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Influence of nano-potassium titanate particles on the performance of NAO brake-pads

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ABSTRACT

The present era is dominated by nano-materials. Researchers are keen to explore the potential of nano-particles (NPs) in composites because of their intensive interaction with the matrix and surfaces due to very large surface area.

Potassium titanate (KT) is used as a filler in a good quality non-asbestos organic (NAO) friction materials (FMs). Based on a little information available in the literature, it is claimed to reduce friction-fluctuations, fade and wear. However, exact role of KT in FMs is not so clear due to contradictory findings. Interestingly, no paper is available on exploration of NPs of KT in FMs.

Keeping this in view, two realistic multi-ingredient NAO FMs in the form of brake-pads with identical compositions but differing in size of a theme ingredient, KT were developed. One composite was without KT (K_0), while two composites containing 3% micro and 3% nano-KT particles were designated as K_M and K_N. These were characterized for physical, mechanical and tribological performance. Initial tribo-evaluation of composites was done on a reduced scale prototype (RSP) under various operating conditions. Final tribo-performance was evaluated on a full scale brake dynamometer following JASO C 406 Schedule using gray cast iron disc. Various performance parameters such as performance μ, fade μ, recovery μ; fade ratio, recovery ratio, wear resistance etc. were used to evaluate the performance of FMs.

Both testing data (prototype and realistic machine) showed that almost all performance parameters were significantly and beneficially affected due to NPs of KT. Nano-KT particles proved to improve performance μ and wear resistance significantly compared to its micro-partner. Worn surface analysis of pads and discs was done to understand wear mechanism by SEM and EDAX technique. It was finally concluded that Nano-KT particles have a potential to enhance performance of FMs significantly.

1. Introduction

Ideal Non-asbestos organic (NAO) FMs must satisfy certain performance requirements such as desired (0.35–0.45) and stable value of coefficient of friction (μ) under various operating and environmental conditions like applied brake pressure, driving speed, increase in disc and pad temperature, humidity etc., apart from counterface friendliness, resistance to wear, fade, squeal, judder etc., simultaneously good recovery performance and damping capacity etc. [1]. To achieve these various conflicting performance requirements NAO FMs are made up with various ingredients (10–20), which are broadly classified into four main classes viz. resin/binder, fibers, fillers and friction modifiers. A lot of new ingredients in varying amounts and combinations are tried to achieve better and better performance.

Potassium titanate (KT) was initially tried in NAO FMs as a partial replacement for asbestos, an unchallenged ingredient in FMs, which was declared as carcinogen [2,3]. It was reported that KT helps in reducing friction fluctuations [4]. Halberstadt et al. [5] reported that partial replacement of asbestos fibers by KT fibers with adjustment of resin in brake linings improved the friction and wear performance at high temperature. Cho et al. [6] studied relative influence of various ingredients on tribo-properties of brake lining and reported that KT reduced μ due to morphology and improved fade resistance. Kim et al. [7] reported the synergism of KT whiskers (KTW) and aramid pulp providing good friction stability and wear resistance (W_R). Cho et al. [8] reported that morphology of KT played an important role in the transfer film formation and tribo-properties. Kumar et al. [9] concluded that for the best fade and recovery (F&R) performance and W_R, 5–7.5% aramid fibers and more than 25% KTW was the best combination, working synergistically. However, most of the times, these reports contradict with the findings from others. Hence exact role of KT particles in FMs is not yet so clear. Moreover, recently KT fibers

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Table 1
Literature on nano-materials used in FMs.

Nano-Materials used	Brief contents	Ref.
SBR & NBR Powder (5%)	Improved μ and W_R	[12]
Carbon Nanotube (CNTs)	CNT increased bulk density and hardness while it decreased porosity, wear rate and average μ.	[13]
CNTs (1.7, 4.7, & 8.5 wt.%)	With increase in % of CNTs contents, high temperature stability, damping capacity & W_R increased at the cost of decreased μ.	[14]
Cu (2%)	Inclusion of 2% Nano-Cu increased density, hardness, thermal conductivity, W_R, F_R, counter face friendliness and recovery performance.	[15]
Graphite (2%)	Inclusion of Nano-graphite led to a stable μ and improvement in W_R & heat resistance (11% and 25% respectively).	[16]
Al_2O_3, SiO_2, SiC (2%)	Nano-abrasives significantly improved friction and wear performance as compared to micro abrasives. Nano-SiC led to best combination of friction properties.	[17]
Nanoclay & Multi-Wall Carbon Nanotube (MWCNT)	MWCNT enhanced the friction and fade performance, but depressed the wear performance, whereas nano-clay improved the wear and recovery performance but simultaneously depressed the friction performance.	[18]

and whiskers were stamped as carcinogens by the International Agency for Research on Cancer (IARC) and hence Hikichi of Akebono Brake Industry [10] produced KT in the form of powders, with the same frictional performance and strength as fibrous KT [11]. Not much is reported on performance of KT powders in the literature though KT is used in some formulations of commercial FMs. Neither anything is reported on the influence of size of KT particles on the performance properties of NAO FMs in an open literature.

The present era is dominated by nano-materials. Researchers are keen to explore the potential of nano-particles (NPs) in the composites because of their intensive interaction with the matrix and surfaces. However very few studies are reported on the potential of NPs in FMs [12–18] as shown in Table 1. Interestingly, no paper is available on exploration of NPs of KT in FMs with realistic compositions.

Keeping this in view two realistic multi-ingredient NAO FMs in the form of brake-pads with identical composition but differing in size of a theme ingredient, KT were developed. One composite without KT was developed for the sake of comparison. These composites were characterized for physical, mechanical and tribological performance. Initial tribo-evaluation of composites was done on a reduced scale prototype (RSP) under various operating conditions. Final tribo-performance was evaluated on a full scale brake inertia dynamometer following Japanese Automobile Standards (JASO C 406) using disc of gray cast iron. Various performance parameters such as performance μ, fade μ, recovery μ; fade ratio, recovery ratio, wear resistance etc. were used to evaluate the role of size of KT powder in FMs.

2. Materials and methodology

Details of selected ingredients are given in Table 2. Technical specifications of theme ingredients are given in Table 3 (Suppliers' data).

2.1. Fabrication of the nano-mixture

Distilled water was used as a dispersion medium for nano-KT particles. 1 g of KT was properly dispersed in 150 ml distilled water by using high intensity probe sonicator (Chromtech, probe diameter 10 mm) for 20 minutes to de-agglomerate nano-KT particles. Slowly inclusion of Barite paste (8 g) was added step-wise to the nano-suspension followed by further sonication for 10 minutes to form a slurry of barite, NPs and distilled water. Same procedure was repeated two more times to complete the combination of 27% paste (3% nano-KT and 24% barite) in distilled water. This mixture was then dried in an oven at 70–80 °C followed by grinding to convert in powder form.

Table 2
Source details of ingredients used in FMs.

Ingredients	Source
Binder	
Phenolic resin (150 Mesh)	Arora Chemical Works, Ghaziabad, India.
Fibers	
Rockwool RB-250 (L. – 125 ± 25 μm; Dia. – 5.5 μm)	Lapinus Fibers, Netherlands.
Glass fiber (L. – 3-6 mm)	Starke International Ltd., Delhi, India.
PAN fiber-CFF 110-1 (Dia. – 10 μm)	Sterling Fibers Inc. USA.
Twaron para aramid pulp 1099 (L. – 1.05 mm)	Teijin Aramid, Netherlands.
Functional fillers	
Brass (100–200 μm)	Starke International Ltd., Delhi, India.
Cashew dust (20–100 Mesh)	Satya Cashew Chemicals, Chennai, India.
Vermiculite (100–350 μm)	Starke International Ltd., Delhi, India.
Potassium Titanate (15–25 μm) (micro)	Otsuka Chemicals.
Potassium Titanate (100 nm) (nano)	Nanoshel LLC, USA.
Inert filler	
Barite (10–100 μm)	Starke International Ltd., Delhi, India.
Friction modifiers	
Graphite (D90 73.3)	Starke International Ltd., Delhi, India.
Alumina 48–100 μm	Hindalco Industries, Delhi, India.

Table 3
Technical details of theme ingredients [Supplier's data].

Properties	Micro- KT powder	Nano- KT powder
Appearance	White powder	White powder
Chemical Name	Lithium potassium titanium oxide	Potassium Titanate
Molecular formula	K(0.5–0.7) Li(0.27) Ti(1.73) O(3.8–3.95)	K_2TiO_3
Avg. Particle Size	15–25 μm	100 nm
True Density (g/cm^3)	3.4	3.1
Melting Point (°C)	1150–1200	1615

2.2. Formulation design and fabrication of the composites

73 wt. % of ingredients of all the three components were fixed. Remaining 27 wt. % contained 3 wt. % theme ingredient in nano and micron sizes while 24 wt. % was space filler barite. Composites containing 3 wt. % micro-KT and 3 wt. % nano-KT were designated as K_M and K_N respectively. For the sake of comparison, one composite was developed without KT powder containing 73 wt. % parent composition and 27 wt. % space filler barite. Formulation design and designation of composites are shown in Table 4.

Fabrication of composites was done as per standard practice

Table 4
Formulations and designations of developed composites.

Ingredients by Wt%	Composites Designation		
	K_0	K_M	K_N
Parent Composition[a]	73	73	73
Micro KT powder	–	3	–
Nano KT powder	–	–	3
Barite	27	24	24

[a] Parent composition - binder: fibers: functional fillers: friction modifiers – 10:21:30:12

Table 5
Mixing schedule of ingredients in plough shear mixture.

Ingredients	Mixing duration (minutes)
Aramid pulp and glass Fibers	1
Aramid-glass fibers mixture + Barite	5
Previous + Rockwool + PAN **(Batch-I)**	10
CNSL + Graphite + Alumina + Vermiculite + Metal powder + Resin **(Batch-II)**	4
(Batch I) + (Batch-II)	2
Total Duration	22 minutes

reported elsewhere in the literature [19–22]. For homogenized mixing all ingredients were mixed in plough type shear mixer with 2800 rpm chopper speed and 250 rpm feeder speed for 22 minutes duration following mixing schedule shown in Table 5. After mixing 60 g mixture of each composite was cold pressed in the shape of brake pads by applying manual pressure in the pre-former. Heat curing of preformed mixture was done in hot press (Kalson, max. 150 t, 550 °C), in eight cavity mold under a curing temperature of 160–165 °C and a uniform pressure of 140 kgf/cm^2 for 9 minutes duration. Before initiation of curing process five intermittent 'breathings' were allowed to expel volatiles to avoid air gaps or voids in the brake pads. Then the brake pads were post cured in an oven at 120 °C for two hours and at 160 °C for five hours. Finally grinding and polishing of brake pad surfaces were done to achieve required thickness and surface finishing of pads.

2.3. Characterization of composites

Developed FM composites were characterized for physical (density, porosity JIS D 4418:1996) and chemical (acetone extraction ASTM D 494) properties as per standard practices followed for FMs characterization [19–22]. Three tests were carried out for each composite and average results of each characterization test is shown in Table 6.

2.4. Test set-up for tribo-testing

Initial tribological testing of FM composites were carried out on reduced scale prototype (RSP) machine and further realistic tribo-performance evaluation of composites were done on a full scale brake inertia dynamometer (Dyno).

Table 6
Physical and chemical properties of the composite.

Properties	K_0	K_M	K_N
Density (g/cc)	1.916	1.873	2.080
Oil porosity (%) (JIS D 4418)	2.36	3.04	2.42
Water porosity (%)(JIS D 4418)	2.89	3.25	3.28
Acetone extraction (%) (ASTM D 494)	2.263	2.248	1.629

2.4.1. Reduced scale prototype (RSP)

The RSP simulates the braking condition of passenger car in city driving mode. The schematic diagram and detailed description of RSP is described elsewhere [17,23]. The tribo-couple used by RSP consists of two square shaped (24 mm × 24 mm) specimens cut from brake-pad. A gray cast iron disc of Maruti Esteem car with composition:- C: 3.3–3.5; Mn:0.65–0.92; Si:1.85–2.15; S: 0.12; P:0.15; Ni:0.60–0.72; Cr:0.15–0.25; Mo:0.2–0.32 was used. For every composite, new disc was used. Two cut samples were mounted in the assembly holder; held against rotor disc. Both specimens were facing towards the same side of the disc and braking pressure was applied by hydraulically actuated system, which was attached to the assembly holder. The controllable parameters through control panel in the RSP were; applied pressure, braking touch time, disc rotational speed and number of brake applications.

2.4.2. Full scale brake inertia dynamometer (Dyno)

The full scale brake inertia dynamometer (Dyno) was used for realistic evaluation of tribo- performance of FMs. The data generated were collected on computer through data acquisition card NI-6008™ and Labview™ based software. The detailed description and schematic diagram of Dyno is available elsewhere [20,21]. The tribo-couple consists of cast iron disc of Maruti Alto® and two brake pads fixed in the calliper assembly of Maruti Alto®.

2.5. Testing schedule

A test schedule based on ECE R 90 as shown in Table 7 was followed for tribo-performance evaluation on RSP while JASO C 406, the most preferred testing schedule in Asian countries for passenger cars, was employed for tribo-evaluation on Dyno. Before going for effectiveness and F&R testing on RSP and Dyno, bedding of brake pad samples was done against rotor disc to establish at least 80–90% conformal contact between a tribo-couple. Once the conformal contact was established, the rotor disc was replaced with a fresh new disc. In both the machines wear was calculated after completing full test schedule by the weight loss method.

2.5.1. Testing schedule for RSP

The test schedule consists of two parts, first effectiveness part was designed to study the influence of only pressure and speed and second F&R part was designed to study the influence of only temperature by keeping pressure and speed constant. In the first part influence of temperature was eliminated by keeping the disc temperature always below 80 °C at the start of each brake with the help of fan. Similarly in the F&R testing pressure and speed were kept constant to eliminate their effect.

The effectiveness part (total 9 tests) was carried out for three different speeds viz. 36, 45 and 54 kmph and three different pressures viz. 2, 3 and 4 MPa. (One test at each speed under three different pressures). Each test consists of 25 brake applications

Table 7
Testing schedule for RSP.

Description	Speed (kmph)	Pressure (MPa)	Initial Temp. (°C)	No. of brake applications	Fan
Bedding Test	27	1	< 80 °C	100	On
Effectiveness	36, 45, 54	2, 3, 4	< 80 °C	225[a]	Off
Fade & Recovery					
Cold mode	45	3	< 80 °C	20	On
Fade mode	45	3	80 °C[b]	550	Off
Recovery mode	45	3	< 80 °C	30	On

[a] Each pressure at constant speed consists of 25 brake applications.
[b] For first brake only.

with braking duration of one second. For this test of 25 brakings, average of only last 15 values of μ was considered as a representative μ.

The same tribo-couble was continued for further testing of F&R cycle. F&R testing consists of cold mode (20 brakes) followed by fade mode (550 brakes) and recovery mode (30 brakes). In both cold and recovery modes temperature was maintained below 80 °C with the help of fan while it was allowed to rise uninterruptedly in the fade mode.

2.5.2. Testing schedule for Dyno

JASO C 406 was divided into three segments of 172 brake applications viz., effectiveness I, F&R I and F&R II as shown in Table 8. Before starting first segment of effectiveness I, 10 brakes of pre-effect were applied. Effectiveness I was done with three different speeds viz., 50, 80 and 100 kmph and eight different decelerations (0.1–0.8 g). Each deceleration at constant speed consists of three brake applications, thus effectiveness I comprise of 72 brake applications. Average of three μ values was considered as a representative μ for corresponding speed and deceleration.

After completion of effectiveness test, 18 brakes of re-burnishing and baseline check was applied before going for F&R I. F&R I was consist of total 22 brake applications, 10 of fade and 12 of recovery. Temperature was allowed to rise uninterruptedly in the fade I while speed (80 kmph) and deceleration (0.45 g) were kept constant. Similarly in the recovery I speed (50 kmph) and deceleration (0.3 g) was constant and temperature was maintained below 80 °C with the help of air blower.

Before starting F&R II burnt surface of brake pads in F&R I was cleaned by application of 28 brakes of re-burnishing and baseline check. Operational parameters of F&R II was same as F&R I.

2.5.3. Definitions of performance parameters

Definitions and formulae for calculating performance parameters used in the current study are given in Appendix A.

3. Results and discussion

3.1. Physical, chemical and mechanical properties

The characterization results of developed composites are shown in Table 6. It shows the density of composite K_M, decreased with the inclusion of 3 wt. % of micro-KT as compared to K_0. This was due to the replacement of high density material (barite, 4.5 g/cc) by low density material (micro-KT, 3.4 g/cc). Acetone extraction shows percentage of uncured resin which was in acceptable range as per industrial tolerance limit of 4–5%. Curing of composite was improved by inclusion of micro-KT while further improvement in curing was observed by replacing micro-KT with nano-KT.

3.2. Tribo-performance of developed composites

In this section, frictional and wear performance of FM composites on RSP and Dyno is discussed.

3.2.1. Tribo-performance on RSP

In this section, effectiveness studies, fade and recovery studies and worn surface analysis of composites on RSP is discussed.

3.2.1.1. Effectiveness studies (μ sensitivity towards pressure and speed). Effectiveness studies covers analysis of sensitivity of μ towards pressure and speed. μ should be least sensitive to any parameter. Brake fade is referred to deterioration of braking performance. Performance of materials may be deteriorated with increase in pressure (pressure fade), speed (speed fade) and temperature (temperature fade). The deterioration in μ due to fade is the important issue during braking in vehicles. Ideally, this deterioration should be minimal and level of μ should be constant with respect to the operating parameters for the comfort of a driver and reliability and repeatability of braking.

3.2.1.1.1. Friction performance of the composites. As seen in Fig. 1 μ_{avg} of the composites was in the range of 0.46–0.62 and decreased with increase in pressure and speed for all composites, which is in tune to the trends in literature [24,25]. The pressure fade is argued due to disproportionally more increase in real area of contact with increment in pressure in case of polymers [26,27]. It was also observed from Fig. 1 that the pressure fade increased with speed. Increase in frictional heat at the surfaces of a tribo-couple with severity of operating conditions lead to degradation of polymer and organic materials at the asperity contact. This degraded matter from the pad surface gets transferred to the disc surface and again back transferred from the disc surface to the pad surface in the form of secondary plateaus which led to decline in μ [28]. The salient features from Fig. 1 (composition specific) are as follows.

- Inclusion of KT (both nano and micro sizes) at the cost of barite in equal amount improved the μ_{avg} of the composites. Nano-KT prove more influential in this aspect.
- Ideal material should have minimal changes in μ_{avg} with any operating parameter and hence lowest slope. In this respect the performance order was; $K_N > K_M > K_0$.

Table 8
Testing schedule for Dynamometer.

Description	Speed (kmph)	Deceleration (g)	Initial Temp. (°C)	No. of brake applications	Air Blower
Bedding Test	65	0.35	100 °C	200	Off
Pre-effect	50	0.3	< 80 °C	10	Off
Effectiveness-I	50, 80, 100	0.1 to 0.8	< 80 °C	72[a]	Off
Fade & Recovery-I					
Re-Burnish-I	50	0.3	80 °C	15	Off
Baseline check	50	0.3	80 °C	3	Off
Fade-I	80	0.45	80 °C[b]	10	Off
Recovery-I	50	0.3	< 80 °C	12	On
Fade & Recovery-II					
Re-Burnish-II	50	0.3	80 °C	25	Off
Baseline check	50	0.3	80 °C	3	Off
Fade-II	80	0.45	80 °C[b]	10	Off
Recovery-II	50	0.3	< 80 °C	12	On

[a] Each deceleration at constant speed consists of three brake applications.
[b] For first brake only.

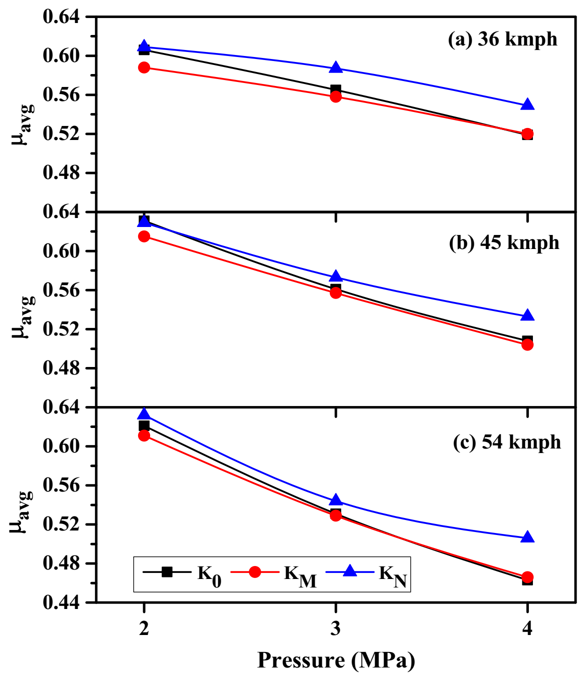

Fig. 1. Friction coefficient as a function of braking pressure; (a) 36 kmph, (b) 45 kmph and (c) 54 kmph.

3.2.1.1.2. Sensitivity of μ towards pressure and speed. Fig. 2 shows the sensitivity of μ towards pressure at mild condition (transition from 2–3 MPa- Fig. 2a) and at severe condition (transition from 2–4 MPa- Fig. 2b). Similarly the sensitivity of μ towards speed at mild condition (transition from 36–45 kmph- Fig. 2c) and at severe condition (transition from 36–54 kmph- Fig. 2d) can be seen. Ideally, for both pressure and speed, the data points should be as close as possible to the X axis confirming least amount of fluctuations in μ. There was no significant difference in performance based on compositions. Following are the salient comments.

- Composites are more sensitive to selected pressure transitions compared to speed transitions since magnitude of fluctuations in μ (0.022–0.158) are higher in first case.
- With increase in severity of parameter (pressure or speed), sensitivity of μ increased (lines are away from the X axis)
- Performance order in general was $K_N > K_M > K_0$ (barring Fig. 2d), though difference was not so significant.

3.2.1.2. Fade and recovery studies (μ sensitivity towards temperature). Fig. 3(a) shows performance μ, fade μ and recovery μ for all composites. For best performing material, all values of μ should be higher. The performance order of composites was as follows:

- Performance μ:- K_0 (0.573) > K_N (0.566) > K_M (0.564)
- Fade μ:- K_N (0.512) > K_M (0.495) > K_0 (0.473)
- Recovery μ:- K_0 (0.589) > K_N (0.583) > K_M (0.567)

Results showed that inclusion of KT (micro and nano sizes) led to lowering of performance μ indicating slightly lubricating effect. However, it was also responsible for restricting the fade in μ.The fade μ of K_N was highest followed by $K_{M.}$ This was a special advantage of KT powder, nano-size being better. Recovery μ of nano-KT was higher than micro-KT.

Fig. 3(b) shows a change in level of μ (friction fluctuations δμ) due to increase in temperature and severe operating conditions. Ideally, δμ should be as small as possible. Results showed that micro-KT inclusion reduced δμ while further improvement was found with the replacement of micro-KT by nano-KT. Nano-KT composite proved best amongst all composite for least friction fluctuations.

Fig. 4 shows % fade and % recovery of composites. Lower values of % fade and maximum recovery of the friction level after fade cycle are most desirable for a good FM. All the three composites showed acceptable range for recovery.

Fade is the important parameter for performance ranking of FMs. It represents deterioration in μ with increase in temperature. Generally, fade occurs by thermal degradation of ingredients due to accumulation of frictional heat on the surfaces of tribo-couple. This degraded material forms sticky film on the disc surface, which

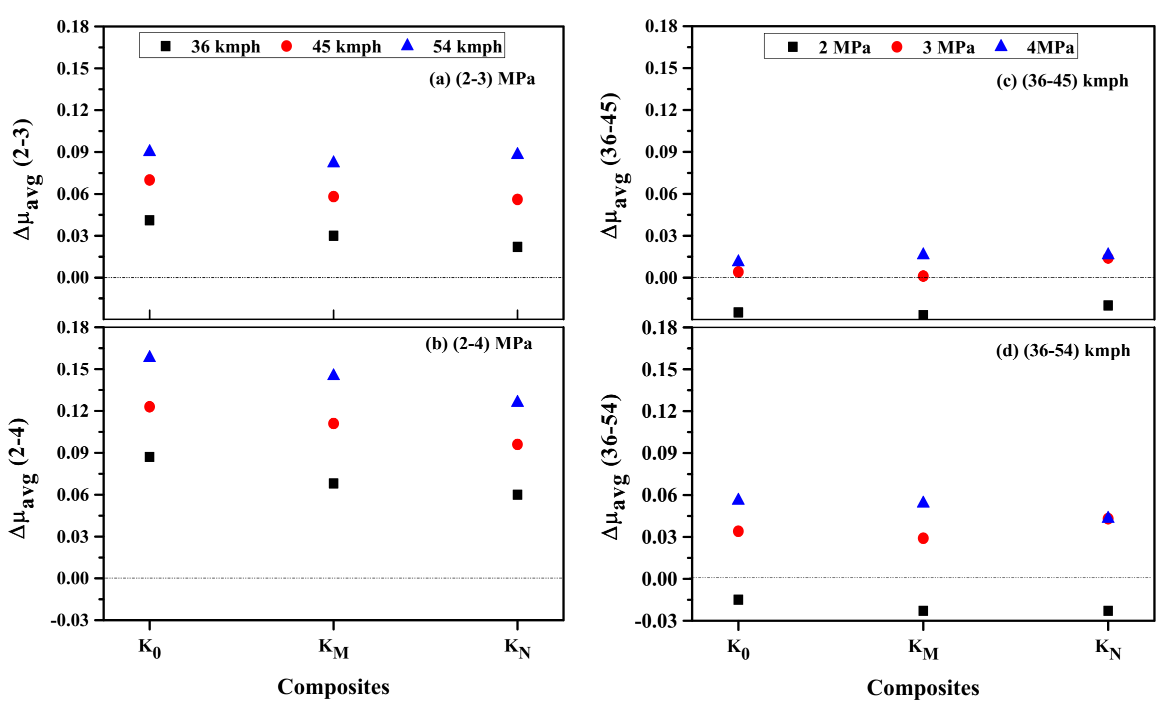

Fig. 2. μ Sensitivity of composites towards the pressure (a & b) and speed (c & d).

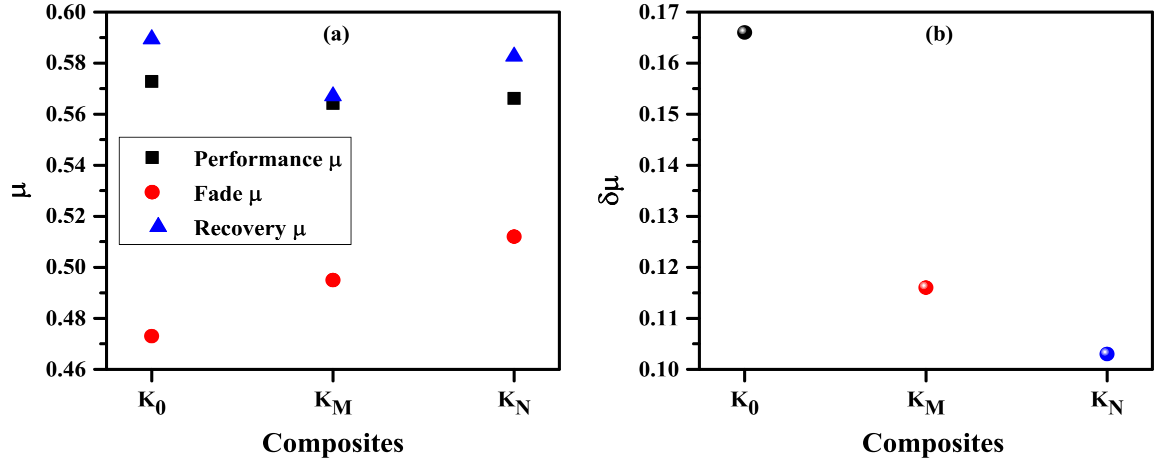

Fig. 3. (a) Performance μ, fade μ and recovery μ of composites (b) Friction fluctuations (δμ) of composites.

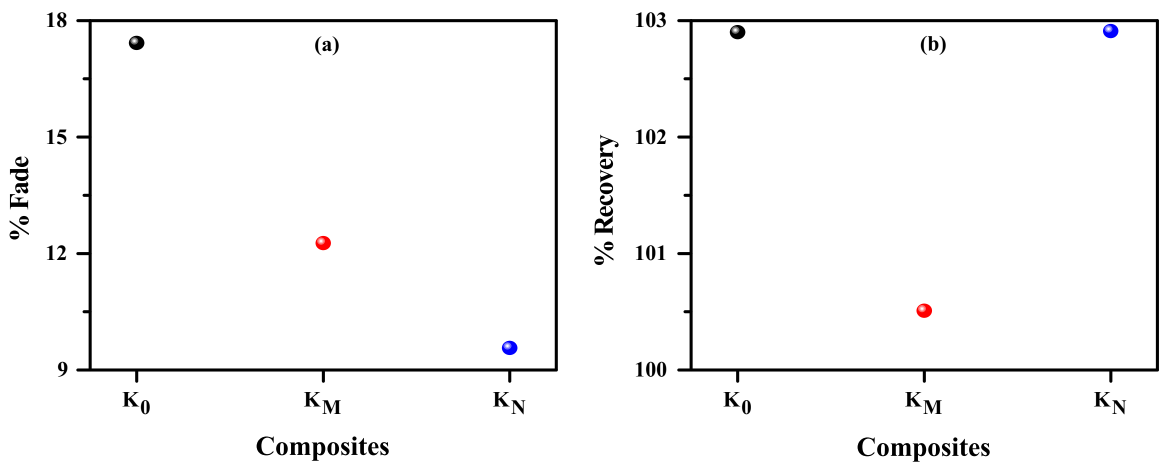

Fig. 4. (a) % Fade of composites (b) % Recovery of composites.

is called as a glaze/transfer film. Glaze is responsible for deterioration in μ and wear performance. Mainly this film defines the tribo-performance and topography of the pad surface. Inclusion of KT reduced % fade approximately from 18 to 12% for micro-KT and to 9% by nano-KT. Thus nano-KT proved very much helpful in reducing fade by almost 50%.

The counter-face friendliness of FMs is decided by certain parameters like maximum disc temperature rise, roughening and scoring, etc. Fig. 5(a) is for disc temperature rise for the composites and the performance order for counter-face friendliness was; K_N (145) > K_M (162) > K_0 (168).

Wear behavior of the composites is shown in Fig. 5(b). Results showed that both micro and nano-forms of KT improved W_R. The performance order was; K_N > > K_M > K_0.

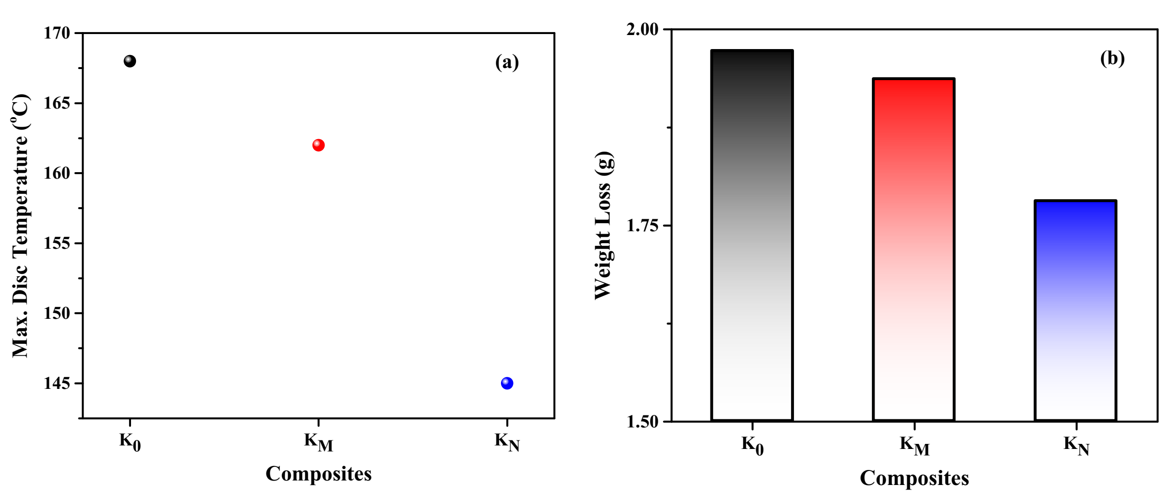

Fig. 5. (a) Maximum disc temperature rise for composites in fade mode and (b) Wear of composites after complete schedule.

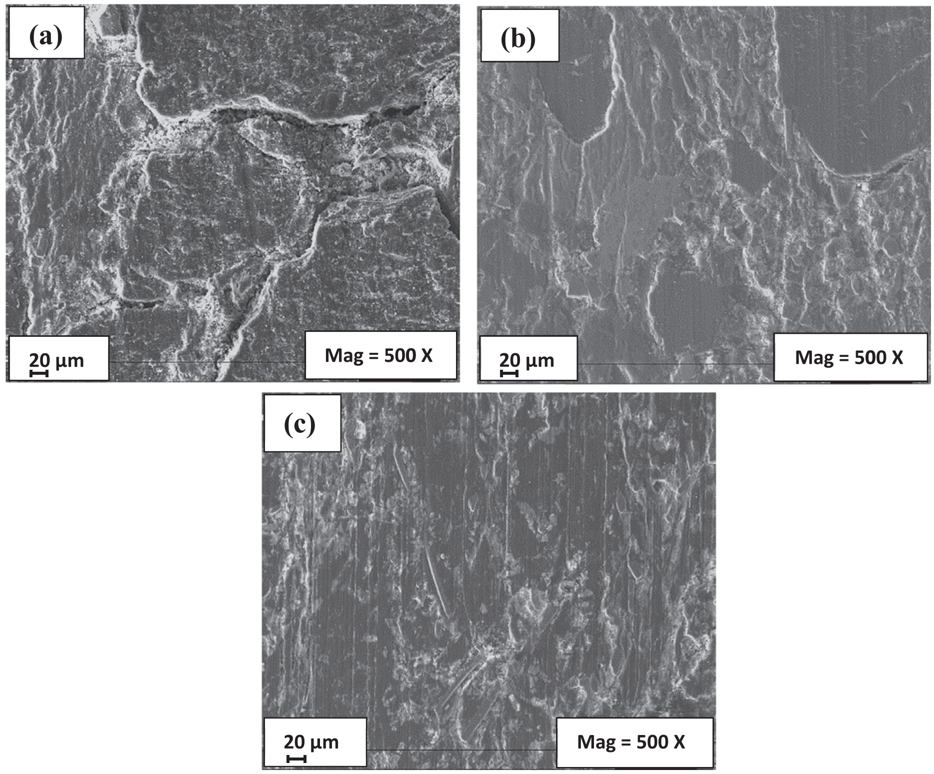

Fig. 6. SEM micrographs of composites (a) K_0 (b) K_M (c) K_N.

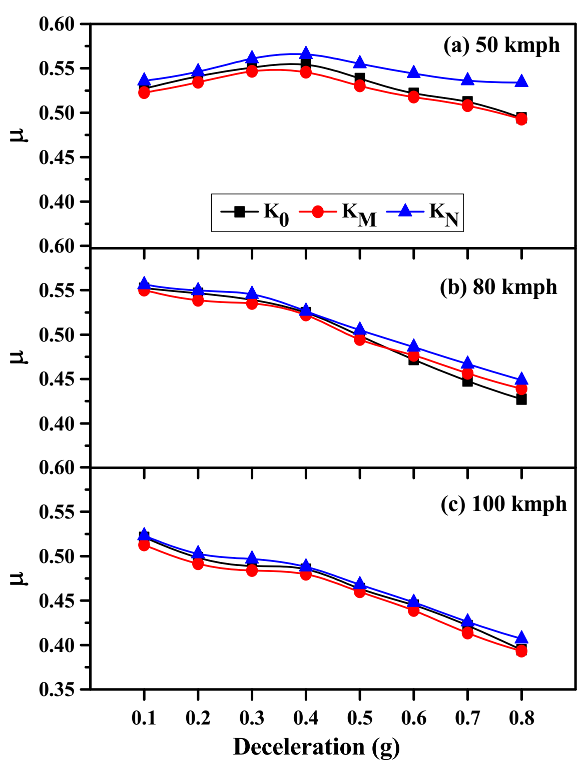

Fig. 7. Variation in μ with increasing deceleration at (a) 50 kmph, (b) 80 kmph and (c) 100 kmph.

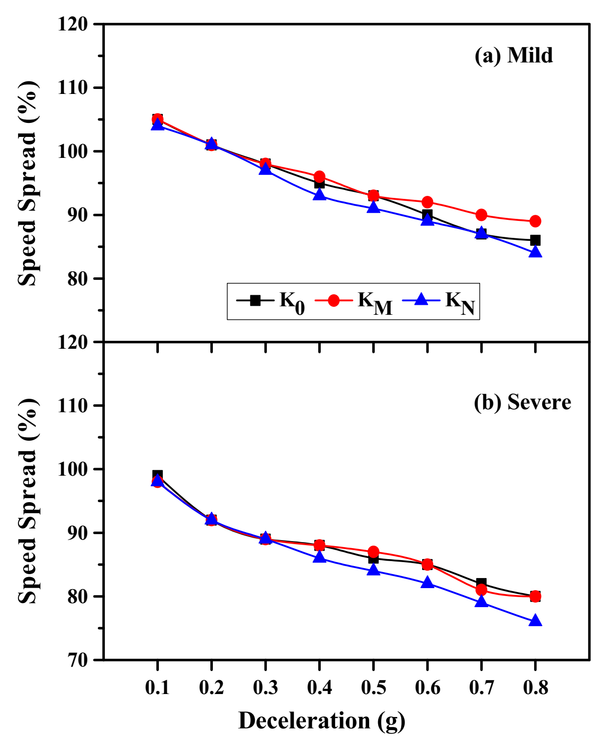

Fig. 8. Variation in speed spread with increasing deceleration at (a) mild conditions (transition from 80 to 50 kmph) and (b) severe conditions (transition from 100 to 50 kmph).

3.2.1.3. SEM analysis of worn surfaces. Fig. 6 shows the SEM micrographs of composites in the increasing order of W_R. The K_0 showed deep cracks on the surface while K_M showed a smooth surface without cracks and typical film transfer. Surface of K_N was ideal showing smooth topography and hardly any evidences of abrupt plucking out of ingredients. The thin film appears to be covering the surface, which could be due to KT.

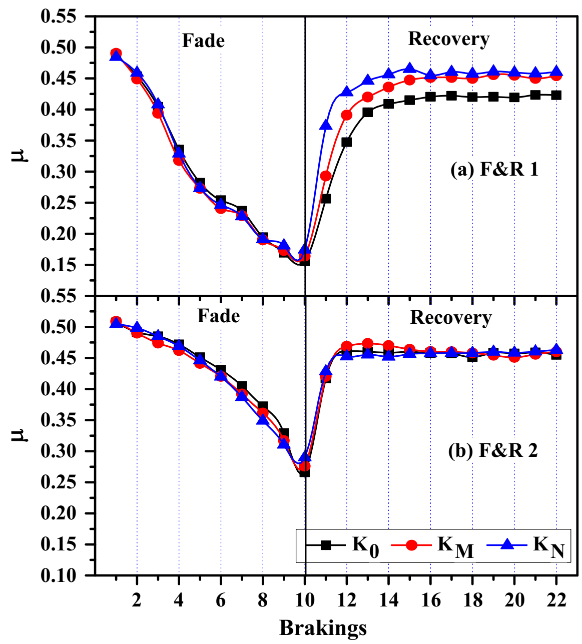

Fig. 9. Fade and recovery behavior of the composites for (a) F&R cycle 1 and (b) F&R cycle 2.

3.2.2. *Tribological performance on Dyno*

In this section, effectiveness, fade and recovery studies and worn surface analysis of composites on Dyno is discussed.

3.2.2.1. Effectiveness studies (μ sensitivity towards pressure and speed). Effectiveness studies on dyno were carried out at three different speeds viz. 50, 80 and 100 kmph and eight different decelerations from 0.1 g to 0.8 g.

3.2.2.1.1. Friction performance of the composites with increasing deceleration –Pressure fade. Fig. 7 shows μ values against eight decelerations for three different speeds. These values are average of two repetitions on dynamometer. Ideal FM should not show any decline in μ with increase in deceleration. The curve should be as straight as possible. In present results, μ of the composites was in the range of 0.56-0.39. At 50 kmph μ initially increased followed by slight decline from 0.4 g. K_N showed lowest decline. Performance of K_0 and K_M was almost similar, K_0 being slightly better. For other speeds also the performance order was same ($K_N > K_0 \geq K_M$).

3.2.2.1.2. Sensitivity of μ towards pressure and speed. Deterioration in μ with increase in deceleration at each constant speed represents the sensitivity of μ towards pressure (shown in Fig. 7). Ideally, the curve should be parallel to the x-axis with minimum slope.

Sensitivity of μ towards speed was defined in terms of % speed spread (% SS). The % SS is the stability in μ when speed changes from first level to a second one. When speed changes from 80 kmph to 50 kmph, it is referred as mild condition (shown in Fig. 8a) while change from 100 kmph to 50 kmph corresponds to severe condition (shown in Fig. 8b). Ideally, Higher the % SS, better is the performance. The slope and undulations of the curve should be minimum. In general, performance decreased with severity of operating conditions. Performance order for % SS was, $K_M > K_0 > K_N$. The FM without KT showed least sensitivity for speed.

3.2.2.2. Fade and recovery studies (μ sensitivity towards temperature). Fig. 9 shows the F&R behaviour of composites for cycle 1 and 2. Ideally, the fade curve should be straight with minimum slope and the μ should be in the range of pre-fade value [20]. In the fade mode temperature of disc was allowed to increase uninterruptedly by switching off the blower. During the recovery cycle, blower was started to maintain the disc temperature below 80° C.

Results showed that fading tendency in second cycle was lower than the first cycle and recovery in second cycle was higher than the first cycle. This is as per general trend for FMs and possibly due to work-hardening of both disc and pad surfaces (due to metallic contents). Composite K_N showed excellent performance amongst all composites in both fade and recovery mode followed by K_M and K_0.

As per the standard pattern of the schedule JASO C406 the following essence of the F&R studies was drawn in the form of Figs. 10–12.

3.2.2.2.1. Fade μ & Recovery μ (higher the better). For both the F&R cycles K_N showed higher level of fade μ amongst all composites and performance order was $K_N > K_M > K_0$ (as shown in Fig. 10). Performance order of recovery μ for first cycle was the same. However, it slightly changed for II cycle ($K_0 > K_M > K_N$).

3.2.2.2.2. % Fade ratio and % Recovery ratio (higher the better). It is one of the most important parameters to measure the variation in μ with increase in temperature and the essence is shown in Fig. 11. The performance order for % fade ratio and % recovery ratio was same ($K_N > K_M > K_0$).

3.2.2.2.3. Friction fluctuations (Δμ) and maximum disc temperature (lower the better). Fig. 12(a) shows Δμ at 50, 80 and 100 kmph and Fig. 12(b) shows maximum disc temperature rise for composites in F&R cycles 1 and 2. Ideally Δμ should be zero. Δμ increased with severity of operating speed. The performance was in the order; $K_N > K_M > K_0$.

In case of rise in temperature of the disc (Fig. 12b), it depends on various complex parameters such as friction level and amount of frictional heat generated and extent of its dissipation with time, which in turn depends on thermal conductivity, diffusivity,

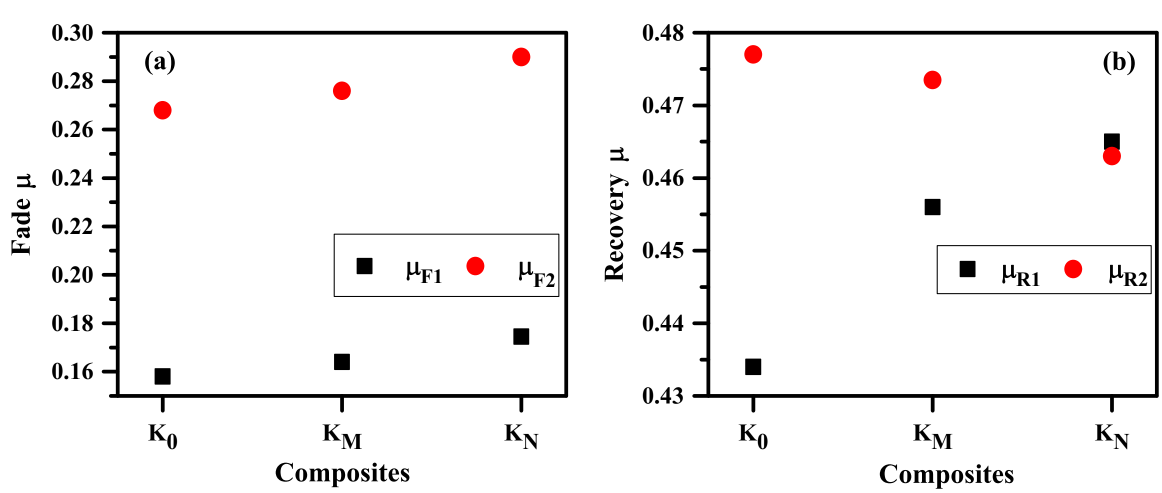

Fig. 10. (a) Fade μ and (b) Recovery μ for F&R cycle 1 and 2.

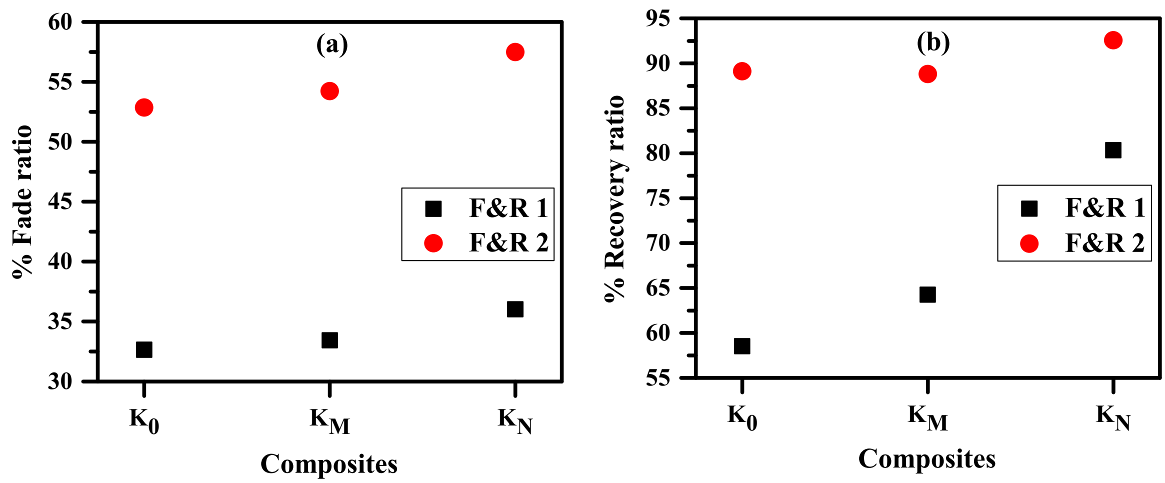

Fig. 11. (a) % Fade ratio and (b) % Recovery ratio for F&R cycle 1 and 2.

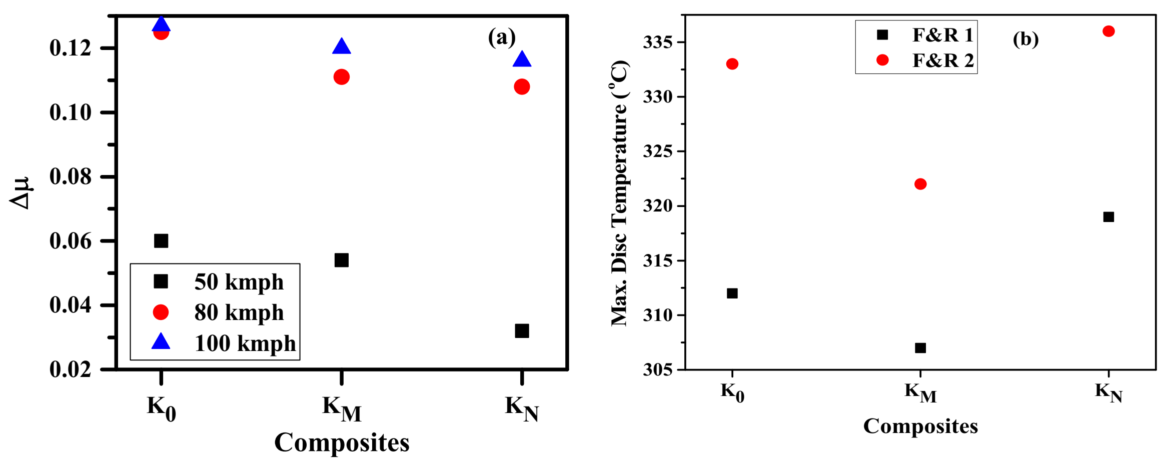

Fig. 12. (a) Δμ at 50 kmph, 80 kmph and 100 kmph (b) Maximum disc temperature rise for composites in F&R cycle 1 & 2.

effusivity etc. of both the surfaces. Composite K_M showed lowest disc temperature rise hence best counter-face friendliness, which could be because of its lowest performance μ and hence lowest amount of frictional heat generated.

Fig. 13 shows wear behavior of the composites (average of two repetitions performed on dynamometer). Though slight improvement was there performance order of W_R was; K_N (4.96) > K_M (5.16) > K_0 (5.26).

3.2.2.3. SEM analysis of worn surfaces. Figs. 14 and 15 show the SEM micrographs of worn out surfaces of brake pads and discs. It seems that for the pad surfaces, topography of K_N was smoothest and without cracks, with least evidence of pulling out of ingredients, cavities indicating consumption of fibers, fillers etc. It clearly showed a very fine film covering the surface. In case of disc surfaces overall, the disc worn against K_N showed smoothers surface and uniform film transfer from the pad. Disc surface due to K_0 was very rough with irregular film transfer.

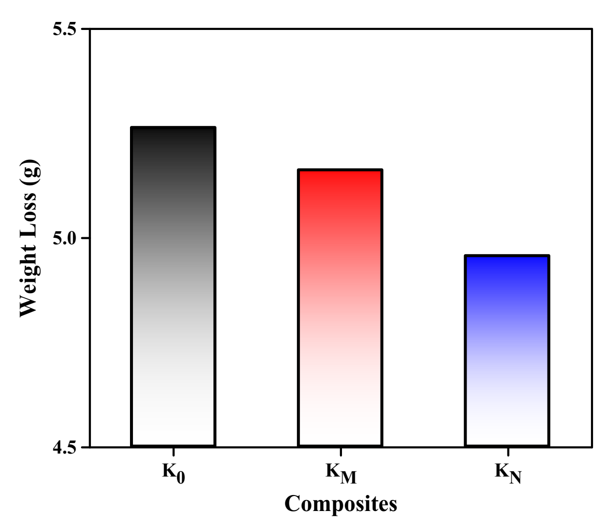

Fig. 13. Wear behavior of the composites.

4. Conclusions

Based on the tribological studies on FM composites containing micro and nano KT conducted on Reduced Scale Prototype (RSP) and brake inertia dynamometer under various operating conditions, following conclusions were drawn.

- Inclusion of 3 wt% KT in FMs decreased overall friction coefficient ($\mu_{performance}$) slightly indicating its lubricating property. Friction fluctuations, on other hand, reduced significantly apart from fade and wear also. Thus, role of KT in FM was clearly seen as a mild solid lubricant which was responsible for reduction in $\mu_{performance}$ (slight), fluctuations in μ and increased wear resistance.

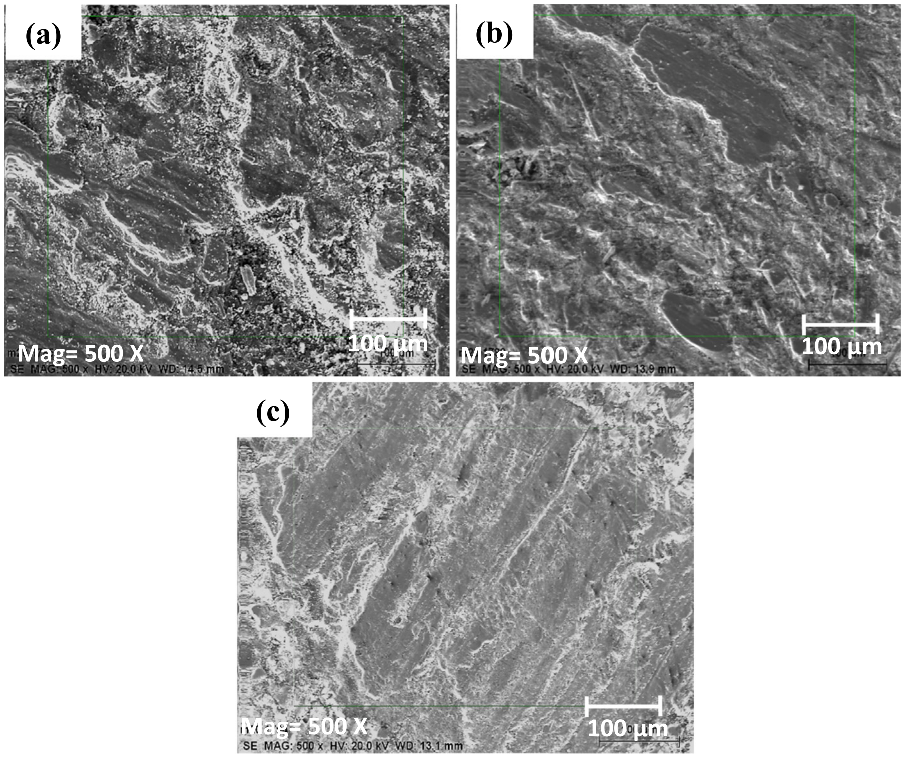

Fig. 14. SEM micrographs of worn surfaces of composites (a) K_0 (b) K_M (c) K_N.

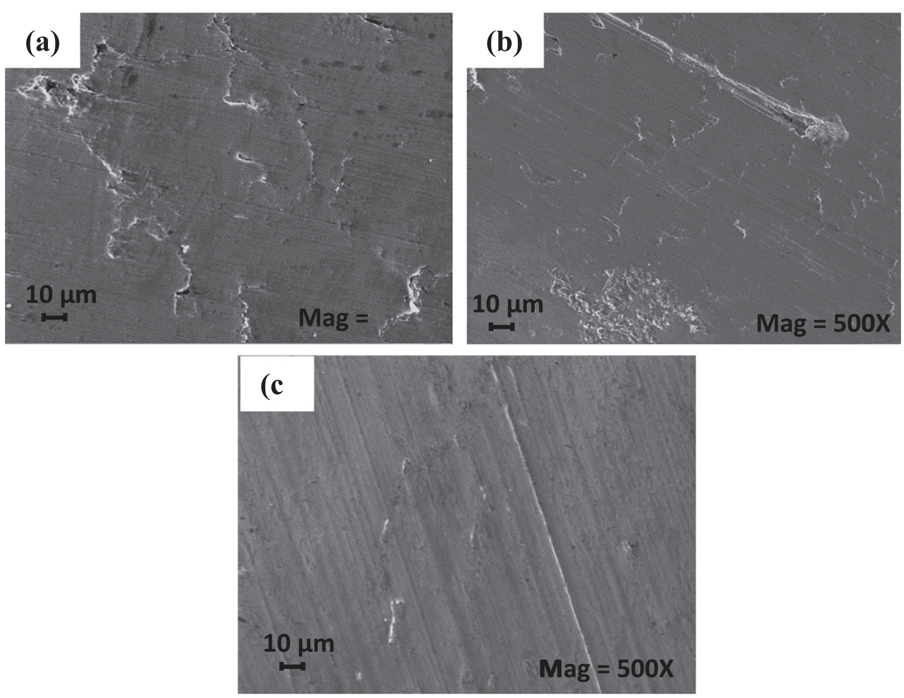

Fig. 15. SEM micrographs of disc surfaces of composites (a) K_0 (b) K_M (c) K_N.

- Almost all tribological properties improved with the replacement of 3 wt% barite by 3 wt% KT. Nano-KT proved significantly better performer than its counterpart (mico-KT), which in turn proved better than the composite without KT.
- Though it is always agreed in the literature that the reduced scale prototypes (RSPs) do not reflect the trends in performance of FMs when tested on realistic conditions on Dynamometer, in this work, the trends matched and performance ranking was identical.

Final conclusion of the work was based on performance ranking as per Table 9, which shows total count, lower the total count better is the composite. The numbering has been given as per performance order for selected property. The best one would get 1 and poorest would get 3. It seems that in both the cases, K_N got ranking of 10, proving it best followed by K_M (counts 17 and 16- almost identical). The K_0 which proved to be poorest in performance got the counts 21, 22.

Table 9
Ranking of composites on the basis of selected parameters

Parameters	RSP			Dynamometer		
	K_0	K_M	K_N	K_0	K_M	K_N
μ_{avg}/μ (higher the better)	2	3	1	2	3	1
$\delta\mu/\Delta\mu$ (lower the better)	3	2	1	3	2	1
Fade μ (higher the better)	3	2	1	3	2	1
% Fade/% Fade ratio*	3	2	1	3	2	1
Recovery μ (higher the better)	1	3	2	3	2	1
% Recovery/% Recovery ratio*	3	1	2	3	2	1
Max. disc temp. rise (lower the better)	3	2	1	2	1	3
Wear (lower the better)	3	2	1	3	2	1
Total	21	17	10	22	16	10

* % Fade & % Recovery are lower the better while % fade ratio & % recovery ratio are higher the better.

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Appendix A

For RSP:

- μ_{avg}: average coefficient of friction of last 15 brake applications during one test of 25 braking cycle. (In effectiveness test of RSP)
- Friction behaviour: variation in μ_{avg} with an increase in pressure at each speed.
- Sensitivity of μ to pressure: change in μ_{avg} with change in pressure. When pressure changes from 2 to 3 MPa it is referred as mild conditions while severe condition corresponds to pressure change from 2 to 4 MPa.
- Sensitivity of μ to speed: change in μ_{avg} with change in speed. When speed changes from 36 to 45 kmph it is referred as mild conditions while severe condition corresponds to speed change from 36 to 54 kmph.
- μ_{min}: This is the lowest coefficient of friction in all three modes (cold, fade and recovery).
- μ_{max}: This is the highest coefficient of friction in all the three modes (cold, fade and recovery).
- Performance μ: This is an average coefficient of friction considered between 21 and 600 brake applications.
- Fade μ: Minimum coefficient of friction for fade mode (between 21 and 570 braking) for RSP and lowest μ in fade cycle for dynamometer.
- % Fade: $\frac{\text{Performance } \mu - \text{fade } \mu}{\text{Performance } \mu} \times 100$ (lower the better)
- Recovery μ: Average coefficient of friction in the recovery mode.
- % Recovery: $\frac{\text{Recovery } \mu}{\text{Performance } \mu} \times 100$ (lower the better)
- Friction variation ($\delta\mu$): $\mu_{max} - \mu_{min}$. (lower the better)

For Dynamometer:

- Speed spread: stability in μ when speed changes from first to second level.
- % speed spread (% SS): $\frac{\mu \text{at higher speed}}{\mu \text{at lower speed}} \times 100$ (higher the better)
- % Fade ratio: $\frac{\mu \text{ min}}{\mu \text{ max}} \times 100$ (higher the better)
- % recovery ratio: $\frac{\mu \text{ min}}{\mu \text{ max}} \times 100$ (higher the better)
- $\Delta\mu$: $\mu_{max} - \mu_{min.}$ (lower the better)

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