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ORIGINAL PAPER

Mechanical and Abrasive Wear Studies on Biobased Jatropha Oil Cake Incorporated Glass–Epoxy Composites

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Abstract An investigation was made to evaluate the effect of the incorporation of jatropha oil cake (JOC) alone and in combination with silicon carbide (SiC) on the mechanical and tribological wear behaviour of glass fabric–epoxy (GE) composites. A vacuum-assisted resin transfer moulding (VARTM) technique was employed to obtain a series of GE composites containing different fillers viz., silicon carbide, jatropha oil cake and a mixture of SiC and JOC. The effect of different loads (22 and 32 N) and abrading distances from 135 to 540 m on the performance of the wear resistance of the composites were measured. The mechanical properties such as tensile behaviour and hardness of the composites were evaluated. A linear relationship was found between the wear volume loss and the abrading distances. The JOC filled GE composite exhibited a lower specific wear rate by 6 and 10% at 540 m abrading distance for a load of 22 and 32 N, respectively, as compared to that of unfilled GE composites. The worn surface features of unfilled and filled GE composites were examined using scanning electron microscopy (SEM).

Keywords Abrasive · Three-body abrasion · Jatropha · Glass–epoxy · Silicon carbide · Wear

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Introduction

The utilisation of biobased materials in the fabrication of composites is expected to reduce the demand and dependency on petroleum based raw materials. Fibre-reinforced polymer matrix composites have received a considerable amount of attention in recent years due to high specific strength, specific modulus and wear resistance as compared to metal based counterparts [1]. The equipment used to handle coal and cement/concrete are typical examples of abrasion during their service. The fibre-reinforced polymer (FRP) composite parts such as gears, seals, bushes, cam, etc. have been used in automotive and aerospace applications [2, 3]. The thorough understanding of the tribological properties of composites have become very important [4, 5] as more than 90% failure of these composite parts (in actual service) are due to the tribological environment. Tribology is the science and technology of interacting surfaces in relative motion. It includes the study and application of the principles of friction, lubrication and wear [6]. The tribological interactions of a solid/composite surface's exposed face with interfacing materials and the environment may result in a loss of material from the surface. The process leading to loss of material is known as "wear". Major types of wear include abrasion, adhesion (friction), erosion and corrosion. Abrasive wear is caused by hard particles that are forced and moving along a solid surface [7]. A number of papers have been published in the past on the abrasive wear mechanism of polymers and polymer composites [8–12].

The improvement of the tribological properties of a polymer with the incorporation of fibres/fillers is well known. The incorporation of fibre has showed both positive and negative results on the tribological properties of a polymer [2, 4, 13, 14]. The reason to incorporate a filler into a polymer is twofold; (a) first to improve the tribological, mechanical and thermal properties and (b) to reduce the cost of the final product. In the last two decades, various filler and fibre materials have emerged as a subject of extensive research. Since 1893, silicon carbide (SiC) particulate filler has been mass produced to be utilised as an abrasive wear resistant material. SiC has been extensively used in abrasive machining processes such as grinding, honing, water-jet cutting and sandblasting due to its high hardness [15]. Suresha et al. [16] in a study concluded that enhanced wear resistance can be obtained with the incorporation of SiC filler into glass fabric–epoxy (GE) composites. In a study, it was revealed that, the type and quantity of graphite filler found to decide the coefficient of friction of GE composites with an increase in sliding velocity for a fixed load [17]. In a comparative study, Pihtili [18] showed that, the glass–epoxy composite possess higher strength and lower wear as compared to glass–polyester resin composites. Basavarajappa et al. [19] showed that, the incorporation of graphite filler in GE composite exhibits lower weight loss and its value was found to decrease with increasing the graphite filler content in the composites.

The high cost of conventional fillers dictated the need for alternative low cost fillers to improve the tribological performance of the composites. Few researchers have investigated the possibility of the utilisation of low cost fillers to improve the tribological performance. Biswas and Satapathy [20] evaluated the tribological performance of red mud (an industrial waste generated during the production of alumina from Bauxite) GE composites using Taguchi experimental design. The authors have concluded that, the utilisation of red mud filled GE composites gives a better wear performance compared to the unfilled ones. A by-product called cenospheres obtained during coal combustion at thermal power plants have been used as a filler in GE composites by Suresha et al. [21]. The mechanical and three-body abrasive wear behaviour of 5 and 10% (w/w) biobased coleus spent (CS) filled and unfilled semi interpenetrating polymer network composites of unsaturated polyester/polymethyl methacrylate (80/20) composites have been studied by Syed et al. [22]. The observed wear resistance was high in filled composites as compared to unfilled ones.

Jatropha oil cake (JOC) is a left over by-product after the mechanical extraction of oil from jatropha seeds. The extracted oil has been used for the production of the alternative fuel known as biodiesel. The approximate yield of JOC is about 500–600 g/kg of jatropha seeds [23]. The reported protein content of JOC was higher than that of soybean oil cake or meal [24]. The JOC was found to contain 58% crude protein, 6.27% crude fibre, 6.57% acid detergent fibre, 8.71% neutral detergent fibres and 0.18% acid detergent lignin. The reported higher ash content (9.82%) of JOC is an indication for the presence of large amounts of minerals [24] which is an added reason to investigate this material for tribological applications. The investigations of Rao et al. [25] revealed that the composites derived from epoxy resin and protein rich poultry feather fibre exhibit better erosive wear resistance and reduced mechanical properties. The abrasive wear behaviour of wood flour (WF) filled epoxy composites was investigated by Dwivedi and Chand [26]. The authors noticed that the composite containing 40 wt% WF exhibited the lowest specific wear rate in abrasive wear mode. Furthermore, the composite containing 20 wt% WF exhibited the lowest specific wear rate in sliding wear mode. The results of Bhuyan et al. [27] provided some insight into the effectiveness of using biobased spent germ filler (a by-product from a wet ethanol production plant) filled thermosetting polymer composites as potential tribomaterials. Being non-edible and renewable, jatropha oil has a strong potential in the production of biodiesel. It was reported in a literature [23] that, the biodiesel production from jatropha oil becomes profitable provided its various parts and the by-products are marketed as valuable products. In the coming years, the utilisation of biodiesel as an alternative to petroleum based diesel due to increased environmental awareness and depletion of fossil fuel resources is expected to generate massive amount of JOC. Hence, the present research is aimed to evaluate JOC as a material in GE composites for tribological applications.

Experimental

Materials

Woven E-glass woven fabric having a weight of 360 g/m^2 was obtained from M/s. Reinforced Plastic Industries, Bangalore, India. Bifunctional epoxy resin (LY 5052) and room temperature curing cyclo aliphatic amine (HY 5052) hardener was obtained from M/s. Huntsman Advanced Materials Mumbai, India. The epoxy resin to hardener ratio was 100:38 w/w. The viscosity and specific gravity of LY 5052 at 25 °C was 1,000–1,500 mPa s and 1.17 g/cm^3, respectively. The viscosity and specific gravity of HY 5052 at 25 °C was 40–60 mPa s and 0.94 g/cm^3, respectively. The commercially available silicon carbide powder having a particle size of 25–40 µm was obtained from M/s. Silicarb Recrystalized Pvt. Ltd., Bangalore. Jatropha oil cake obtained after the extraction of oil from the seeds in our laboratory (at the National University of Malaysia) was dried and ground prior to use. The particle size of the JOC was in the range of 80–90 µm. The residual oil content present in the JOC was 3–5%.

Fabrication of Composites

The composite fabrication involves three steps: (a) mixing of the epoxy resin and filler using a mechanical stirrer, (b) mixing of the curing agent with the filled epoxy resin, and (c) fabrication of composites. In the first step, a known quantity of filler was mixed with epoxy resin using a high speed mechanical stirrer to ensure the proper dispersion of filler in the epoxy resin. In the second step, the hardener was mixed into the filled epoxy resin using a mechanical stirrer. The ratio of epoxy resin to hardener was 100:38 on a weight basis. In the last step, the epoxy resin was manually smeared onto the glass fabric and the resultant composites were fabricated using the VARTM process as described elsewhere [28]. The composites were cured at room temperature under a pressure of 14 psi for 24 h. The glass fibre: matrix (epoxy): filler ratio was 60:34:6. The unfilled glass epoxy composites were designated as GE, SiC- and the JOC filled GE composites were designated as GE + SiC and GE + JOC, respectively. The composite containing both JOC and SiC in a weight ratio of 3:3 was designated as GE + SiC + JOC. The specimens (200 × 200 × 2.5 mm) from the composites were cut using a diamond tipped cutter.

Techniques

The tensile behaviour of the composites was evaluated using an Instron universal testing machine (UTM, UK) as per ASTM D 3039. The test was conducted at a cross head speed of 10 mm/min. The surface hardness (shore D) of the composites were measured as per ASTM D 2240 specification using a Durometer.

The modified dry sand/rubber wheel abrasion test set up as per ASTM G-65 was used to conduct the three-body abrasive wear experiments The surfaces of the samples were cleaned using a soft paper soaked in acetone before the test. The weight of the specimen was recorded using a digital electronic balance before being mounted on the sample holder. The wear loss was calculated from the difference between the initial and final weight of the specimen. A minimum of three trials were conducted to ensure the repeatability of the results. Silica sand having angular shape (53–75 μm) was used as the abrasive material in this study. The abrasive material was fed at the contacting face between the rotating rubber wheel and the test sample. The tests were conducted at a rotational speed of 200 rpm. The rate of feeding the abrasive material was 250 ± 10 g/min. The effect of different loads (22 and 32 N) at a constant rubbing velocity of 2.33 m/s on the wear loss of the composites was investigated. In addition, at each load, the abrading distances were varied in steps of 135 m from 135 to 540 m. The wear behaviour was measured by the loss in weight, which was then converted into wear volume using the measured density data. The specific wear rate (K_s) was calculated from the equation;

$$K_s = \frac{\Delta V}{Ld}(\mathrm{m}^3/\mathrm{Nm}) \tag{1}$$

where, ΔV is the volume loss in m^3, L is the applied load on the specimen in Newton and d is the abrading distance in meters. A JEOL JSM-840 scanning electron microscope was used to study the features of the worn surface of the samples. The samples were gold sputtered prior to SEM analysis.

Results and Discussion

The measured mechanical properties of unfilled, silicon carbide (SiC), jatropha oil cake (JOC) and a mixture of SiC + JOC filled glass fabric–epoxy (GE) composites are given in Table 1. The observed tensile strength was high for SiC filled GE composite and low for unfilled GE composite. The increased tensile strength in the case of SiC filled GE composite may be attributed to the presence of tetrahedral crystals of carbon and silicon atoms with strong bonds in the SiC lattice [29]. A similar behaviour was noticed by Suresha et al. [30] in SiC filled GE composites. The incorporation of JOC into GE composite did not reduce the tensile strength and tensile modulus. The tensile strength of the GE + SiC + JOC composite was found to be higher than the GE + JOC composite but lower than the GE + SiC composite.

The wear volume loss as a function of abrading distance at two different loads (22 and 32 N) is given in Fig. 1a, b. Irrespective of the sample and load, wear volume loss was found to increase with increasing the abrading distance. In general, the observed wear volume loss was high at a 32 N load as compared to a 22 N load. With the increased load, the incorporated filler in the composite may become loosened owing to the wear of the matrix material and detach itself from the system leading to a higher wear loss [31]. The highest wear volume loss was noted for the unfilled GE composite at both 22 and 32 N loads. The observed wear volume loss trend for the unfilled GE composite is in line with the results reported elsewhere [16]. The lowest wear volume loss was noticed for the composites containing SiC. This may be due to the presence of SiC particles on the surface of the composites which can act as an effective barrier to prevent the large scale deformation of the epoxy resin. The SiC incorporated epoxy resin in GE composite is thought to create a hard surface due to the high degree of hardness of SiC (2,400 kg/mm^2) [32] and this may also be the reason for the reduced wear loss in SiC filled GE composite. The JOC and SiC + JOC filled GE composite

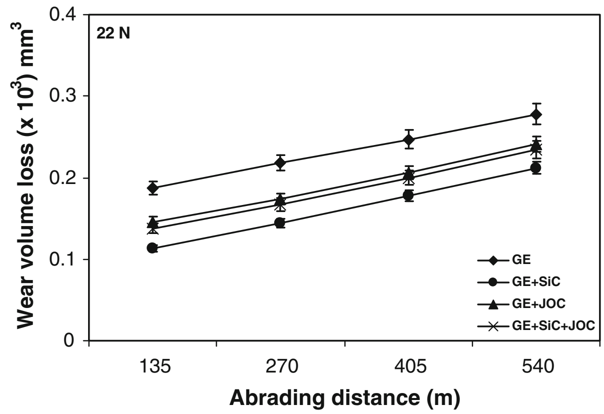
Table 1 Mechanical properties of unfilled and SiC, JOC and SiC + JOC filled glass epoxy (GE) composites

Properties	Samples			
	GE	GE + SiC	GE + JOC	GE + SiC + JOC
Tensile strength (MPa)	311	339	312	310
Standard deviation	2.88	3.12	2.93	3.28
Elongation at fracture (%)	3.80	3.48	3.90	4.05
Standard deviation	0.21	0.24	0.21	0.22
Product (σe)	1,182	1,179	1,217	1,255
$1/(\sigma e)$ factor ($\times 10^{-4}$)	8.46	8.48	8.21	7.96
Surface hardness (Shore D)	85	90	82	83
Standard deviation	1.31	2.07	1.15	1.18

exhibited a low wear volume loss as compared to unfilled GE composite. Due to the presence of a small amount of un-extracted oil in JOC, may have acted as a lubricant and this may be the reason for the low wear loss in JOC filled GE composite as compared to that of the unfilled composite. The wear resistance offered by different composites of the present research investigation followed the sequence: GE + SiC > GE + SiC + JOC > GE + JOC > GE. The wear data revealed that, the specific wear rate (Fig. 2) tends to decrease with increasing the abrading distance. The low specific wear rate was noticed for all filled GE composites as compared to the unfilled GE composite. The specific wear rate of the composites followed a trend similar to the wear volume loss. The lowest specific wear rate was noted for the SiC filled GE composite followed by the SiC + JOC and JOC systems.

The correlation between wear volume loss and (σe) factor ('σ' is ultimate tensile strength and 'e' is ultimate elongation) has been reported in the literature [16]. In general, the incorporation of fibre/filler increases the tensile strength (σ) and reduces the ultimate elongation (e). Hence the product σe may become smaller in the case of a filled system compared to an unfilled system. In the present study, among a series of composites, the SiC filled GE composite having the highest wear resistance had the lowest (σe) factor. The highest and lowest hardness was noted for SiC and JOC filled GE composites respectively. This can be attributed to the rigid and soft characteristic nature of SiC and JOC, respectively.

Figure 3 shows the SEM image of the un-abraded surface of the GE composite. The absence of any specific characteristic surface features (fibre, debris, fibre cutting, etc.) can be observed from the Fig. 3. The characteristic worn surface features of unfilled and filled composites at 135 and 540 m abrading distances with an applied load of 32 N were analysed using SEM (Figs. 4, 5, 6, 7). In general, the SEM images of all the composites exhibited relatively more wear at the 540 m abrading distance compared to that of 135 m. This may be due to the



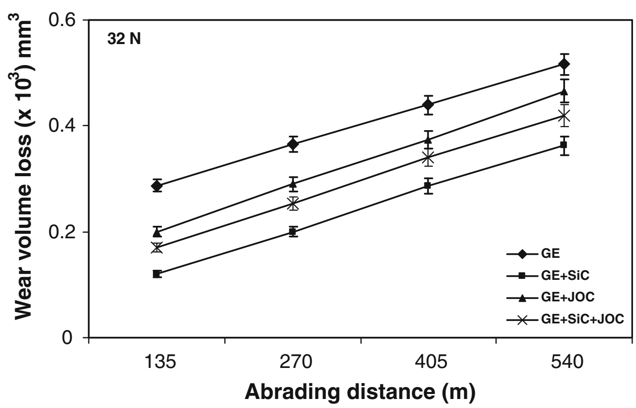


Fig. 1 Wear volume loss of unfilled and filled GE composites as a function of abrading distance at 22 and 32 N loads

detachment of the matrix material at higher loads and loosening the filler material responsible for wear resistance and contributing to the exposure of fibres to wear and breakage.

The SEM image of the unfilled GE composite is shown in Fig. 4a, b. The matrix and fibre damage due to the cutting action by abrasive particles could be seen from the microphotographs. The fibre fracture process appears to cause more damage to the fibre and matrix due to the cutting action. The exposure of several fibres due to the

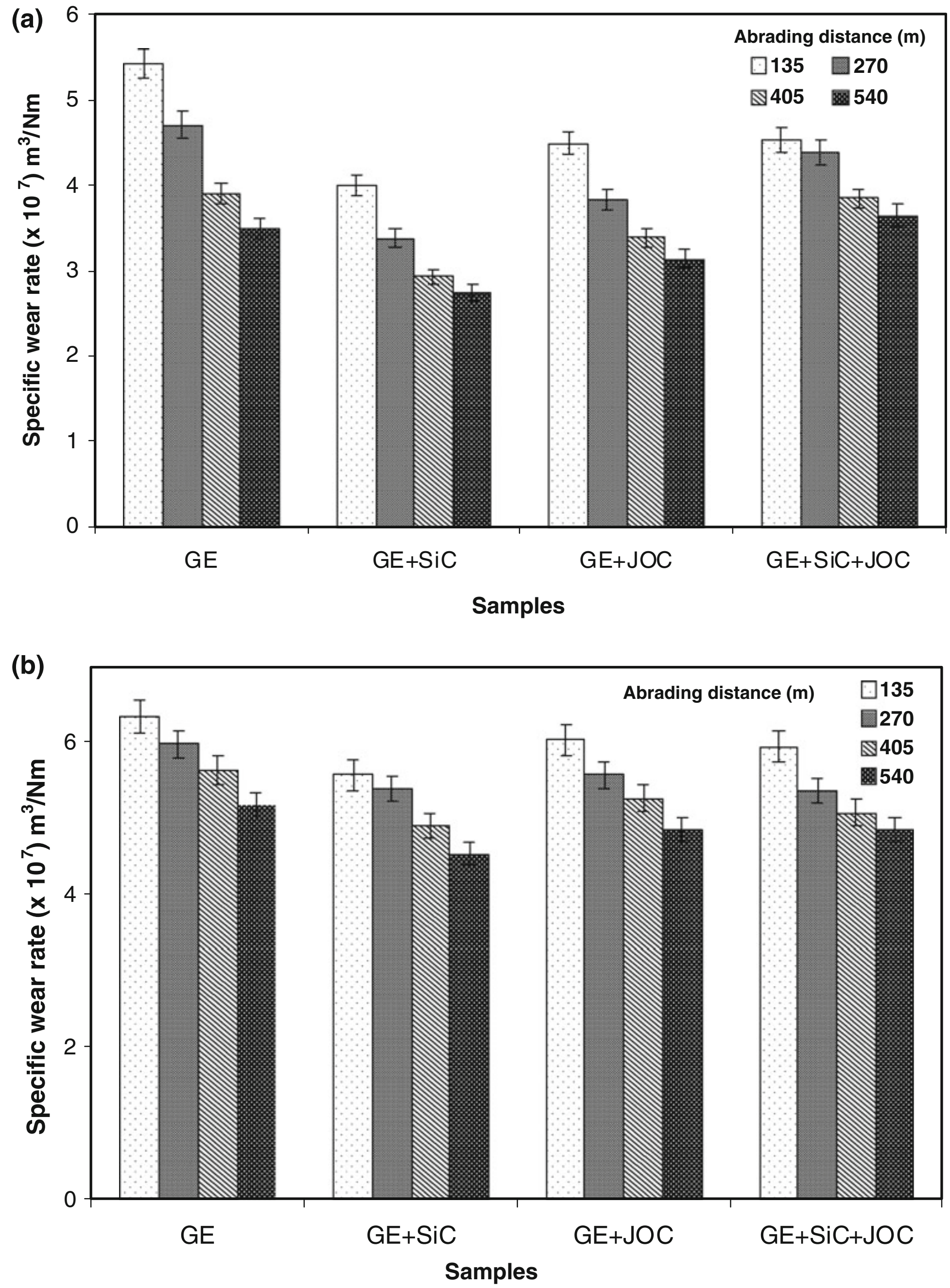


Fig. 2 Specific wear rate of unfilled and filled GE composites as a function of abrading distance at **a** 22 and **b** 32 N load

matrix detachment on the abraded surface is an indication of more wear loss. The poor matrix ductility and fibre-matrix adhesion may be the reason for the higher wear rate. The fibre fracture may be due to abrasion and transverse bending by sharp abrasive particles resulting in fibre fragmentation and the fibres being torn from the matrix.

Figure 5a, b shows the worn surface features of SiC filled GE composites at 135 and 540 m abrading distances. The amount of observed fibre fracture is relatively low compared to the unfilled GE composite. The exposure of less fibre on their abraded surfaces can be observed from their SEM images. The presence of characteristic wear debris as small white particles in the SEM images may be attributed to the ploughing and cutting action of the abrasive particles. Relatively, the high wear resistance in SiC filled GE composite may be due to less matrix and fibre breakage. The hard SiC particles containing the epoxy matrix present on the surface of the composite may be acting as an effective barrier to prevent the damage and exposure of the fibre due to abrasive action.

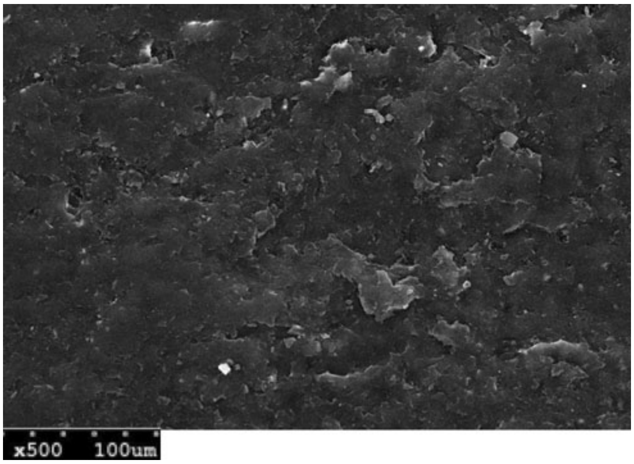


Fig. 3 SEM image of unfilled and un-abraded GE composite

A significant difference between the SEM images of unfilled and JOC filled GE composite could be observed. Both at high and low abrading distances, the JOC filled GE composite (Fig. 6a, b) exhibited relatively less fibre opening and fibre fracture compared to the unfilled GE composite. The SEM images of the GE composite containing a mixture of SiC and JOC (Fig. 7a, b) revealed a better and improved abrasion resistance compared to unfilled and JOC filled GE composites. The observed fragmentation of matrix and fibre was relatively low compared to unfilled and JOC filled GE composite. At the 135 m abrading distance, only loosening of the matrix could be observed without the exposure of any fibres (similar to the SiC filled GE composite). At 540 m abrading distance, a little detachment of matrix and fibre

Fig. 4 SEM image of worn surface of GE composite at **a** 135 and **b** 540 m abrading distance

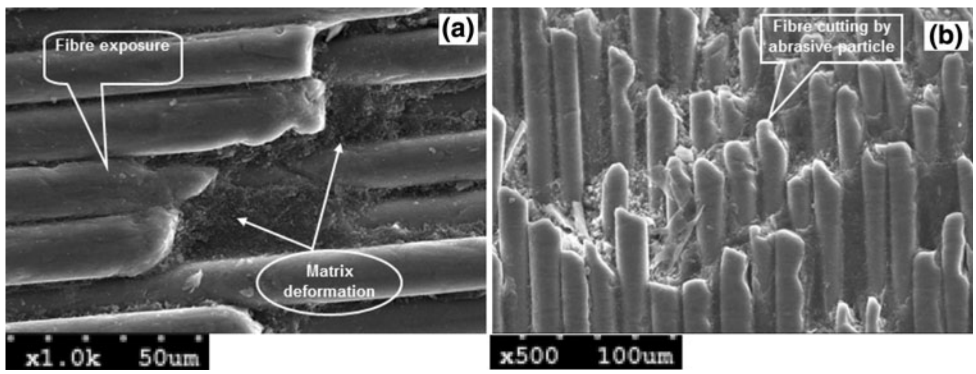


Fig. 5 SEM image of worn surface of SiC filled GE composite at **a** 135 and **b** 540 m abrading distance

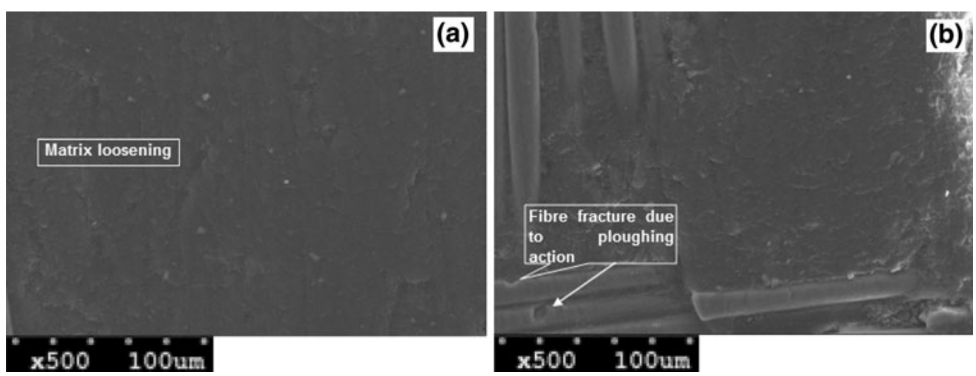
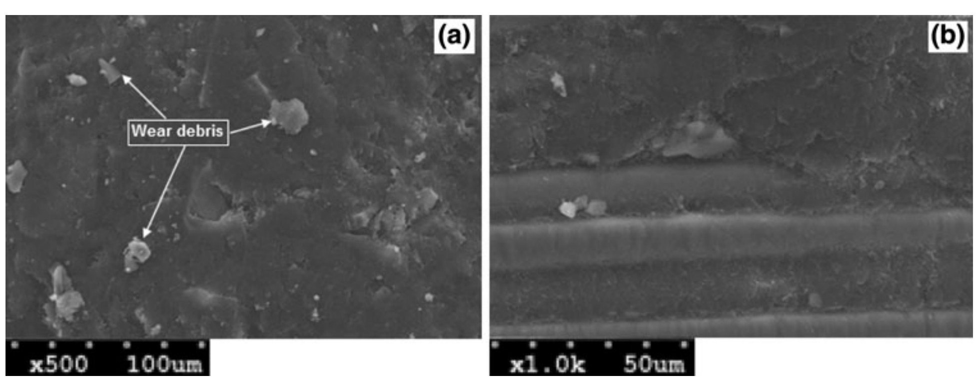


Fig. 6 SEM image of worn surface of JOC filled GE composite at **a** 135 and **b** 540 m abrading distance



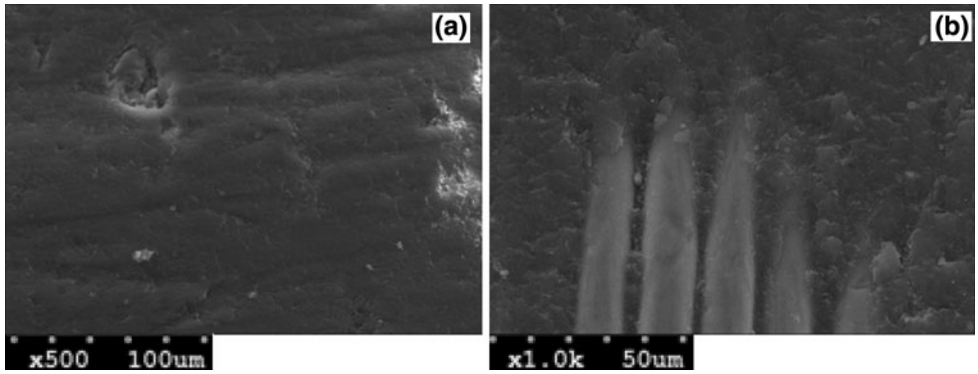


Fig. 7 SEM image of worn surface of SiC + JOC filled GE composite at **a** 135 and **b** 540 m abrading distance

can be seen. The improvement in the abrasion resistance in SiC + JOC filled GE composite compared to JOC filled ones may be due to the synergistic effect of hard SiC particles and soft oily JOC (acting as lubricant) which may not have allowed the abrasive particles to cause more wear.

Conclusions

The following conclusions can be drawn from the mechanical properties and three-body abrasive wear behaviour of unfilled and filled GE composites;

1. The incorporation of JOC did not show any negative effect on the tensile strength of GE composites. The observed hardness was low in the GE JOC composite compared to that of the unfilled GE composite. Relatively, the JOC filled GE composite exhibited a higher percentage elongation than other composites.
2. The three-body abrasive wear behaviour of both unfilled and filled GE composites was found to depend on the abrading distance and the applied load. The incorporation of JOC into the GE composite exhibited a higher wear resistance as compared to the unfilled GE composite. The mixed JOC + SiC filler further exhibited a higher wear resistance as compared to only JOC filled GE composites. However, the SiC filled GE composite showed highest wear resistance.
3. SEM studies of the worn surfaces of the composites revealed the fibre-matrix damage due to the cutting action by the abrasive particles.
4. The incorporation of silane treated JOC may enhance the mechanical and abrasive wear resistance of glass epoxy composites. Hence future work has been planned to follow a similar direction.

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