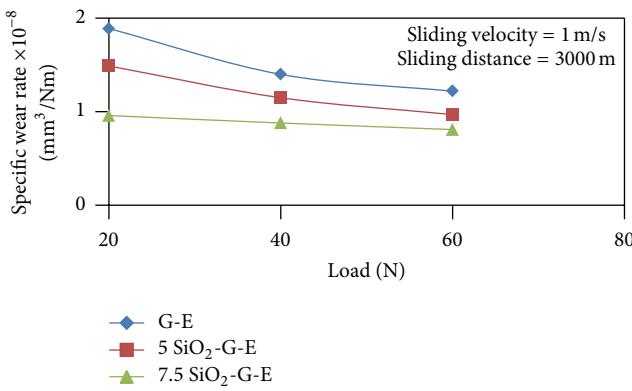
FIGURE 14: SEM of worn surface of 7.5 SiO_2 G-E Material.

The frictional force of unfilled G-E is far better compared to the SiO_2 filled G-E composites. In microcomposites, the increase in frictional force with increase in SiO_2 loading is due to bigger particle size and irregularity in shape. During sliding at contact surface, the frictional force generated might have pulled out SiO_2 particles along with wear debris. These pulled out particles that remain on the surface of the sample might have acted as third body and avoid the direct contact of the sample with the counter surface resulting in increased frictional force.

3.3.2. Worn Surface Morphology. Figure 13 indicates fiber breakage, microcracks, disorientation of fibers, and higher damage to the matrix as compared to fibers. Figure 14 indicates higher damage to the matrix and less fiber breakages. Microcracking of the epoxy matrix and higher debris formation consisting of SiO_2 and broken fibers can also be observed.

3.3.3. Effect of Load on Mass Loss of Composites. Figure 15 shows typical variation of wear loss with normal load. There is an increasing trend of wear loss with increase in load, for obvious reasons. Similar trend has been observed under different operating conditions.

3.3.4. Specific Wear Rate. Figure 16 indicate decreasing trend of specific wear rate with increasing normal load. This can be attributed to flattening of asperities at higher loads, thereby increasing the contact area between sliding surfaces.

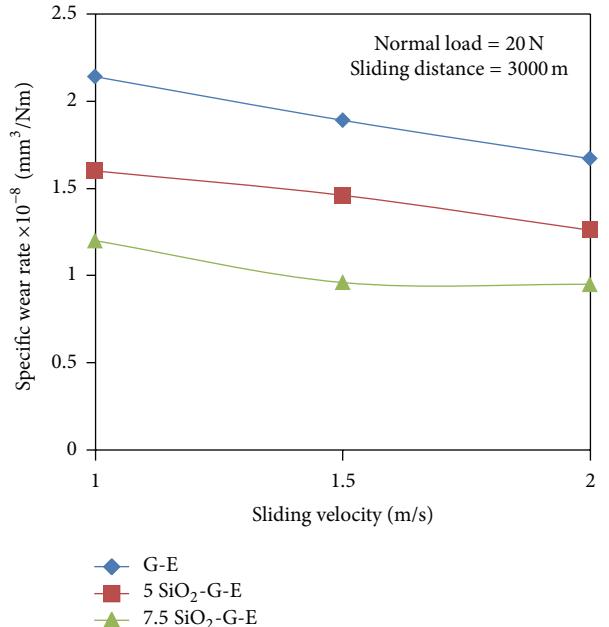
FIGURE 15: Mass loss of SiO₂ filled G-E composites.FIGURE 16: Specific wear of SiO₂ filled G-E composites.

Increased contact area causes decreased contact pressure, resulting in reduction in wear rate. Similar trend has been observed under different operating conditions.

3.3.5. Influence of Sliding Velocity on Specific Wear Rate. Figure 17 represents the variation of specific wear rate with sliding velocity. Specific wear rate is observed to decrease with increase in sliding velocity, which can be attributed to the formation of transfer film on the counter body, due to increased temperature at higher sliding velocity. Formation of transfer film would result in decreased wear rate at higher sliding velocities. Similar trend has been observed under different operating conditions.

3.4. Analysis of Wear by the Taguchi Technique. The parametric influence on dry sliding wear behaviour of the composite test specimen has been determined by adopting the Taguchi technique. Experiments have been conducted as per standard L₉ orthogonal array, using 3 levels for each of 4 operational parameters, that is, filler content, load, sliding velocity, and sliding distance. The details of orthogonal array along with the output parameter, that is, mass loss, is as shown in Table 5.

The influence of different operating parameters on wear loss is assessed based on the S/N ratios, which indicated in Figure 18.

FIGURE 17: Specific wear rate of SiO₂ filled G-E composites.

In order to corroborate the significance of operational parameters, that is, filler content, load, sliding velocity, and sliding distance, analysis of variance (ANOVA) is performed on experimental data. The ANOVA allows analyzing the influence of each variable on the total variance of the results. Table 6, shows the results of ANOVA for the wear of the test samples. This analysis was performed with a level of confidence of 95%. From Table 6 it is evident that sliding distance plays a significant role followed by load, velocity, and filler material percentage.

4. Conclusions

Increased usage of polymeric composites for tribological applications has necessitated investigation to study the behaviour of these materials under various operating conditions. The present investigation has been carried out to study the influence of SiO₂ filler on the mechanical and dry sliding wear behaviour of G-E composites. The following conclusions can be drawn based on the results obtained from the investigations.

- (1) The addition of the SiO₂ filler material has resulted in increased mechanical properties like density, hardness, flexural strength, and impact strength of the composite structure.
- (2) Results of sliding wear tests indicate increase in wear volume with increase in sliding distance, load, and sliding velocity.
- (3) The addition of SiO₂ filler material is found to decrease the wear volume and specific wear rate of different composites. Increase in percentage of SiO₂ filler has resulted in better sliding wear resistance

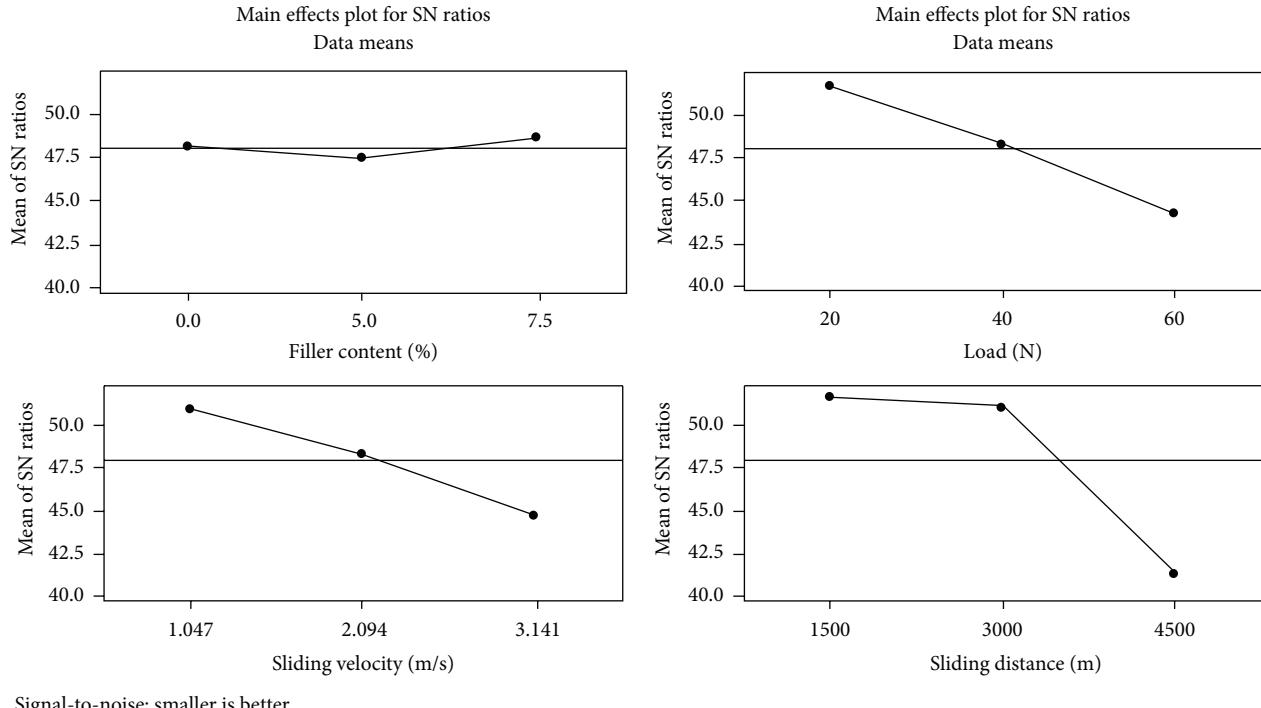


FIGURE 18: Main effect plot for signal-to-noise ratio.

TABLE 5: Standard orthogonal L₉ array with output result.

Experiments	Filler content (%)	Load (N)	Sliding velocity (m/s)	Sliding distance (m)	Wear loss (g)
1	0	20	1.047	1500	0.0012
2	0	40	2.094	3000	0.0026
3	0	60	3.141	4500	0.0195
4	5	20	2.094	4500	0.0058
5	5	40	3.141	1500	0.004
6	5	60	1.047	3000	0.0033
7	7.5	20	3.141	3000	0.0025
8	7.5	40	1.047	4500	0.0057
9	7.5	60	2.094	1500	0.0037

TABLE 6: Analysis of variance table for wear loss.

Source	DF	Seq SS	Adj SS	Adj MS	F	Contribution % (P)
Load (N)	2	86.177	86.177	43.089	48.39	24.45
Velocity (m/s)	2	58.967	58.967	29.484	33.11	16.73
SD (m)	2	205.52	205.52	102.76	115.41	58.82
Error	2	1.781	1.781	0.890		
Total	8	352.44				

due to better mechanical properties of the composite materials.

- (4) Taguchi's test data indicate that sliding distance plays a significant role followed by load, sliding velocity, and filler content.
- (5) Microscopic analysis of the worn out surfaces of different composite samples at different operating

parameters indicate, smooth surface and microploughing under lower loads and speeds, whereas severe damage of matrix with debonding and fiber breakage at higher loads and speeds.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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