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Effect of Nanofillers on Abrasion Resistance of Carbon Fiber Reinforced Phenolic Friction Composites

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Abstract

The present study focuses on the development of polymeric friction composites with short carbon fiber, micron and nano-sized fillers, additives with varying weight% in phenol formaldehyde (PF) matrix using hot compression moulding process. The composites prepared with fillers viz. Molybdenum disulfide or Molykote (MK) and multi walled carbon nanotubes (MWCNTs) in carbon fiber reinforced PF matrix is designated as Set-I composites. Inclusion of graphite and nano-clay in carbon fiber reinforced PF matrix is designated as Set-II composites. The prepared composites are tested in Dry sand rubber wheel abrasion wear test rig, following ASTM standards for evaluating the abrasive wear behaviour. From the routine experiments, it was observed that the presence of combined micro and nanofillers *i.e.* 11.5 wt% MK + 0.5 wt% MWCNTs of Set-I, has shown superior abrasion resistance among the study group. The test results of the Set-I and Set-II composites are analyzed using Taguchi experimental design followed by analysis of variance (ANOVA) to understand the contributions of wear control factors affecting the abrasive wear characteristics. Further, worn surface of selected samples is analyzed using scanning electron micrographs.

Keywords

Friction Composites, Nanofillers, Abrasion, Design of Experiments, Scanning Electron Microscope

1. Introduction

Frictional materials used in automobile brake linings are multifarious composite materials, and they are well-known as: i) High-steel (semi-metallic) brake pads containing 30% - 65% metal, ii) Low-steel (low-metallic) brake pads containing 10% - 30% metal, iii) Organic brake pads (also known as "non-asbestos organic" (NAO)) and iv) Hybrid brake pads, being a compromise between materials from group's ii and iii. High-steel and low-steel friction materials possess several disadvantages such as tendency to corrosion, low thermal stability, uneven wear of brake disk, etc., have restricted their braking applications. Modern friction materials familiarly known as non-asbestos organic (NAO) made of thermoset composites have been utilized extensively, starting from bicycles, light commercial vehicles, heavy vehicles, airplanes, etc. [1] [2] [3]. These friction materials are a mixture of several ingredients, which includes many fillers, lubricants, friction modifiers and reinforcing fibers bonded together by a thermosetting resin [4] [5] [6] [7]. Of several kinds of reinforcing fibers as support in polymer matrix composites, fibers made of glass, carbon, aramid and so on are broadly used. They are categorized by their aspect ratio. Polymers are further can be strengthened with different fillers that are accessible normally or synthesized in many forms such as, flakes, platelets, particles and so on to enhance their processability, mechanical, tribological and other performance, and in addition to reduce material cost. Filler particles of nano size with optimum loading percent have yielded the outstanding and synergistic performance in several characterization process. Many researchers have carried out the research on friction materials and the details of their research works has been discussed in **Table 1**.

Also, many investigations are made on tribological characterization aiming to evaluate fade and recovery behaviour of friction materials. However, abrasive wear from loose debris which is formed due to high pressure application need to be studied considering influential parameters like load, abrading distance and abrasive particle size. Hence, in view of cited literature above, the objective of the present work is to study the abrasive wear behaviour of phenolic friction materials. In particular, the three-body abrasive wear (3-BAW) behaviour of short carbon fiber reinforced phenolic friction composites with varying wt% of micro and nano-fillers, using Taguchi design of experiments and ANOVA to understand the control factors and their contributions affecting the wear characteristics.

2. Experimental Details

2.1. Materials

The source of the materials used to prepare the micro and nano fillers filled phenolic polymer composites in the present investigation are presented in **Table 2**.

2.2. Fabrication Method

The details of the fabrication method followed in the present work are discussed elsewhere [19]. The details of the hybrid composites prepared are listed in **Table 3**.

2.3. Three-Body Abrasive Wear Test

The phenolic friction composite in automotive application as brake pads encounters the metallic surface of the drum during braking. This results in the generation of debris leading to the peel out of the fillers from the composite brake pads. These fillers act as the third body at the interface of the brake pad and the metallic drum surface. Hence the study of three-body abrasive (3-BAW) behaviour of phenolic friction composites is worth to discover. These tests were carried out using Magnum make Rubber wheel abrasion tester (RWAT) in accordance with ASTM G-65-16 [20]. **Figure 1** shows the photograph of RWAT

Table 1. Literature review on polymeric friction materials.

Reference	Research carried	Research outcome
Blau [8]	Reported on classification, typical properties and functions of various binders, fillers additives along with reinforcing fibers used in commercial brake materials formulation.	Role of each constituent in friction and wear control, and effect of their composition, form, distribution, and particle size was briefly discussed.
Bijwe ***et al.*** [9]	Reviewed on friction materials for automotive braking application, emphasizing development of semi metallic or resin bonded metallic and various fibers reinforced non-asbestos organics (NAO) braking materials.	New classes of non-asbestos fiber reinforced organic polymeric friction materials have completely replaced asbestos based brake materials because of their superior performance and their environmentally friendly nature.
Gurunath ***et al.*** [10]	Discussed about drawbacks of phenolic resin. In order to overcome this, an alternative resin was synthesized and tribo-tested to explore the possibility of replacing the currently used phenolic.	Composites prepared with new resin (N) proved better than the composite with traditional phenolic in all the tribo-performance properties.
Chan ***et al.*** [11]	Reviewed the various materials and constituents used in automotive brake friction material after the phasing-out of asbestos.	Mineral fillers (ceramic fillers) such as barite and clay are added to increase the volume as well as to reduce the overall cost of a composite on a volume basis.
Kim ***et al.*** [12]	The effects of reinforcing fibers on friction and wear characteristics were investigated with an emphasis on the friction film formation at the friction interface.	The friction film with both aramid pulp and potassium titanate maintained smooth friction surface and durable transfer film resulted in improved wear resistance and steady friction force.
Kato ***et al.*** [13]	The importance of friction modifiers in friction materials, such as abrasives and solid lubricants in achieving desired range of friction was discussed.	Functional fillers and inert fillers or space fillers can be used to reduce the cost without affecting functionality of the friction materials.
Tanaka ***et al.*** [14] **Moraw** ***et al.*** [15]	Proposed weight percentage of matrix, fiber and friction modifiers for polymeric friction composites.	Material comprising 5 wt% - 20 wt% binder resin, 10 wt% - 50 wt% carbon or aromatic polyamide fiber, 5–30 wt% solid lubricant and 5 wt% - 20 wt% ceramic powder exhibits stable friction force and excellent wear resistance
Ho ***et al.*** [16].	Investigated on the effect of different short fiber reinforcement in friction materials.	Short fibers reinforcement, is most often used to obtain synergistic effect in mechanical and tribological performance of friction materials.
Friedrich ***et al.*** [17]	Investigated the influence of particle size and filler contents on the wear performance of nanoparticles reinforced thermoplastics and thermosets.	Presence of traditional fibers and/fillers with inorganic nanoparticles yields an optimal effect and showed a clear improvement in wear resistance of both thermosetting and thermoplastic composites.
Gopal ***et al.*** [18]	Analyzed the synergistic effect of multi-ingredients on friction and wear characteristics of friction materials.	Suggests that, the combination of several ingredients (fibers, micro/nano fillers and modifiers) and their synergism in a commercial friction material makes it rather difficult to analyze the friction and wear characteristics completely.

Table 2. Materials used in present investigation

Sl. No.	Materials system	Designation	Density (g cm^{-3})	Particle size	Suppliers
	Binders				
01	Phenol formaldehyde	PF	1.05	35 µm	Claro India Pvt. Ltd, Chennai, India
02	Cashew nut shell oil	CNSL	0.95	--------	Sathya Cashew Chemical Ltd, Chennai, India
03	Carbon powder	CP	1.80	75 µm	Mysore Pure Chemicals, Mysuru, India.
04	Plaster of Paris	POP	2.07	30 µm	Murugan Hardware, Erode, India
	Fiber				
05	Short carbon fiber	SCF	1.60	Φ 10 µm length 6 mm	Murugan Hardware, Erode, India
	Fillers				
06	Cashew dust	CD	0.65	15 µm	Sathya Cashew Chemical Ltd, Chennai, India
07	Copper powder	Cu	8.92	25 µm	Metal Powder Company India PVT Ltd, Sivakasi, India
08	Silicon carbide	SiC	3.20	25 µm	Carborundum Universal Ltd (CUMI), Cochin, India
09	Iron powder	FS	7.20	20 µm	Kumar Hardware, Coimbatore, India
10	Alumina	Al2O3	3.95	20 µm	Triveni Chemicals, Gujrat, India
	Functional fillers				
11	Molybdenum disulfide or Molykote.	MK	4.80	50 µm	Mysore Pure Chemicals, Mysuru, India.
12	Graphite	Gr	2.26	50 µm	Gowtham Chemicals, Chennai, India
13	Multi walled carbon nano tube	MWCNT	2.60	35 - 100 nm	Nanopar Tech, Chandigarh, India
14	Nano clay	NC	2.25	30 - 180 nm	

Table 3. Designations and constituents of thecomposites for Set-I and Set-II

Designation	Composition				
	Fibers (wt%)	Binders (wt%)	Fillers (wt%)	Functional fillers (wt%)	
Sample Set-I	SCF	PF-17, CNSL-9.5, POP-1.5 and CP-2	Cu-8, FS-8, SiC-5, CD-7 and Al_2O_3-5	MK	MWCNT
S1C1CF	25	30	33	12	0
S1C2CF	25	30	33	11.75	0.25
S1C3CF	25	30	33	11.5	0.5
Sample Set-II	SCF	PF-17, CNSL-9.5, POP-1.5 and CP-2	Cu-8, FS-8, SiC-5, CD-7 and Al_2O_3-5	Gr	NC
S2C1CF	25	30	33	12	0
S2C2CF	25	30	33	11.75	0.25
S2C3CF	25	30	33	11.50	0.5

apparatus used for the 3-BAW tests. The size of the test samples are maintained to 75 mm × 25 mm. The test procedure followed in the present work is as discussed elsewhere [21] [22], further the specific wear rate (K_s) are determined as mentioned in the reference [23]. The parameters for conducting the 3-BAW tests are listed in the **Table 4**.

2.4. Statistical Tool for Wear Characterization

The 3-BAW routine experiments were conducted for the test parameters listed in the Table 4. However, the K_s were found to be significant with applied normal load of 15 N and abrasive particle size of 300 µm. Hence they are considered as the constant factors in the present study. Further to understand the effect and contribution of abrading distance and filler content on K_s, they were considered for statistical analysis. Significant K_s was found at the abrading distances of 280, 570 and 1140 m, hence abrading distance at these levels were considered for statistical analysis. The control factors and levels listed in Table 5, are used for statistical analysis of wear. An orthogonal array (OA) L_9 was chosen and the factors considered affecting the wear process are abrading distance (A) and filler

Figure 1. Dry sand rubber wheel abrasion test rig (RWAT).

Table 4. Test parameters considered for routine 3-BAW.

Test Parameter	
Abrasive feed rate (g/min)	255 ± 5
Material of the wheel	Chlorobutyl rubber
Rubber wheel diameter (mm)	228
Speed of rubber wheel (rpm)	200
Abrading distance (m)	280, 410, 570, 840 and 1140
Applied normal load (N)	5, 7.5, 10, 12.5 and 15
Quartz abrasive particle size (µm)	150, 210, 300, 354 and 400

Table 5. Abrasive wear control factors and levels.

Control factors	Levels		
	1	2	3
Abrading Distance (A), (m)	280	570	1140
Nano Filler content (B), (wt%)	0.00	0.25	0.50

content (B). The experimental data obtained are transformed into signal-to-noise (SN) ratio by considering the minimization of K_s. Analysis of variance (ANOVA) is used to reveal the level of significance of factors influencing wear. The percentage contribution by each of the process parameter in the total sum of the squared deviations can be used to evaluate the importance of the process parameter change on the performance characteristic. If the P-value (probability of significance) for a factor in the table is less than 0.05 (95% confidence level) then it can be considered that, the effect of the factors is significant on the response.

3. Results and Discussion

3.1. Three Body Abrasion Wear Study

The abrasive wear behaviour of phenolic friction composites are determined on Rubber wheel abrasion tester (RWAT). Figure 2 demonstrates the 3D surface plot with filler content in X-axis, abrading distance in Y-axis and K_s in Z-axis, revealing the combined effect of filler content and abrading distance on wear behaviour of the phenolic friction composites.

The K_s decreases with the increase in abrading distance and with the inclusion of MWCNT along with molykote (Set-I series) and nano clay along with graphite (Set-II series), maintaining almost the same trend with marginal difference in K_s. However, the K_s of Set-I series is comparatively lesser than that of the Set-II series. The role of filler content can be observed in the plot, which results in the significant reduction of K_s. The K_s was found to be high without the nano fillers and inclusion of the same upto 0.5 wt%, has resulted in the decrease of K_s. However, routine experiments were conducted with the phenolic composites loaded with higher concentration of (*i.e.* 0.5 wt% to 1.0 wt%) nano fillers. It was

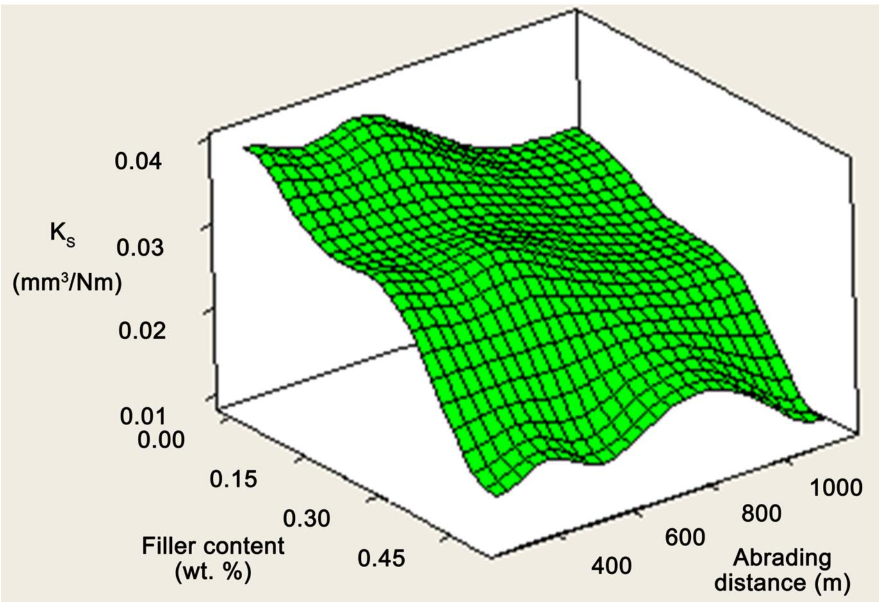

Figure 2. Surface plot revealing the combined effect of filler content and abrading distance on K_s of Set-I composites.

observed that the K_s increased beyond 0.5 wt% loading of nano fillers. The similar findings were observed by other researchers [24] [25]. Hence in the present analysis, the composite with 0.5 wt% nano filler loading was considered.

3.2. Worn Surface Morphology

Figure 3 presents the SEM image of worn surface of polymeric friction composites (PFC) filled with 11.5 wt% of molykote + 0.5 wt% of MWCNT subjected to 15 N applied normal load, abraded to a distance 1140 m with abrasive particle size of 300 μm. This composite has demonstrated high resistance to abrasion wear in the study group. Very few filler pull-out (indicated as FP), fiber pull-out (indicated as SP) and fiber rupture (indicated as SR) can be seen in the Figure 3. This has resulted in low wear volume of the composite in the study group. Good bonding of matrix with the fiber is evident from the image resulting in improved abrasive wear resistance.

Figure 4 presents the SEM image of worn surface of PFC filled with 12 wt% graphite subjected to 15 N applied normal load, abraded to a distance 1140 m

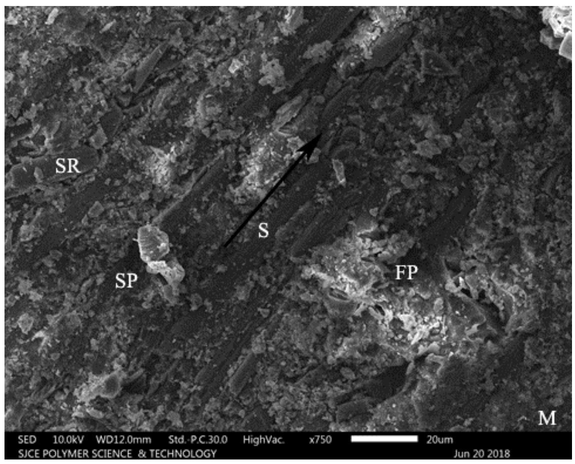

Figure 3. SEM image of worn surface of PFC filled with 11.5 wt% molykote + 0.5 wt% MWCNT.

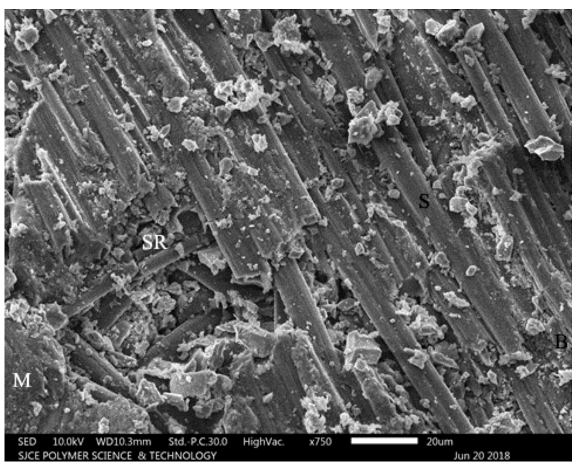

Figure 4. SEM image of worn surface of PFC filled with 12 wt% graphite.

with abrasive particle size of 300 μm. Inclusion of graphite has deteriorated the hardness of the composite resulting in low resistance to the abrasion wear. The matrix material (indicated as M) has been abraded and the short carbon fiber have been exposed to abrasive particles resulting in fiber rupture (indicated as SR), fiber pull-out and filler pull-out. This has resulted intense abrasion wear loss.

3.3. Statistical Analysis of Three-Body Abrasive Wear Data

Wear parameters that is abrading distance and filler content are considered as controlling factors at three different levels. Table 5 shows the control factors and their levels considered for the experimentation. The L_9 orthogonal array (OA) of experiments along with the experimental wear responses are shown in Table 6. The responses was analyzed to obtain signal to noise ratio, using the MINITAB 17 software, specifically used for the design of experiments (DOE) applications. The mean of signal to noise ratio was found to be 33.8783 dB for Set-I and 32.766 dB for Set-II composites respectively.

Figure 5 shows the graphs denoting the effect of the control factors on the K_s of Set-I and Set-II composites respectively. Process parameter settings with the highest SN ratio always gave the optimum quality with minimum variance. The graphs show the change of the SN ratio when the setting of the control factor was changed from one level to the other. The best K_s were at the higher SN ratio values in the response graphs. From the graph, it is clear that control factor combination of A3 and B3 gives minimum K_s. Thus, minimum K_s for the developed composite materials are obtained when the abrading distance (A) and filler content (B) are at the highest level. The SN ratio response of Set-I and Set-II composites is presented in Table 7. The SN ratio delta values of A and B for Set-I composites are 2.65 and 8.35: and for Set-II composites are 2.65 and 9.21 respectively. The strongest influence on the K_s was shown by factor B, followed by factor A, in both material groups under study.

Table 6. OA of experiments, responses and corresponding SN ratios.

Exp. No.	Abrading Distance (m)	Filler content (wt%)	K_s (mm^3/Nm)	SN Ratio (dB)	K_s (mm^3/Nm)	SN Ratio (dB)
			Set-I		Set-II	
1	280	0.00	0.0347763	29.1743	0.039041	28.1696
2	280	0.25	0.0247382	32.1326	0.031838	29.9411
3	280	0.50	0.0133018	37.5218	0.013519	37.3810
4	570	0.00	0.0341662	29.3281	0.038356	28.3234
5	570	0.25	0.0243042	32.2864	0.031279	30.0948
6	570	0.50	0.0130685	37.6755	0.013282	37.5347
7	1140	0.00	0.0256247	31.8268	0.028767	30.8221
8	1140	0.25	0.0182281	34.7852	0.023459	32.5936
9	1140	0.50	0.0098014	40.1743	0.009962	40.0335

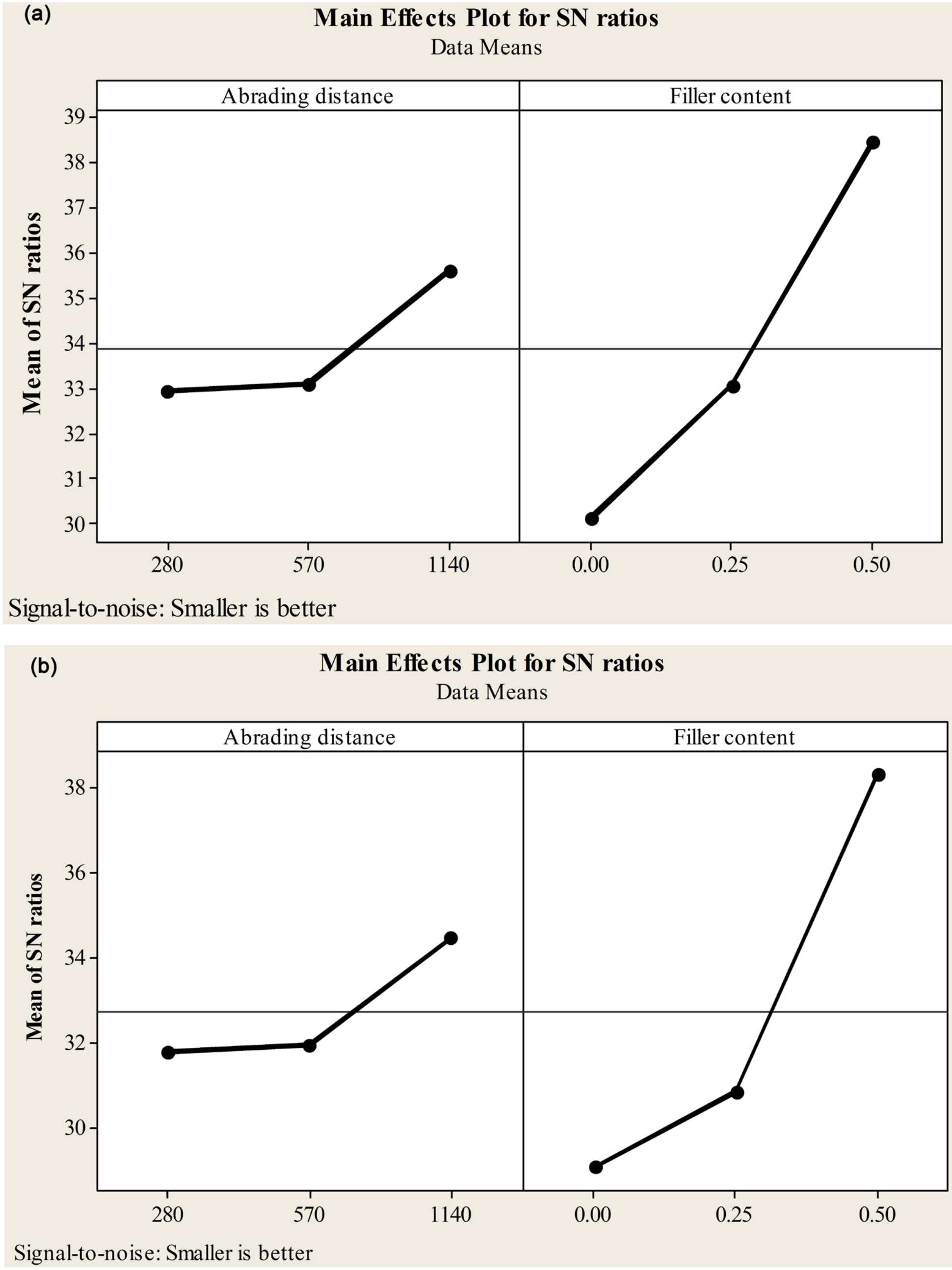

Figure 5. Plot demonstrating the main effects for SN ratio, (a) Set-I and (b) Set-II.

Table 7. Response table for SN ratio of Set-I and Set-II composites.

Level	Abrading Distance	Filler content	Abrading Distance	Filler content
	Set-I		Set-II	
1	32.94	30.11	31.83	29.11
2	33.10	33.07	31.98	30.88
3	35.60	38.46	34.48	38.32
Delta	02.65	08.35	02.65	09.21
Rank	02	01	02	01

3.4. Analysis of Variance and the Effects of Control Factors

The ANOVA with K_s results are listed in Table 8 for Set-I and Set-II composites respectively. This analysis was undertaken for a level of significance of 5%, that is, for level of confidence 95%. The last column of the table indicates the order of significance among control factors. It could be observed from Table 8 that the control factor B (P value = 0.000) has greater static influence of 86.806% and A (P value = 0.013) has an influence of 11.6687% on K_s of the material system under study. Also for Set-II from Table 8 the control factor B (P value = 0.000) has greater static influence of 87.8719% and A (P value = 0.016) has an influence of 10.5838% on K_s of the material system under study. The present analysis shows that 3-BAW test parameters that are, abrading distance and filler content have both statistical and physical significance.

3.5. Confirmation Test

The confirmation test is the final step in the DOE process. The purpose of the confirmation test is to validate the conclusions drawn during the analysis phase. The estimated SN ratio for K_s using the optimum level of parameters are calculated as discussed elsewhere [26] [27].

The results of experimental confirmation were carried out by comparing the predicted K_s with the actual K_s using the optimal wear parameters are shown in Table 9. The change in the predicted to experimental results is about 1.41% (Set-I) and 1.19% (Set-II), which is well within the statistical confidence level. Therefore the K_s of the friction composite material under study can be predicted with an allowable limit of 1.41% and 1.19% respectively.

Table 8. Analysis of Variance for K_s.

Set-I Composites							
Source	DF	Seq SS	Adj SS	Adj MS	F	Pvalue	PC (%)
Abrading distance (A)	2	0.0000765	0.0000765	0.0000383	15.31	0.013	11.6687
Filler content (B)	2	0.0005691	0.0005691	0.0002846	113.86	0.000	86.8060
Error	4	0.0000100	0.0000100	0.0000025			01.5253
Total	8	0.0006556					100.000
S = 0.00158090, 0R-Sq = 98.48%, R-Sq (adj) = 96.95%							
Set-II Composites							
Source	DF	Seq SS	Adj SS	Adj MS	F	Pvalue	PC (%)
Abrading distance (A)	2	0.0001028	0.0001028	0.0000514	13.71	0.016	10.5838
Filler content (B)	2	0.0008535	0.0008535	0.0004268	113.86	0.000	87.8719
Error	4	0.0000150	0.0000150	0.0000037			01.5443
Total	8	0.0009713					100.000
S = 0.00193597 R-Sq = 98.46% R-Sq (adj) = 96.91%							

DF: Degree of Freedom, Seq SS: sequential sum of squares, Adj MS: adjusted mean squares, F: variance P value: test statistics, PC (%): percentage of contribution.

Table 9. Confirmation test for the tested friction composite.

Set	Abrading distance	Filler content	Predicted K_s (mm^3/Nm)	Predicted SN ratio	Experimental K_s (mm^3/Nm)	Experimental SN ratio	Percent change
I	570	0.5	0.0141	37.0443	0.0139	37.6755	1.41
II	570	0.5	0.01346	37.9813	0.0133	37.5347	1.19

4. Conclusions

- Inclusion of 11.5 wt% of MK and 0.5 wt% of MWCNT in PF composites exhibited highest abrasion resistance among the composites under study.
- Nano fillers had beneficial effect on the abrasion behaviour of PF composites under study.
- Filler concentration played a vital role with a contribution of around 87% in 3-BAW behaviour of the PF composites.
- It is observed that minute agglomerates facilitated in forming a network which helped in improving the abrasion wear resistance.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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