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ORIGINAL PAPER

Investigation on Sliding Wear Behaviour and Mechanical Properties of Jatropha Oil Cake-Filled Glass-Epoxy Composites

N. Mohan · S. Natarajan · S. P. KumareshBabu · Siddaramaiah

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Abstract The effect of Jatropha oil cake (JOC) filler incorporation into glass fabric-epoxy (G-E) composites and its sliding wear and mechanical properties were evaluated. The sliding distance and applied load are the process parameters at constant sliding velocity. The results show that JOC-filled composites exhibit better sliding wear performance at all test conditions. The wear loss increases with an increase in sliding distance and applied load, whereas under the same conditions, the specific wear rate decreases. The operating wear mechanisms have been studied at higher and lower sliding distances at applied loads of 10 and 20 N by using scanning electron microscope (SEM). The SEM results indicate more severe damage to matrix and glass fibre in unfilled composite systems compared to JOC-filled composite.

Keywords Sliding wear · Glass fabric-epoxy · Jatropha · Wear mechanisms

Introduction

The incorporation of jatropha oil cake (JOC) in the polymer matrix composites (PMCs) is expected to reduce the demand and dependency on petroleum-based raw materials. Polymer-based composites are extensively used in tribological sectors because of their light weight, excellent strength-to-weight ratios, resistance to corrosion, non-toxic nature, being easy to fabricate, design flexibility, self-lubricating properties, better coefficient of friction, and better wear resistance as compared to metal-based counterparts [1]. The fibre-reinforced polymer composite parts, such as gears, cams, wheels, brakes, bearing liners, rollers, seals, clutches, bushings, etc., find extensive application in automotive and aerospace sectors [2]. The material removal process in glass fabric reinforced plastic (GFRP) composites in dry sliding wear is dominated by four wear mechanisms, namely matrix wear, fibre sliding wear, fibre fracture, and interfacial debonding. Amongst these, fibre fracture and interfacial debonding are commonly considered as a combined process [3]. The incorporation of natural fillers makes PMCs a promising material with good tribological properties [4, 5]. Basavarajappa et al. [6] showed that the incorporation of silicon carbide and graphite filler in glass-epoxy composite exhibits lower weight losses. Cenospheres, a by-product of coal combustion in thermal power plants, have been used as a filler in G-E composites by Suresha et al. [7]. Syed et al. [8] studied the abrasive wear behaviour of coleus spent (CS) filled unsaturated polyester/polymethyl methacrylate (USP/PMMA) semi-interpenetrating polymer network (SIPN) composites. The CS-filled USP/PMMA SIPN composites showed better abrasion wear resistance as compared to unfilled SIPN systems.

JOC is a by-product left over after the mechanical extraction of oil from jatropha seeds. The approximate yield of JOC is about 500–600 g/kg of jatropha seeds [9]. 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

N. Mohan · S. Natarajan (✉) · S. P. KumareshBabu
Department of Metallurgical and Materials Engineering,
National Institute of Technology, Tiruchirappalli 620 015, India
e-mail: drsnatarajan.nitt@gmail.com

Siddaramaiah
Department of Polymer Science and Technology,
Sri Jayachamarajendra College of Engineering,
Mysore 570 006, India

of the presence of large amounts of minerals [10], which is an added reason to investigate this material for tribological applications.

Dwivedi and Chand [11] studied abrasive wear behaviour of wood flour-filled epoxy composites, and they noticed that the composite containing 40 wt.% wood flour exhibited the lowest specific wear rate in abrasive wear mode. Furthermore, the composite containing 20 wt.% wood flour exhibited the lowest specific wear rate in sliding wear mode. Bhuyan et al. [12] provided some insight into the effectiveness of using bio-based spent germ filler (a by-product from a wet ethanol production plant) filled thermosetting polymer composites as potential tribomaterials.

The present research work aims to explore the possibility of using biowaste-based JOC in epoxy matrix and to study the wear behaviour of jatropha-filled and -unfilled G-E composites. It thus opens a new way to implement locally available inexpensive jatropha biowaste products as a filler and produce a new candidate for tribo material.

Experimental

Materials

E-Glass-woven fabric (360 g/m^2) was procured from M/s. Reva composites, India. The 18-μm-diameter glass fabric was used as reinforcement as shown in Fig. 1. Bifunctional epoxy-diglycidyl ether of butane diol (LY-5052) and cyclo aliphatic amine (HY-5052) (room temperature cure system) were obtained from M/s. HAM, Mumbai, India. The resin is a clear liquid with viscosity at 25 °C, 1,000–1,500 mPa, and specific gravity 1.17 g/cc. The hardener is a liquid with viscosity 40–60 mPa and specific gravity 0.94 g/cc. The JOC powder with particle size ranging from 50 to 70 μm was obtained from the National University of Malaysia and was used as a filler material. The residual oil content present in the JOC was 3–7%.

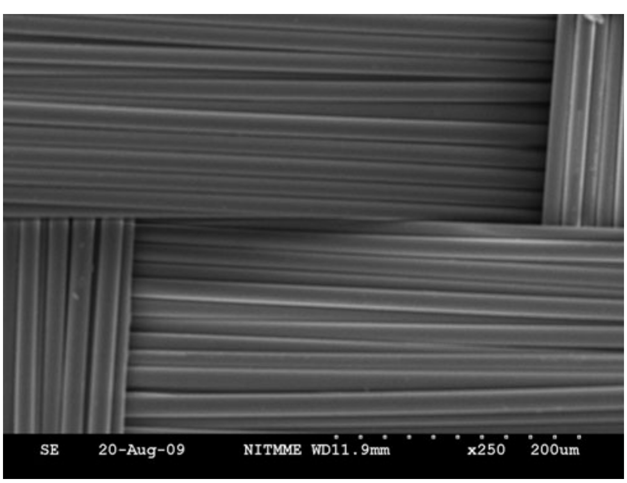

Fig. 1 SEM image of dry glass fabric of diameter 18 μm (before fabrication)

Fabrication of Composite Specimen

A resin mixture of epoxy and hardener was prepared at a ratio of 100:38 by weight by using a magnetic stirrer. A releasing agent is applied on the smooth surface of the mould for easy removal of the composite stack and to obtain better surface finish. A bidirectional E-glass woven fabric (360 g/m^2) was cut as per the required dimension and placed one above the other in the mould. For each placement of fabric, a well-stirred mixture of resin and hardener was applied between the fabrics by using a roller and brush. The exposed side of the stack was covered by a cellophane membrane and sealed around the periphery by using a suitable sealant. The weight percentage of glass fabrics in composites was maintained around 60 ± 2%. The laminate was cured under a pressure of 0.0965 MPa for 24 h at room temperature in an H-type press and post cured up to 100°C for 3 h in a controlled oven. The formulations of the composites are listed in Table 1. The JOC is incorporated into the resin mixture and stirred well for the fabrication of filled composite. The laminate was fabricated to the dimension of 300 mm × 300 mm × 2.6 mm, and the specimen was cut to the required dimension by 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 of the composite was measured using a shore D Durometer (M/s. PSI Sales Pvt. Ltd., India) as per ASTM D 2240 specifications.

A dry sliding pin-on-disc (POD) setup was used for the sliding wear tests. The fabricated specimen (5 mm × 5 mm × 2.6 mm) was glued to the pin of 10 mm diameter and 25 mm height, which came in contact with a hardened En32 steel disc of hardness 55 HRC. The steel disc was polished with waterproof SiC emery paper (600 grade) in

Table 1 Formulations of composite specimens

Specimen code	Epoxy (wt. %)	Glass fibre (wt. %)	Filler (wt. %) (JOC)
Glass fabric-epoxy composite (G-E)	40	60	0
JOC-filled glass fabric-epoxy composite (JOC-G-E)	34	60	6

order to obtain a surface roughness of 0.65 μm. The test pin was mounted on the lever arm. The applied normal loads were 10 and 20 N, and the sliding velocity was 1.5 m/s. Sliding distances varied from 2,000 to 8,000 m in steps of 2,000 m. The surface of the specimen was cleaned with a soft tissue paper soaked in acetone and compressed air before and after the test. The specimen weight was recorded using a digital electronic balance of 0.1 mg accuracy. The difference between initial and final weight of the specimen was a measure of slide wear loss. A minimum of three trials was conducted to ensure repeatability of test data. The digital photograph by a pin-on-disc (POD) machine is shown in Fig. 2.

Results and Discussion

Mechanical Properties

The measured tensile properties and surface hardness results of the unfilled and JOC filled G-E composites are listed in Table 2. The G-E composites with JOC filler exhibit better mechanical properties. Figure 3a, b shows the specimens after the tensile test. For G-E specimens tested along the longitudinal direction, the failure mode shows brittle fracture with matrix fracture at the surface of the fibre, and this could be due to plastic deformation of the matrix after fibre-matrix debonding (Fig. 3a). In JOC-filled G-E composites tested along a longitudinal direction, the fractured surface revealed the simultaneous breakage of fibres and matrix, which evidenced better interfacial adhesion (Fig. 3b) and led to a slightly higher tensile strength. The correlation between wear volume loss and mechanical properties ('σ' is the ultimate tensile strength and 'e' is the ultimate elongation at fracture) has been reported in the literature [13]. In general, the incorporation of filler increases the tensile strength (σ) and reduces the elongation (e). Hence, the product (σe) factor may become smaller in the case of a filled system compared to an unfilled system. In the present study, the JOC-filled G-E composite having the higher wear resistance had the lowest (σe) factor. Due to incorporation of JOC in G-E, the increase of elongation (e) is greater than the increase in tensile strength (σ). This can be attributed to the elastomeric nature of JOC, which has good bonding with the epoxy matrix. If the product (σe) factor becomes bigger, the specimen behaves in a brittle manner because of a decrease in elongation (e) that is greater than the increase in tensile strength (σ). This leads to lower wear resistance and initiation of failure by fibre-matrix debonding.

Table 2 Mechanical properties of unfilled and JOC-filled G-E composites

Properties	G-E	JOC-G-E
Tensile strength at break (MPa) (σ)	308	311
Standard deviation	2.73	3.39
Tensile modulus (GPa)	16.22	18.61
Standard deviation	0.14	0.20
Elongation at fracture (%) (e)	3.6	3.8
Standard deviation	0.22	0.34
Surface hardness (Shore-D)	84	82
Standard deviation	1.30	1.22
Product (σe)	1,108	1,181
1/(σe) factor ($\times 10^{-4}$)	9.02	8.46

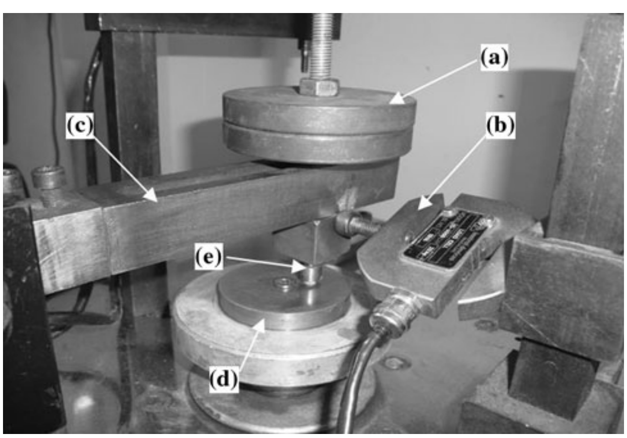

Fig. 2 A digital photograph of a dry sliding pin on a disc wear test rig. **a** Load cells, **b** force transducer, **c** lever arm, **d** hardened alloy steel disc; **e** composite sample is *glued* with specimen holder

Sliding Wear Behaviour and Specific Wear Rate

The graphic plots of wear loss as a function of sliding distance of an unfilled and JOC-filled G-E composite specimen rubbed against a hardened alloy steel disc are shown in Fig. 4a, b, respectively. It is obvious that the wear loss of composites increases with an increase in sliding distances. This is due to an increase in interfacial temperature with higher sliding distances and hence thermal softening; the matrix was detached from the specimen surface [14]. The wear debris consists of a shear-deformed polymer matrix containing broken pulverized glass particles and wear powders on the counterface. These particles can either be lost from the contact zone or remain there for a fixed time as a transfer layer on the steel counterface [15–17]. In such cases, their polymer component can be a barrier to the counterface asperities and reduce the effective toughness, but the broken pulverized glass particles can act as a third body abrasive leading to enhanced roughening of the counter surface. The wear loss of the unfilled G-E composite is much higher than the JOC-filled

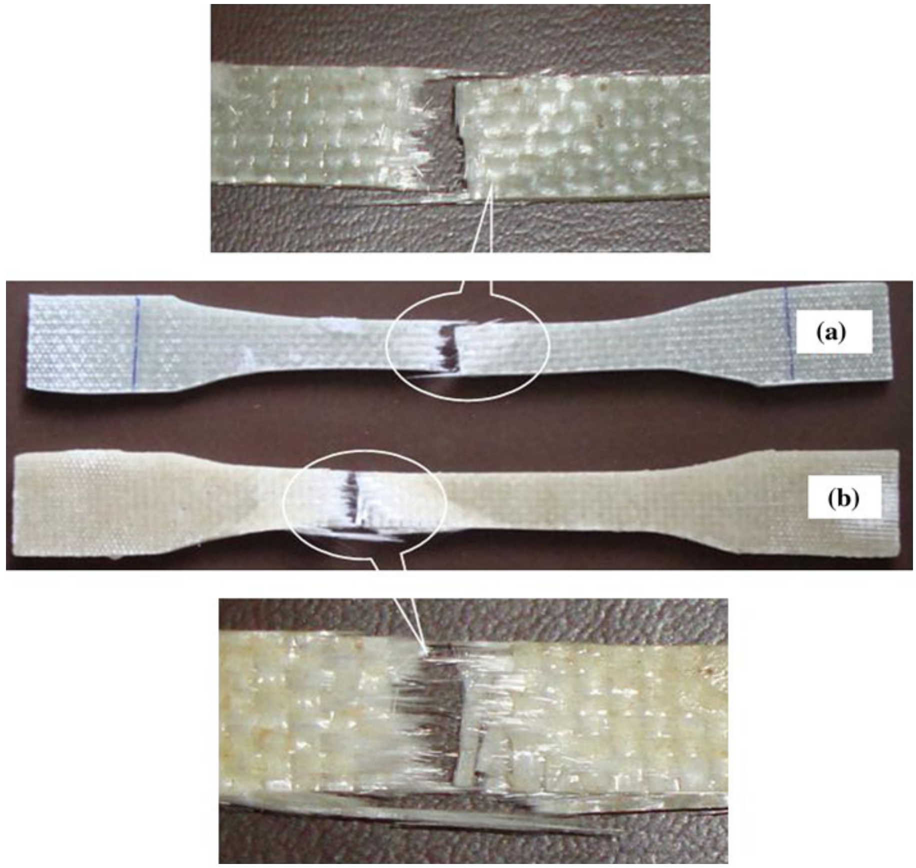

Fig. 3 Digital photographs of composite specimens after the tensile test. **a** G-E and **b** JOC-G-E

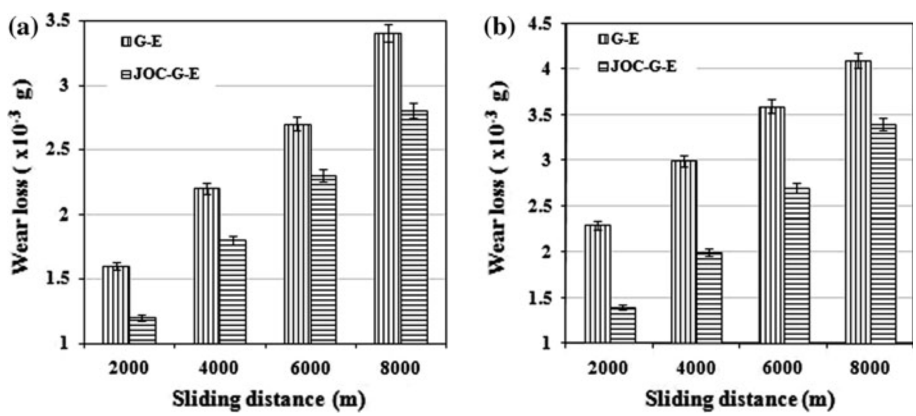

Fig. 4 Effect of sliding distance on wear loss of G-E and JOC-G-E specimens at **a** 10 N and **b** 20 N loads

G-E composite at all experimental conditions under investigation.

The JOC-filled G-E composite exhibited better wear resistance at different sliding distances. This behaviour can be attributed to the presence of jatropha particles on the surface, which act as effective barriers to prevent large-scale fragmentation of epoxy. The glass-fabric strengthens the composite, whilst the soft and oily nature of JOC particles in epoxy act as a lubricant, thus providing enhanced wear resistance.

The wear loss of 1.2×10^{-3} g was observed for the JOC-G-E composite at 2,000-m sliding distance at 10 N load, whilst for G-E composite at 8,000 m sliding distance at 20 N load, it was 4.1×10^{-3} g. The wear data of the composites revealed that the wear loss strongly depends on the applied load and sliding distance.

The graphical representations of specific wear rate (K_s) as a function of sliding distance at 10 and 20 N are shown in Fig. 5a, b, respectively. The specific wear rate decreases with an increase in sliding distance and applied load. The lowest K_s value of 1.2×10^{-14} m^3/N m at 8,000-m sliding distance and at 20 N load for JOC-G-E composite and the highest K_s values of 4.4×10^{-14} m^3/N m at 10 N load for G-E composite were observed. Two types of wear were

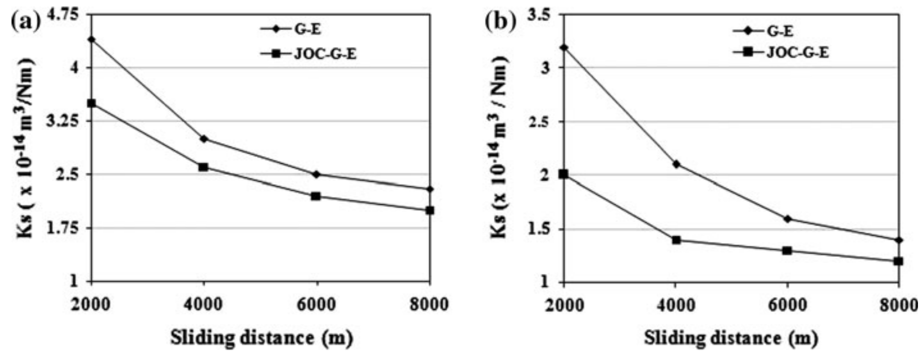

Fig. 5 Specific wear rate as a function of sliding distance of G-E and JOC-G-E specimens at **a** 10 N and **b** 20 N

Table 3 Coefficient of friction (μ) in G-E and JOC-G-E composite specimens under all experimental conditions

Load (*N*)	Coefficient of friction (μ)							
	Sliding distance 2,000 m		Sliding distance 4,000 m		Sliding distance 6,000 m		Sliding distance 8,000 m	
	G–E	JOC-G–E	G–E	JOC-G–E	G–E	JOC-G–E	G–E	JOC-G–E
10	0.34	0.27	0.30	0.4	0.28	0.47	0.24	0.59
20	0.66	0.5	0.53	0.69	0.45	0.85	0.41	0.95

observed in an unfilled G-E composite: (1) matrix wear and (2) fibre wear. The matrix wear occurs due to plastic deformation and cracks during sliding. Fibre wear occurs due to fibre rubbing, fibre rupture, fibre cracking, and fibre pulverizing. As compared to the unfilled G-E composite, the JOC-filled composite showed a lower specific wear rate. This is due to rich proteins present in the JOC, which form a self-tribofilm, and the slippery nature of this film, which reduces specific wear rate.

Coefficient of Friction

The measured coefficient of friction (μ) of the composites is given in Table 3. For the unfilled G-E composite with an increase in sliding distance, μ decreases, whereas with an increase in load, μ increases.

The JOC-filled composite shows a higher value of μ, and μ increases with an increase in sliding distance and applied load. This indicates that μ depends on the sliding distance and applied load. It was observed that at a lower sliding distance, the JOC-filled G-E composite shows a low value of μ as compared to the unfilled G-E composite. As sliding distance increases, more heat is generated, and consequently more fibre damage occurs. The oily nature of JOC particles in the epoxy matrix acts as a third body and is uniformly distributed on the surface of the specimen. They can easily roll and slip in between the counterface. Hence, JOC-filled composite shows a lower wear rate and high coefficient of friction. In G-E composites due to an increase in interface temperature, rupture of adhesive bonds occurs in between the counterface and specimen, and fibre glass particles dig into the specimen surface, giving a high wear rate and lower coefficient of friction.

Worn Surface Morphology

The SEM features of the worn surfaces of unfilled and JOC filler-filled G-E composite specimens at two loads (10 and 20 N) and at two sliding distances (2,000 m and 8,000 m) are shown in Figs. 6a–d and 7a–d, respectively.

The short run specimen in Fig. 6a indicates spread of the matrix, matrix fracture, and matrix masking the fibres. Hence, exposure of a fibre was not observed. Figure 6b depicts that severe fibre damage results in greater wear of the matrix and fibre, confirming the effect of longer sliding distance (white curved arrow indicates the direction of sliding). As compared to the short run specimen (Fig. 6a), a long run specimen (Fig. 6b) shows more damage. The surface damage increases with an increase in applied load. As compared to a specimen run at a lower load (Fig. 6a, b), the specimen run at a higher load (Fig. 6c, d) depicts more severe damage and matrix away from the fibre surface, and also exposure of fragmented fibre was observed in brittle manner. Compared to a short-run specimen (Fig. 6c), a long-run specimen (Fig. 6d) shows more severe fibre damage, more exposure of longitudinal fibre with broken ends, and void formation due to chipping out of the matrix (marked by *inclining arrow line*). This can be attributed to the higher sliding distances as more heat is developed in between the specimen surface and counterface.

The surface features of the JOC-filled G-E composite specimen are shown in Fig. 7a–d. Figure 7a shows that

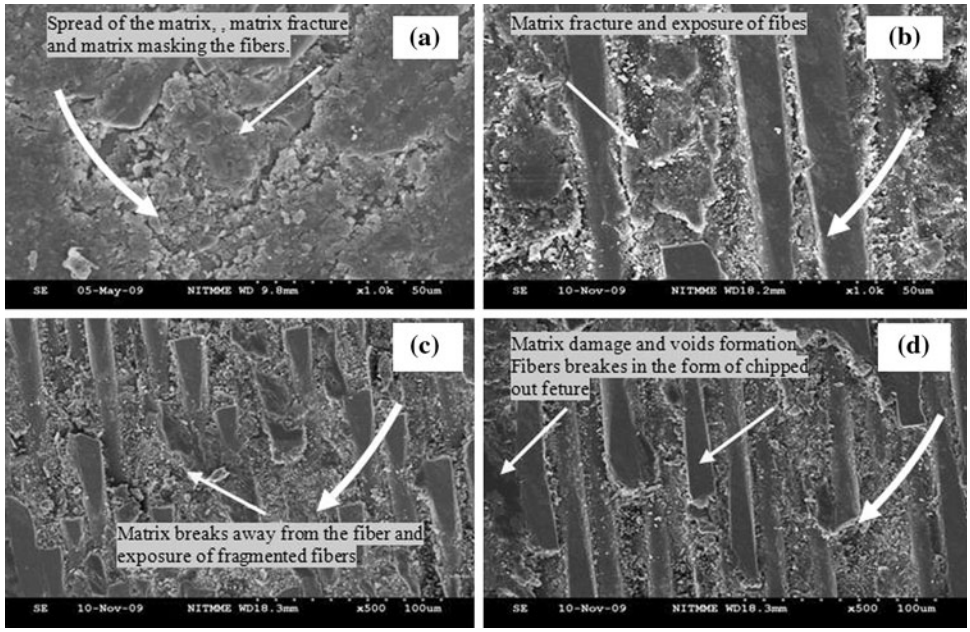

Fig. 6 SEM images of G-E specimens at 10 N load: **a** 2,000 m, **b** 8,000 m sliding distance and at 20 N load, **c** 2,000 m, and **d** 8,000 m sliding distance

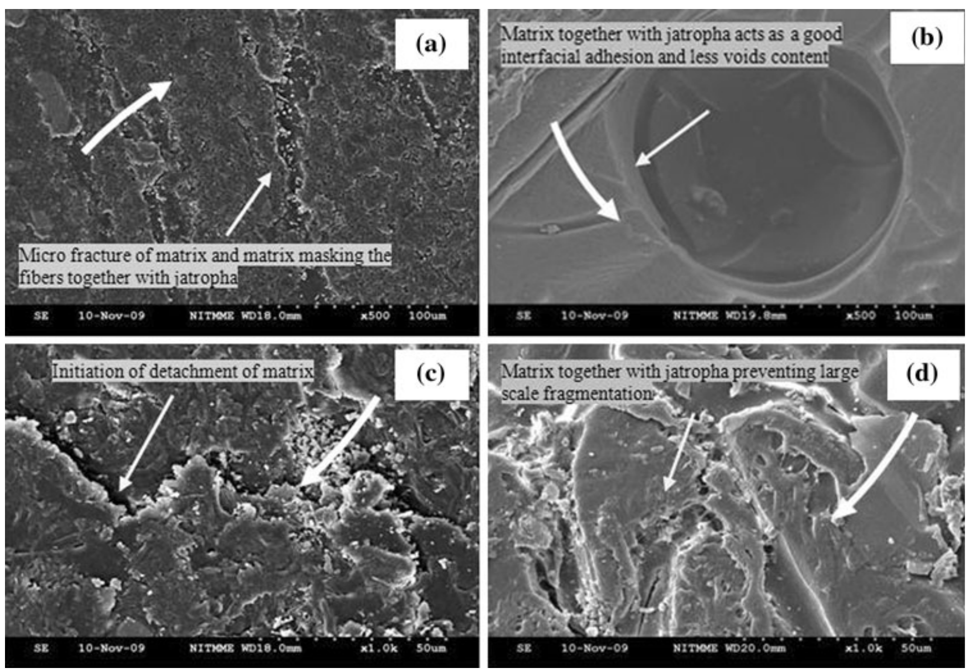

Fig. 7 SEM images of JOC-filled G-E specimens at 10 N load: **a** 2,000 m, **b** 8,000 m sliding distance and at 20 N load **c** 2,000 m, and **d** 8,000 m sliding distance

short run specimen surfaces were much smoother, and the glass fibre detachment was greatly limited with the addition of JOC. In a long run specimen (Fig. 7b), masking of the fibres by the epoxy matrix together with jatropha in preventing the large-scale fragmentation was observed.

Figure 7c, d depicts initiation of the detachment of the matrix. As compared to lower load, at higher load SEM images show more surface damage and less masking of fibre and formation of debris with a thin transfer film on the surface of the JOC-G–E specimen. Better wear resistance, good interfacial adhesion, and less void content are exhibited after incorporation of the oily nature of JOC in the G-E composite.

Conclusions

- Tribological behaviour of unfilled and JOC-filled G–E composites strongly depends on the experimental test parameters such as sliding distance and applied load.

- Wear loss increases with an increase in sliding distance and applied loads. However, the JOC-filled G-E composite system showed better wear resistance.
- The specific wear loss decreases with an increase in the sliding distance.
- The change in the coefficient of friction with sliding distance does not follow a fixed pattern for either composite.
- The incorporation of JOC in the G-E composite shows a positive effect on tensile strength and better improvement in the tribological properties.
- SEM images indicate matrix debris formation, fibre breakage, matrix cracking, void formation due to chippinh out of the matrix, and exposure of fibres due to wear of the matrix.

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