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Influence of calcium sulfate whiskers on the tribological characteristics of automotive brake friction materials

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ABSTRACT

Functional fillers enhance the frictional performances of automotive brake friction materials (BFM). The influence of Calcium sulfate whiskers ($CaSO_4$) in BFM's and its effect as a functional filler on the tribological performances were investigated. Four compositions containing all essential ingredients of BFMs were developed, varying the amount of $CaSO_4$ whiskers (5%, 10% and 15%) and one composition without $CaSO_4$ whiskers. The tribological studies were carried out on an inertia brake dynamometer as per JASO C 406 standard. Addition of $CaSO_4$ whiskers was found to improve the mechanical properties of BFM composites. The inclusion of $CaSO_4$ whiskers enhanced the frictional performance during fade. Surface analysis of the worn pads was carried out to understand the wear mechanism using scanning electron microscope. The friction materials with the inclusion of calcium sulfate whiskers experienced lesser wear. The composite with 10 wt% of $CaSO_4$ whiskers exhibited the most stable friction coefficient even at high sliding speeds.

1. Introduction

The brake system is the most essential aspect for the safety side in an automobile. It must be able to stop the vehicle under any speed. At the braking interface while applying brake the kinetic energy generated is converted to heat energy. The heat generated during braking degrades the organic polymer matrix in the surface of the friction pads. The frictional heat leads to the loss of mechanical properties of the friction materials. This leads to a drop in the frictional performance of the brake friction material [1–3]. Fade is defined as the loss of frictional performances due to the increase in temperature at the contact interface during braking. The organic fibers and friction modifiers generates heat at the interface which results in fade and the friction film at the contact zone also affects the frictional performance [4]. The effect of temperature on the braking effectiveness (fade) and the ability of the brake friction materials to regain its frictional stability (recovery) is the main desired characteristics of a brake friction material. The acceptable aspects of a brake friction composite are lower rate of fade and faster rate of recovery [5–15]. It is a difficult task to develop a friction material satisfying various performance parameters [16]. The effects of various raw materials on the tribological performances of BFMs have been extensively studied in the literature [17–22]. Hence, it is necessary to monitor appropriately the material composition of the BFM composite for improved friction stability.

The friction materials approximately contain 10–20 ingredients, these ingredients are categorized as fillers, reinforcing fibers, friction modifiers and binders [23–27]. Potassium titanate whiskers possess good thermal resilient, but it is very hard which will wear the counter disc [28]. Among the numerous reinforcing fillers, whiskers are one of the best option as it has a perfect crystal structure. The whiskers are expensive and it processes excellent reinforcing effect in NAO brake friction materials [29]. $CaSO_4$ whiskers is a kind of mineral and it is in single crystal structure. Each whisker has a diameter of 1–4 μm and length about 10–200 μm. The development of calcium sulfate whiskers are less costly compared to other whiskers [30]. From the literature it was found that calcium sulfate whiskers ($CaSO_4$) possess high thermal stability due to their perfect crystal structure [31]. Inclusion of $CaSO_4$ in NAO brake friction material has not been widely reported.

In the present study, Influence of calcium sulfate whiskers on the tribological characteristics of BFMs were studied. Four different compositions of BFMs were fabricated by varying the proportion of

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$CaSO_4$ whiskers. The effect of fade and recovery were evaluated on inertia dynamometer as per JASO C 406 standard. The worn out surfaces of the pads were studied using FESEM micrographs.

2. Materials and methodology

2.1. Selection of raw materials

Four brake friction material composites containing multiple ingredients were fabricated. In the friction material formulation, there are organic reinforcing fibers, phenolic based primary binders, rubber based secondary binders, synthetic graphite as lubricant and the cashew dust as friction modifier. $CaSO_4$ whiskers (Hefei Jiankun Chemical Industry, China) were used as functional fillers in this formulation. The technical data of calcium sulfate whiskers received from the supplier are given in Table 1. In Table 2, the composition of the four formulations, viz. C_0, C_1, C_2 and C_3 are given.

2.2. Manufacturing of brake friction material

The homogeneous mixing of all the ingredients is the initial process in the BFM fabrication. The mixing of all the raw materials was performed in a plough shear mixer in a sequence for 22 min. The feeder runs at 150 rpm and the chopper works at 3000 rpm. The sequence of mixing all the ingredients are shown in Table 3. A shot blasted back plate applied with adhesive is placed in the cavity of a hot compression moulding machine and 120 g of the final mixture was filled. The compression was carried out under 17 Mpa pressure at 160 °C for 20 min. The pads were then cured for 6 h at 120 °C in an oven. The developed pad surfaces were grinded to achieve the desired thickness. The manufacturing process flowchart is shown in Fig. 1.

Table 3
Mixing sequence of the ingredient.

SI. No	Type of ingredient	Duration of mixing in min
1	Reinforcement fibers	10
2	Fillers and barites	7
3	Flakes and other powdery ingredients	4
4	Glass fiber	1

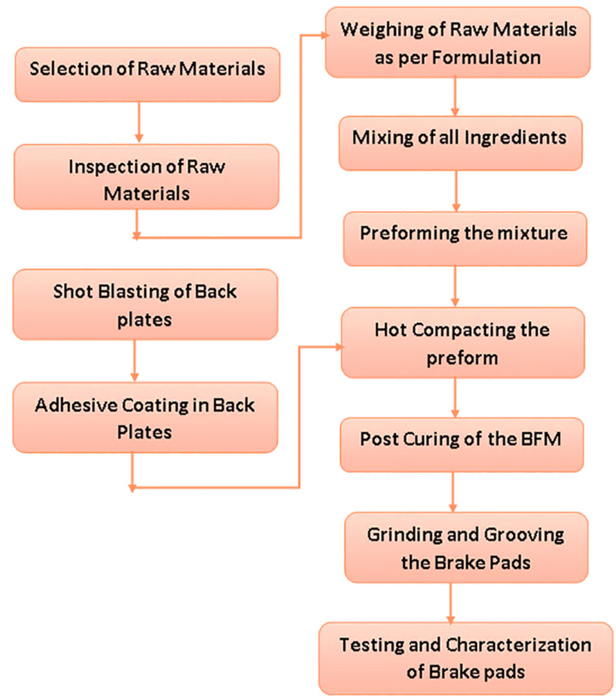

Fig.1. Manufacturing process flowchart.

2.3. Characterization of brake friction material.

The physical and chemical properties of the developed BFMs were characterized as per industrial standards. As per ASTM D792, the density of the friction materials was calculated [32] and the porosity, as per JIS D4418 [33]. As per ASTM D785, the hardness of the friction materials was determined [34]. The quantity of uncured resin content on the developed pads was obtained as per IS 2742 [35]. After the tribological tests, described in the next section, the worn-out surfaces of the friction pads were analysed from the images of scanning electron microscope. For the SEM analysis the full brake pad cannot be employed, hence a small portion of samples were cut out from the pads and eight random locations were chosen for study.

Table 1
Technical data of calcium sulfate whiskers.

Properties	$CaSO_4$ Whiskers
Appearance	White powder
Fineness	80–120 mesh
Absolute density	2.69 g/cc
Particle shape	Needle shaped fiber
Length	10–200 μm
Diameter	1–4 μm
Moisture content	≤1.5%
Melting point	1450 °C
Heat resistance:	1000 °C

2.4. Tribo-evaluation of brake friction material

The tribological performance of the developed BFMs was evaluated using full scale inertia brake dynamometer. The rotor disc and pad set up are shown in Fig. 2. The dynamometer could operate in the inertia ranges between 1 kg-m^2 to 1570 kg-m^2 [36].

Table 2
Brake friction material formulation.

Constituents	Functionality	Amount wt%			
		C_0	C_1	C_2	C_3
Aramid Fiber (=10%) Rockwool (=10%)	Reinforcing Fibers	20	20	20	20
Phenolic resin, NBR, SBR	Binders	15	15	15	15
$CaSO_4$ whisker	Functional Filler	**0**	**5**	**10**	**15**
Barites	Inert Filler	20	15	10	5
Synthetic Graphite, Cashew dust, Tin sulphide, potassium titanite	Friction additives	45	45	45	45

The bold values are the main objective values we have modified in our research.

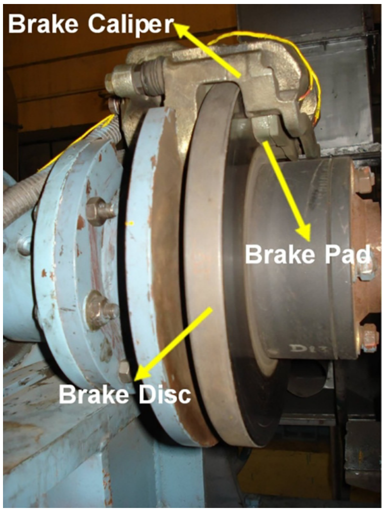

Fig. 2. Rotor disc and pad arrangement in dynamometer.

The developed BFMs were tested on the full scale inertia brake dynamometer as per Japanese JASO C 406 standard [37]. The friction materials were studied for pressure and speed sensitivity from the effectiveness test. The fade and recovery tests were performed to investigate further the temperature sensitivity of the BFMs. On first braking application during fade cycle the temperature was 60 °C. The temperature started to rise and during the fade test the highest temperature recorded was 480 °C

Friction coefficient (μ), Performance Friction coefficient (μp), Fade Friction Coefficient (μf) and recovery Friction coefficient (μr) were acquired from the dynamometer test. From the following formulae the fade and recovery ratios were determined.

$$\%\ \text{Fade ratio} = \left(\left(\mu_{p-}\mu_f\right)/\mu\right) \times 100$$

$$\%\text{Recovery ratio} = (\mu_r/\mu) \times 100$$

For the fade ratio: lower the μ is better. While for the recovery ratio is opposite: the higher the μ is better.

Speed spread (SS) is calculated as the ratio of μ at a given lower speed (mild condition – 50 to 100 kmph), to the subsequent higher speed (severe condition – 100 to 130 kmph) and is measured in terms of %. An excellent characteristic of a BFM is that the speed spread should be higher with fewer undulations [38].

In the dynamometer test, the grey cast iron brake disc of commercially available Maruti Wagon-R was used as a counter disc. Initially the friction pads were subjected to burnishing, which aids in providing efficient contact between the friction pad and the counter disc.

3. Results and discussion

3.1. Chemical and physical properties of the BFMs developed

The chemical and physical properties of the developed BFMs are given in Table 4. The density of the BFM varies according to the percentage of barites. As the barites density (4.5 g/cc) is higher than the density of $CaSO_4$ whisker (2.69 g/cc), the density changes are in the order, viz. $C_0 > C_1 > C_2 > C_3$. There is only a minor variation in the hardness of the BFM composites. Higher values of porosity were found for the C_1 composite, this is mainly because of higher density of the composite C_1, which have a positive impact on the tribological performances of the BFM. Lower values of acetone extraction is found for the composite C_2, it shows the better curing ability of the composite. The amount of uncured resin from the acetone extraction process for all the composites was in the acceptable industrial range of maximum 5% [14].

3.2. Tribological performance of the BFM developed

(a) Effectiveness II

The effect of braking pressure on the frictional performances at different speeds is observed in this effectiveness study.

3.2.1. Pressure sensitivity of the brake friction material

The friction materials developed were tested for its frictional performances at various speeds as per the second effectiveness schedule in dynamometer (Table 5). The braking pressure was expressed in terms of deceleration (g). The tribological performances of the developed friction materials at different speeds are given in Fig. 3. Expressing a steady friction coefficient (μ) even at severe conditions, is an endorsed characteristic of a brake friction material. Fig. 3a shows the friction coefficient (μ) for 50 km/h condition, which was noted to be in the range of 0.36–0.55. The friction coefficient (μ) for all the composites had a similar pattern with little variation. The composites 'C_2' exhibited the most stable frictional behaviour of all, while the composites 'C_1' and 'C_3' exhibited similar behaviour and the specimen 'C_0' exhibited the most unstable behaviour compared with the other composites. The increase in the contents of $CaSO_4$ whiskers in the formulation helps in providing a stable friction. This phenomenon is because the $CaSO_4$ whiskers has high thermal stability, it protects the friction surface from thermal degradation while braking. It also helps in the reduction of gaseous layer formation between the pad and disc, which enhances the friction stability. The trend we have discussed is about the relation between the amount of $CaSO_4$ whiskers and the friction stability. The heat generation at the interface increases when the deceleration rate increases, this frictional heat generated leads to the frictional performance loss [38,39]. The stability of friction was better up to 0.7 (g) for all the composites and then there was a little fading in the frictional performance. The friction coefficient at 130 km/h condition was in the range of 0.41–0.47 (Fig. 3c). The pressure fade was experienced from 0.6 (g) in all the composites. The frictional performances of the BFM composites at several decelerations are almost in the same range with less fluctuation. Overall, 'C_2' exhibited good stability in friction coefficient even at the higher speeds.

3.2.2. Speed sensitivity of the brake friction material

The speed spread on both the conditions are shown in Fig. 4. It was observed from Fig. 4a, the speed spread during mild condition did not have much variations. For all the developed friction

Table 4
Physical and chemical properties of the friction composites developed.

Properties	Unit	Test Standards	C_0	C_1	C_2	C_3
Porosity	%	JIS D 4418	4.12	7.32	5.16	6.13
Hardness	HRS	ASTM D785	86	87	90	89
Acetone extraction	%	IS 2742 (part 3):1994	1.79	1.70	1.61	1.68
Density	g/cc	ASTM D792	2.45	2.41	2.39	2.36

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Table 5
Dyno test procedure for tribo evaluation as per JASO C 406 standard.

Description	Deceleration (g)	Speed (Km/h)	Initial Temperature (°C)	No of Braking Application
Effectiveness II	0.1–1	50	50 or less	The brake applications were repeated until the record can be obtained for 4 measuring points as equally as possible, within the range of specified braking deceleration.
Low Temp. Effect. Test				
Normal Temp. Effect. Test	0.1–1	50 100 130	80. Front	
Fade and Recovery-I	0.5	50	80	3
Base line check				
Fade-I	0.3	80	60 at first braking	10
High Temp. Effect. Test	0.5	80	–	1
Recovery-I	0.5	50	–	12

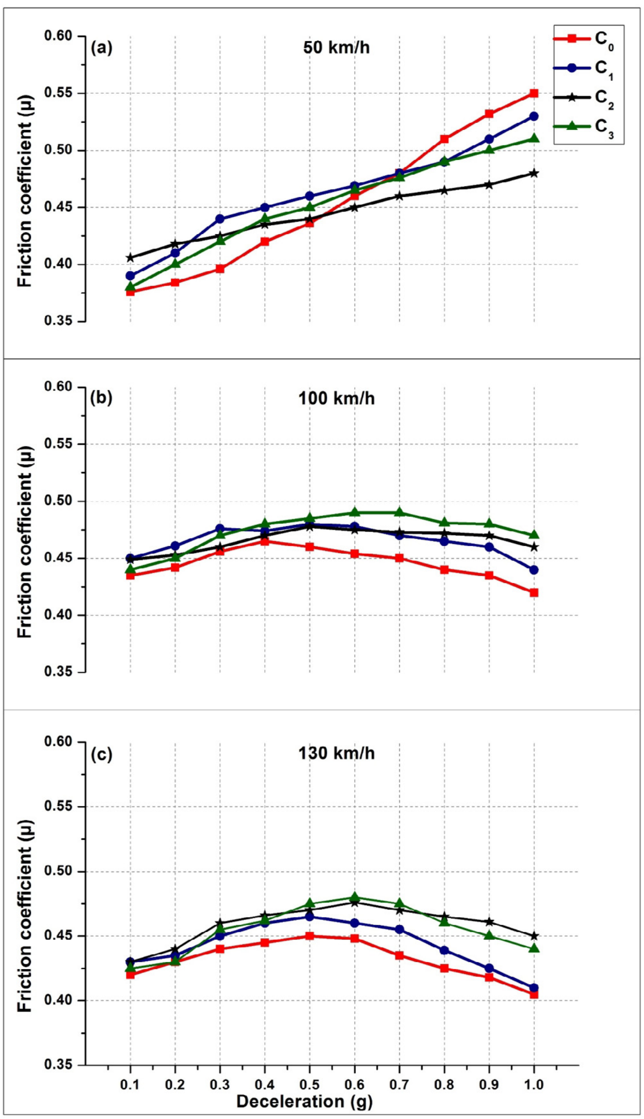

Fig. 3. Pressure sensitivity of the developed composites.

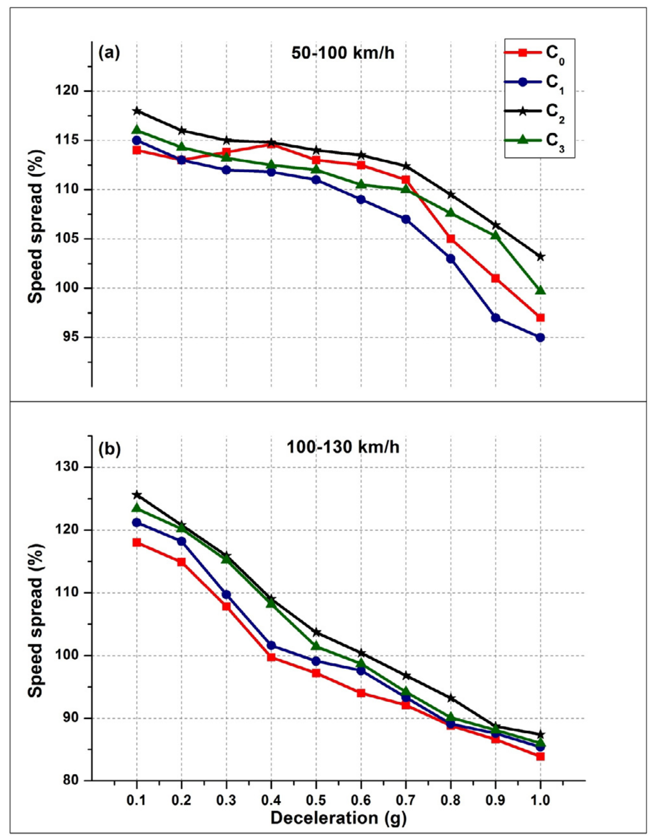

Fig. 4. Speed sensitivity of the friction composites at (a) mild condition (b) severe condition.

materials, SS are found to be in the range of 95–118%. The speed spread declined up to 0.5 (g) deceleration with lesser variations. Compared to all friction composites developed 'C_2' exhibited a better performance. During the severe condition (shown in Fig. 4b) the trends were not much different. SS are found to be in the range of 128–85%. 'C_2' and 'C_3' composites exhibited a similar performance, while the 'C_0' and 'C_1' composites showed a little variation. The speed spread deteriorated profoundly between deceleration values of 0.4 and 0.6 after which there was a gradual decline in the performance of all the BFM composites. The fluctuations of the 'μ' can be attributed mainly due to the high heat resistance of the $CaSO_4$ whiskers and vibration due to the frictional force generated [14].

From the effectiveness studies, It was observed that the μ of all the developed BFMs decreased with respect to the applied speed and pressure which attuned with past literatures [5,40,41]. The performance of μ for all the BFM composites varied with respect to the speed change and also the speed sensitivity varied depending on the BFM formulations and its raw materials.

(b) Temperature sensitivity of the BFM composite

The effect of temperature on the fade and recovery of the developed BFMs was observed in this study.

3.2.3. Fade studies

Increase in temperature led to a drop in frictional performance; such a characteristic is defined as 'fade'. The effect of fade on the developed BFM composites is shown in Fig. 5. All the developed friction composites attributed to a loss in frictional performance from 7th to 10th braking cycles. It was mainly due to the rise in temperature which is referred to as thermal fade, due to the fade there is a deep decrease in the frictional performance of all the developed BFM composite. From the Fig. 5, it is noted that the composite 'C_0' records a highest fade (0.37), this is mainly due to the absence of high heat resistant $CaSO_4$ whiskers on the 'C_0' composition. Hence, it can be concluded that the heat generated at the interface decomposes the organic polymer matrix due to the lack of bonding strength of $CaSO_4$ whiskers [39]. The composite 'C_2' showed a better friction performance during the fade studies than the other BFM composites.

It was observed from the fade studies, that the inappropriate braking is the major cause behind the inferior frictional performance during 'fade' irrespective of the composition.

3.2.4. Recovery studies

The effect of recovery on the developed BFM composites is shown in Fig. 6. All the friction composites started to recover its frictional performance from 7th to 12th braking cycles. The porosity has a greater influence on the thermal properties since the pore size and distribution can change the thermal conductivity [42]. In general, the primary plateaus are the load bearing elements of the brake friction materials. During breaking the fibers and various organic ingredients are exposed and it results in the formation of primary plateaus. The shining patches on the surfaces are the caused due to the bake transfer and retransfer of organic ingredients which is referred as secondary plateaus. In our scenario the formation of secondary plateaus is lesser compared to the primary plateaus. Porosity provides way for heat dissipation; hence the formation of secondary plateaus will be less [43]. The heat dissipation is more due the increase in porosity which enhances the faster recovery rate (Table 4 and Fig. 6). All the composites with calcium sulfate have only small variations in porosity. Normally after the fade cycle the blower will be switched on in the dyno to reduce the temperatures of the disc and drum. Faster the recovery of frictional characteristics the better is the recovery rate. In our recovery tests all the friction composites with calcium sulfate whiskers exhibited quicker recovery rate, hence it was concluded as better recovery. The C_1 and C_3 composites has the higher porosity values, which in turn enhanced the recovery rates. After the fade cycle the speed of regain of frictional characteristics is the recovery rate. In the case of C_1 the recovery happened at the second braking, but in case of C_3 the recovery happened at third braking. It is because the porosity values of C_1 was higher than that of C_3. The frictional performance during 'recovery' of all the developed composites showed a similar trend with few variations.

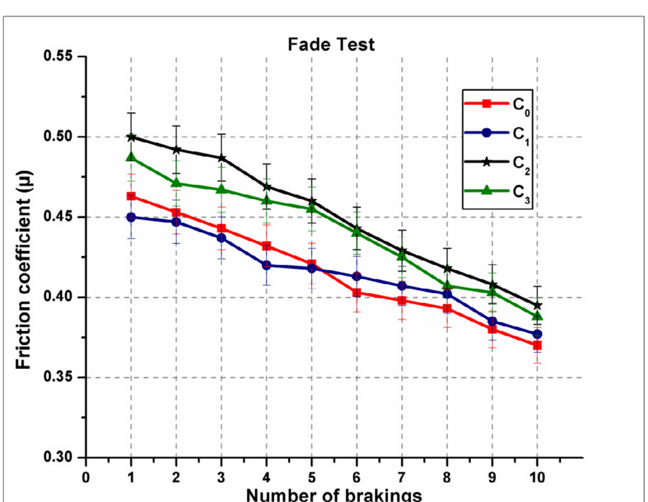

Fig. 5. Fade of the developed BFM composites.

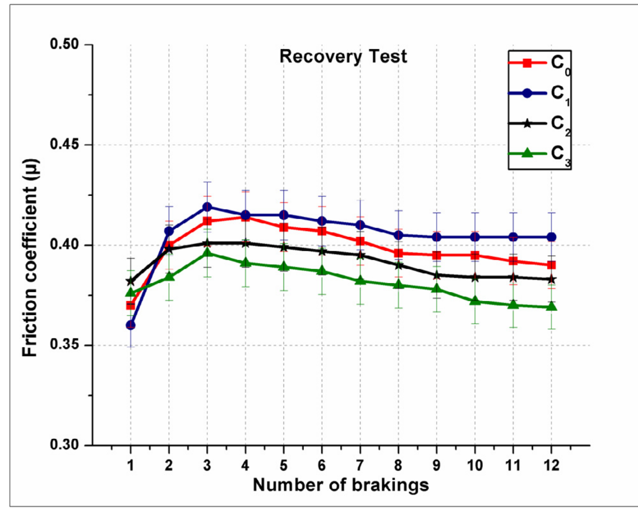

Fig. 6. Recovery of the developed BFM composites.

During recovery, the composite 'C_1' with 5% of $CaSO_4$ whiskers suffered from whisker pull-out which, in turn, absorbed the fracture energy, thus preventing fast failure of the composite matrix [31]. It was observed that the whisker pull-out helped in efficient stress transfer leading to performance recovery. From the recovery test, it was observed that the primary reason behind the better recovery behaviour of the BFM composite was the polymer matrix with presence of whiskers and aramid reinforcements in the polymer matrix.

3.2.5. Investigation on friction parameters

The frictional performance parameters are acquired from the fade and recovery studies are presented in Fig. 7. For an acceptable friction material, it should express lesser fade and faster recovery [44]. The composite 'C_2' exhibited lesser fade and the composite 'C_1' recovered faster compared with other friction material composites developed. The composite 'C_0' exhibited a higher fade rate but the recovery rates of composite 'C_1' were found to be better.

3.3. SEM analysis of worn-out surface

Fig. 8 shows the SEM images of worn out surfaces of the developed BFM pads. All the worn-out composites had uneven surfaces

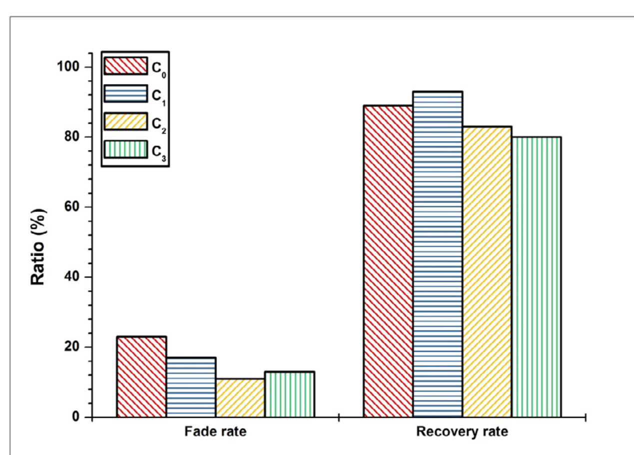

Fig. 7. Friction parameters of BFM composites.

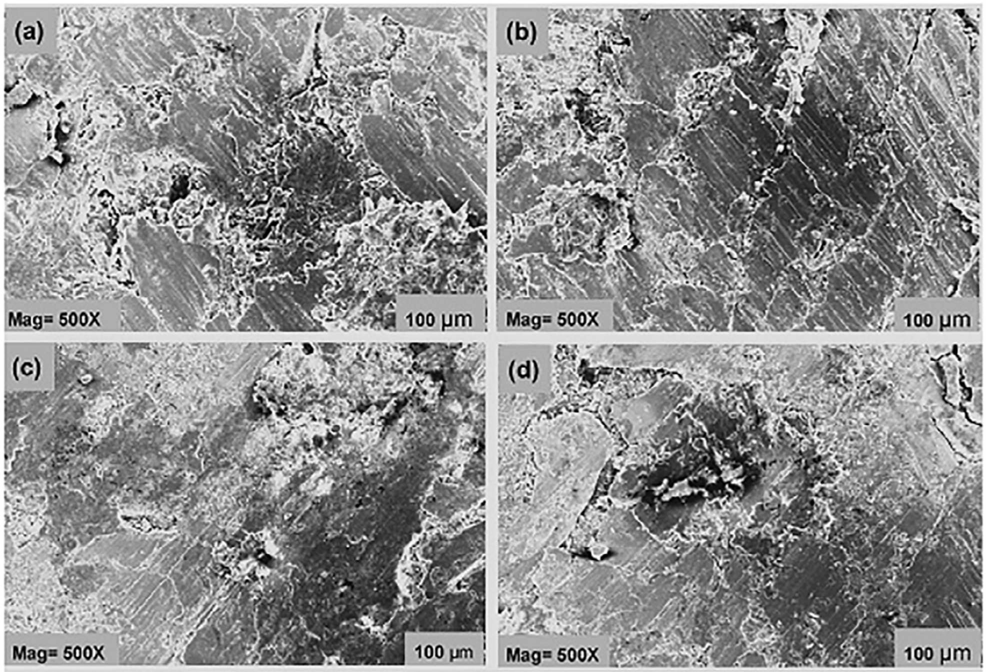

Fig. 8. SEM micrographs showing the worn surfaces of (a) C_0 (b) C_1 (c) C_2 (d) C_3.

with small debris. Huge frictional forces generated at the frictional interface deformed the polymer matrix and the whiskers were pulled-out. It absorbed the shear stress and the energy from the load applied. In the worn-out surfaces of the composites with whiskers, both the Secondary and primary plateaus were found. Formation of secondary plateaus on the worn-out surfaces of the pads enhanced the wear resistance. 'C_0' composite had the maximum wear due to the absence of whiskers. It is clearly seen in the Fig. 8, that the debris on the worn-out surface induced a three-body abrasive wear. From SEM micrographs of the worn surfaces of the composite C_1 it is found that there are some smooth surfaces with lesser cracks. From the cracks the fibers and whiskers were pulled out which acts as a load bearing medium in the friction interface. Those smooth surfaces is due to the back transfer for materials from the counter part to the pads. The smooth patches are the secondary plateaus, which are mostly found in the worn-out surface of the composite C_1. Hence, the composite with 5 wt% of $CaSO_4$ whiskers (C_1) showed the best improvement in wear resistance. All the composites with whiskers had some smooth layers on the surface which were mainly due to the secondary plateaus. Overall, the inclusion of whiskers on the BFM increased the wear resistance.

4. Conclusion

Four friction materials containing increasing amounts (5%, 10% and 15%) of $CaSO_4$ whiskers were developed and its effects on the tribological properties were investigated, it was observed that:

- All the tribological properties showed a better improvement with the inclusion of $CaSO_4$ whiskers. The composite C_2 presented the most stable coefficient of friction regarding to its variations with speed and deceleration rate, as well it presented the higher speed spread and the lower value of fade ration among all specimens, while for the recovery ratio of the composite C_1 presented the best result. The inclusion of $CaSO_4$ whiskers showed a better improvement on the physical and chemical properties of the BFMs.
- The friction composites containing $CaSO_4$ whiskers exhibited lesser wear compared to the composite without $CaSO_4$ whiskers. This was mainly possible due to the smoother surface containing the secondary plateaus and the combination of $CaSO_4$ whiskers in the polymer matrix of a BFM.
- Among the four composites studied, the one with 10 wt% of $CaSO_4$ whiskers (C_2) showed stable friction coefficient even at high sliding speeds.
- The composite with 5 wt% of $CaSO_4$ whiskers (C_1) showed lesser wear compared to the other composites.

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