Optimisation of testing parameters on two-body abrasive wear behaviour of nano-oMMT filled carbon-epoxy composites based on the Taguchi method

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Abstract: Two-body abrasive wear behaviour of carbon fabric reinforced epoxy (C-E) composites and nanosized organo modified montmorillonite (oMMT) particles dispersed C-E hybrid composites was investigated. Abrasive wear tests were carried out using a pin-on-disc machine under multi-pass condition. Silicon carbide (SiC) waterproof abrasive papers of different particle sizes were used under dry abrasion conditions. The influence of tribological parameters like applied normal load, abrading distance, filler loading and particle size of the SiC abrasives on the wear volume was investigated. Routine experimental results indicate that the wear volume depends on wt % of oMMT loading and the specific wear rate decreases with increase of abrading distance and increases with increasing load for all the composites. The scanning electron microscopy pictures indicate the reasons

for failure of composites and influencing parameters. The orthogonal array, signal-to-noise ratio and analysis of variance were employed to study the optimal testing parameters on oMMT filled carbon-epoxy composites with $20~\mu m$ and $40~\mu m$ SiC particle sizes. The experimental results demonstrate that the abrading distance was the major parameter on abrasive wear, followed by filler loading and applied normal load. The particle size of SiC, however, was found to have a neglecting effect. Moreover, the optimal combination of the testing parameters could be predicted. Regression analysis was used to arrive at a mathematical model to predict the wear volume in terms of tribological parameters. The predicted wear volume of the composites was found to lie close to the experimental values.

Keywords: oMMT filled C-E; two-body abrasion; Taguchi method; wear volume; regression analysis.

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1 Introduction

Fibre reinforced polymeric composites are the most rapidly growing class of materials, owing to their good combination of high specific strength and specific modulus. They are widely used for variety of engineering applications. The importance of tribological properties convinced many researchers to study the tribological behaviour and to improve the wear resistance of polymeric composites.

Wear is defined as damage to a solid surface, generally involving progressive loss of material, owing to relative motion between that surface and contacting substance or substances. Among different types of wear, abrasive wear is the most important among all the forms of wear because it contributes almost 63% of the total cost of wear [1,2]. Different types of polymer show different tribological behaviour. However, neat polymer is very rarely used as bearing materials and wear-resistant materials because unmodified polymer could not satisfy the demands arising from the situations wherein a combination of good mechanical and tribological properties is required [3]. Abrasive wear situation encountered in vanes and gears, in pumps handling industrial fluids, sewage and abrasive-contaminated water, roll neck bearings in steel mills subjected to heat, shock loading; chute liners abraded by coke, coal and mineral ores; bushes and seals in agricultural and mining equipment, have received increasing attention [4,5].

The modification of tribological behaviour of fibre-reinforced polymers by the addition of filler material has been reported [6–9] to be quite encouraging. Fillers perform more function than mere filling. They reduce the shrinkage, increase modulus and hardness and improve electrical properties. The use of fillers has grown dramatically in recent years encouraged by the growing demand for high performance plastics and increasing polymer prices.

A literature survey indicated that the short fibre reinforcement, in general, led to the deterioration in the abrasive wear resistance of the polymer matrix [2,10]. Fabric reinforcement, on the other hand, show improved abrasion resistance of the thermoset polymers [11]. Many researchers studied the two-body wear behaviour for polymers in general and polymer composites in particular [12-15]. Researchers have carried out investigations on tribological behaviour of different thermoplastics with fibre reinforcement. Many of them have reported deterioration in wear performance of the thermoplastic polymer composites [12-14]. Friedrich [15] investigated the abrasive wear behaviour of epoxy reinforced with carbon, glass and aramid fabrics and reported the wear performance of the fabrics in the order carbon > glass > aramid. The woven fabrics carry with them the advantages of balanced properties and the ease of handling during the fabrication. Wear resistance of neat polymers have shown to increase on being reinforced with fabrics [16,17]. Suresha and Kumar [17] reported that carbon fabric reinforced vinyl ester composites displayed enhanced tensile characteristics as well as abrasive wear resistance when compared to that of glass fibre reinforced vinyl ester composites. Carbon fabric reinforcement has shown improved wear resistance and thermal conductivity, as reported by Bijwe et al. [18]. Suresha et al. [19] carried out research work on three-body wear tests of woven glass fabric and carbon fabric reinforced epoxy composites. The results showed that carbon epoxy composites exhibited better wear resistance. Carbon fibre is graphitised carbon with the hexagonal planes of its crystals aligned perpendicular to the fibre axis. The lubricating function of the graphitised carbon is thought to be responsible for the reduction of friction coefficient and wear rate as the composites slide against the mating surface. Besides the lubricating function, carbon fibre also enhances the thermal conductivity and the mechanical properties of the polymer matrix, which is believed to be beneficial to the wear resistance as well.

Incorporation of fillers and fibres in most polymer composites is found to improve the abrasive wear performance significantly [20,21]. Amount of filler added, filler matrix interaction, and type of the matrix used affect the abrasive wear performance [22-27]. In view of benefits of nanoparticles, quite a few works have been carried out on the investigation of mechanical properties of nanoparticles filled PMCs [28-30]. oMMT particulates are being increasingly used in PMCs [31,32]. The ceramic nature of oMMT particulates as well as their high hardness helps in enhancing the hardness and consequently the wear resistance of the polymer composites. Hence, in the present work, bidirectional carbon fabric reinforced epoxy composites filled with nanosized clay particulates are taken up for characterisation of a few mechanical properties and their abrasive behaviour under multi-pass two-body abrasive mode. In addition, statistical techniques such as Taguchi method, analysis of variance (ANOVA) were employed to critically analyse the influence of each test parameters on abrasive wear of the composites in study. A mathematical model was developed using regression analysis to predict wear volume. Minitab R17 was used for the purpose of statistical analyses.

2 Experimental procedure

2.1 Materials and sample preparation

The composite materials considered in the present investigation utilised bidirectional carbon fabric (T300) of about 6–8 µm diameter as reinforcement. LY 556 epoxy resin with HY951 grade room temperature curing hardener (all supplied by Hindustan Ciba Geigy) mix was used for the matrix material. oMMT particulates of average size of about 20–25 nm were employed as filler material. The carbon–epoxy composite was prepared by hand lay-up procedure followed by autoclave curing. The panels have been fabricated by autoclave mould technique (pressure 0.735 MPa and temperature 90°C). Table 1 shows the details of the composites used in this investigation.

Table 1 Composites used in	the current investigation
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Composites	Designation	Carbon fabric (wt.%)	Matrix (wt.%)	Filler (wt.%)
Carbon-Epoxy	С-Е	60 ± 2	40	_
1.5 wt.% oMMT filled Carbon-Epoxy	1.5 oMMT C-E	60 ± 2	38.5	1.5
3 wt.% oMMT filled Carbon-Epoxy	3 oMMT C-E	60 ± 2	37	3
5 wt.% oMMT filled Carbon-Epoxy	5 oMMT C-E	60 ± 2	35	5

2.2 Two-body abrasive wear test

Two-body abrasive wear tests were performed on pin-on-disc wear testing machine according to ASTM G99 (Make: DUCOM, Bangalore). The detailed test procedure can be found in an earlier work [27]. The Tribo parameters used in the wear tests are tabulated in Table 2.

 Table 2
 Tribo parameters used in two-body wear tests

Tribo parameters	Values
oMMT (wt.%)	0, 1.5, 3 and 5
Abrading distance (m)	5, 100, 150 and 200
Load (N)	5 and 10
SiC abrasive particle size (μm)	35 and 52.5

The difference between the weights of the specimen before and after the test is the abrasive wear loss. The weight loss of the specimen (ΔW) owing to wear test was then used to find wear volume (ΔV) using the density of the composites (Density of the composites was measured as per ASTM D792 standards, using METTLER AE200 densometer). The specific wear rate (K_s) were calculated from the equation (1):

$$K_s = \frac{\Delta V}{L \times D},\tag{1}$$

where

 K_s : specific wear rate in mm³/Nm

 ΔV : wear volume in mm³ L: load in Newton

D: abrading distance in m.

3 Results and discussion

In this section, the results of two-body wear tests and subsequent worn surface morphology aspects are discussed.

3.1 Two-body abrasive wear test

3.1.1 Specific wear rate

The specific wear rate of unfilled and oMMT-filled C-E hybrid composites abraded against SiC abrasive papers of particle size 35 μ m and 52.5 μ m at 5 N and 10 N loads are shown in Figures 1 and 2 respectively.

It is evident that the specific wear rate of oMMT filled C-E composites was lower than that of the unfilled composites for different abrading distances and at both applied load values. As the oMMT loading increased, the specific wear rate was found to decrease. The specific wear rate was also found to decrease with increasing abrading and increasing the applied normal load. Further, it was observed that the specific wear rate

decreased with a decrease in the particle size of abrasive paper. A significant decrease in specific wear rate was witnessed as the abrading distance increased, for 280 grit SiC abrasive paper (particle size $52.5 \, \mu m$), as shown in Figure 2(b). This decreasing tendency gradually attained saturation level especially in the abrading distance range of 150 m to 200 m for all the specimens examined.

Figure 1 Specific wear rate of unfilled and oMMT-filled C-E hybrid composite against the abrading distance at 5 N using (a) 400 grit siC paper and (b) 280 grit siC paper (see online version for colours)

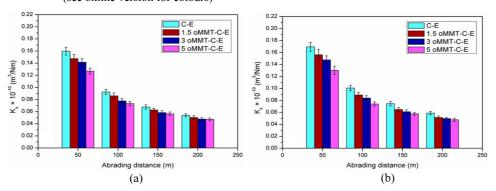
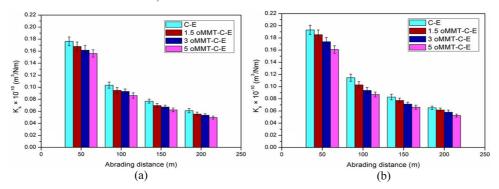


Figure 2 Specific wear rate of unfilled and oMMT-filled C-E hybrid composite against the abrading distance at 10 N using (a) 400 grit siC paper (b) 280 grit siC paper (see online version for colours)



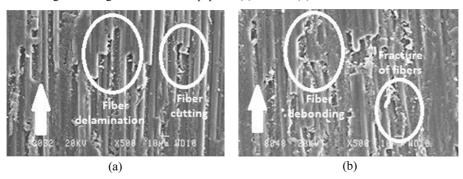
The incorporation of nanoparticles into polymer composites strongly influenced their abrasive wear behaviour [31,33]. Most thermoset polymers show an improvement in abrasive wear performance after the incorporation of filler/ fibre materials [34,35]. The observations made in the current study are in line with the cited works.

3.1.2 Worn surface morphology

To understand the involved two-body abrasive wear mechanisms, scanning electron photomicrographs of worn surfaces of wear test specimens were captured. Worn surface morphology of unfilled and 5 wt.% oMMT-filled C-E composites after multi-pass two-body abrasive wear tests at higher abrading distance of 200 m (for SiC abrasive paper grit size of 280) are shown in Figures 3 and 4. Arrow marks indicate the direction

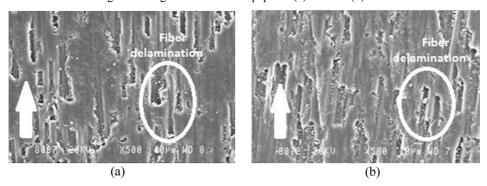
of abrasion. Abrasion led to the removal of material from the sample surfaces through several mechanisms. Photomicrographs enable the identification of prevailing wear mechanisms involved. The various wear mechanisms include micro-cracking in the matrix, micro-ploughing, micro-cutting, micro-fracture of fibre, and micro-fatigue.

Figure 3 Worn surface morphology of unfilled C-E composite for 200 m abrading distance against 280 grade siC abrasive paper at (a) 5 N and (b) 10 N



For unfilled composites at higher load (Figure 3(b)), wear scars and grooving in the abrading direction are visible. Wear debris from the epoxy matrix are also seen. The weakening of interface adhesion between the fibres and the matrix because of repeated application of load, followed by fibre debonding is also evident from the photomicrographs. This implies that at higher load, predominant wear mechanism involved was micro-cutting of fibres. Fewer fibres were cut and removed from the worn surface during abrasion at lower load can be seen from the photomicrograph 3a.

Figure 4 Worn surface morphology of 5 wt.% oMMT filled C-E composite for 200 m abrading distance against 280 grade siC abrasive paper at (a) 5 N and (b) 10 N



For 5 wt.% oMMT filled composites at 5 N load (Figure 4(a)), the damage to the matrix and fibre is less when compared to unfilled C-E worn surface shown in Figure 3(a). At lower load, the predominant wear mechanism is micro-cutting, whereas, at higher load (Figure 4(b)), by abrasion along the fibres length. Further, the worn surface indicated that the surface damage is caused by combination of matrix removal, fibre fracture and removal of few fibres from the sample surface.

4 Statistical methods

4.1 Taguchi method

The Taguchi method was employed to analyse the two-body abrasive wear behaviour of the composites under present study. The test factors and their levels used in the current investigation are shown in Table 2. Since, the levels used for different factors are not same, mixed-levels approach was employed and Taguchi orthogonal array L16 was created. The software Minitab R17 was used for the analysis of the wear test results.

Table 3 shows the values of different factors according to L16 array along with the response (wear volume, ΔV). After tabulating the values of the responses, the results were analysed using the software. The 'smaller is better' option was used as we intend to minimise the wear volume. The signal to noise ratio (S/N) was calculated using equation (2).

$$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum y^2\right). \tag{2}$$

The following observations were made by the analysis of the Taguchi array.

Table 4 is the response table for *S/N* ratios observed for different factors under different levels. The values tabulated in the table are the mean values of *S/N* ratios of the factor at the particular level obtained from Taguchi analysis. The control factors are ranked as shown in the last row of the Table 4 depending upon their contribution to wear volume loss.

Table 3	Result of two-body abrasive wear tests as per orthogonal array L_{16}
I able 5	Result of two-body abrasive wear tests as per orthogonal array L_{16}

L_{16}	Filler loading (% oMMT)	Abrading distance (m)	Load (N)	SiC grit (particle size)	Wear volume loss (mm³)	S/N ratio
1	0	50	5	35	7.986	-18.05
2	0	100	5	35	9.236	-19.31
3	0	150	10	52.5	12.431	-21.89
4	0	200	10	52.5	13.056	-22.31
5	1.5	50	5	52.5	7.810	-17.85
6	1.5	100	5	52.5	8.905	-18.99
7	1.5	150	10	35	10.438	-20.37
8	1.5	200	10	35	11.095	-20.9
9	3	50	10	35	8.077	-18.15
10	3	100	10	35	9.308	-19.38
11	3	150	5	52.5	9.154	-19.23
12	3	200	5	52.5	9.923	-19.93
13	5	50	10	52.5	8.049	-18.12
14	5	100	10	52.5	8.699	-18.79
15	5	150	5	35	8.455	-18.54
16	5	200	5	35	9.431	-19.49

The results are shown graphically in Figure 5 which shows the means of S/N ratios for different factors at different levels. The peaks of the three curves in Figure 5 represent the optimum parameter setting of the factors (A4-B1-C1-D1) and their levels as shown in Table 5. It was confirmed from the test results that the wear volume loss recorded for the values of test parameters indicated in Table 5 is 6.34 mm³, which is the lowest of all the readings from two-body abrasion tests. It also showed 20% decrease in wear volume as compared with first test case of L_{16} orthogonal array.

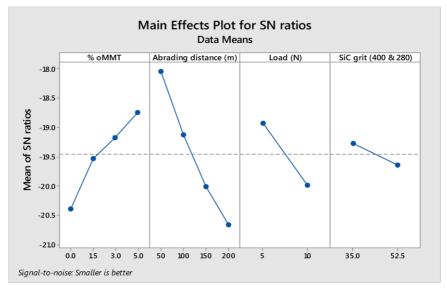
Table 4 Response table for *S/N* ratios for abrasive wear volume

Level	Filler loading (% oMMT)	Abrading distance (m)	Load (N)	SiC grit (particle size)
1	-20.39	-18.04	-18.93	-19.27
2	-19.53	-19.12	-19.99	-19.64
3	-19.17	-20.01		
4	-18.73	-20.66		
Delta (Max~Min)	1.66	2.62	1.06	0.37
Rank	2	1	3	4

 Table 5
 Optimum parameter setting for minimum wear loss

Optimum parameter setting for A4 B1 C1 D1				
Factor	Value			
Filler loading (% oMMT)	5			
Abrading distance (m)	50			
Load (N)	5			
SiC grit (particle size, μm)	35			

Figure 5 Effort of control factors on wear volume of composites (see online version for colours)



4.2 Analysis of variance (ANOVA)

Analysis of variance (ANOVA) was done at a confidence level of 95% (significance level of 5%). ANOVA helps in arriving at percentage contribution of all the factors on the variance of the response. Table 6 shows the results of the ANOVA. The sixth column of Table 6 indicates P value which provides the probability of significance of the control factors. The last column presents the % contribution of main control factors as well as their interactions. Since ANOVA was carried out for significance level of 5% (0.05), the factors for which the P-value is less than 0.05 (in other words, confidence level more than 0.95 or 95%) were considered to be significant ones. One can notice from Table 6 that for all the factors, P-values are less than 0.05 and hence, all of them are significant factors. It can also be observed that the value of P for the interaction of the factors is more than 0.05 and hence the interaction is insignificant in the analysis of wear volume. The factor with the highest % contribution to wear volume loss is the abrading distance (52.71 %), followed by filler loading (24.65%) and the applied load (18.02%) and SiC particle size (2.74%). This order of factors confirms with the ranking of factors obtained from Taguchi analysis (Table 5). Error is found to be very low (0.35%). A high percentage of R-sq (99.65%) indicates better correlation of wear volume in terms of the control factor.

Table 6 Analysis of variance for two-body wear volume

Source of variance	DoF	SS	MS	F	P	% Contribution
Filler loading (% oMMT)	3	8.9813	2.99376	93.28	0.000	24.65
Abrading distance (m)	3	19.2020	6.40065	199.43	0.000	52.71
Load (N)	1	6.5675	6.56754	204.63	0.000	18.02
SiC grit (particle size)	1	1.0002	1.00023	31.17	0.005	2.74
{(Abrading distance) \times Load (N) \times SiC grit (35 μ m and 52.5 μ m)}	3	0.5492	0.18308	5.70	0.063	1.50
Error	4	0.1284	0.03209			0.35
Total	15	36.4286				100.00

S = 0.179150; R-sq = 99.65%; R-sq(adj) = 98.68%.

4.3 Contour plots

Contour plot depicting wear volume with filler loading along x-axis and abrading distance along y-axis is shown in Figure 6. It can be clearly observed from the graph that the top-left corner is the vulnerable zone for wear volume. This part of the graph corresponds to high abrading distances and low amounts of filler loading. It is obvious from the figure that at lower abrading distances and as the filler content in the composites increases, the wear volume was found to decrease

Contour plot showing wear volume with filler loading along x-axis and applied load along y-axis is displayed in Figure 7. The plot clearly depicts that the wear volume loss is very high in the top-left corner of the graph pertaining to high value of the applied load and lower filler loading. At extreme right end of the graph pertaining to higher filler loading, it is seen that the wear volume is minimum for different values of applied loads.

Figure 6 Contour plot for wear volume against filler loading and abrading distance (see online version for colours)

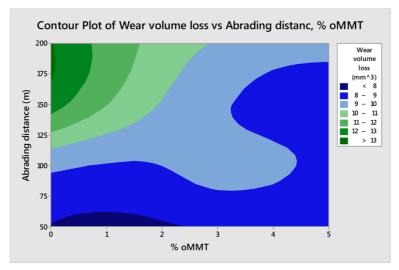


Figure 7 Contour plot for wear volume against filler loading and applied load (see online version for colours)

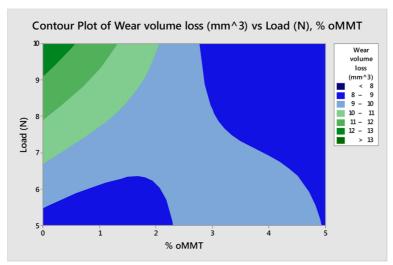


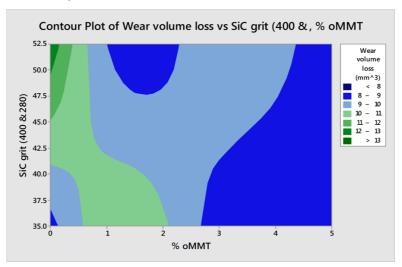
Figure 8 depicts contour plot showing wear volume with filler loading along x-axis and SiC abrasive particle size. When the abrasive particle size is high (52.5 μ m) and the filler loading is low, the wear volume is found to be high near the top-left corner. The right end of the graph with higher filler loading results in reduced wear volume loss for different particle sizes of the SiC abrasive paper.

4.4 Regression analysis

Regression models were developed for response (performance) parameter to predict the response (the wear volume), in terms of the test parameters, and the comparison of the

same with the experimental values were also performed. Minitab R17, statistical software is used for experimental design, data analysis and to develop regression models. Regression analysis has been carried out using the wear volume of the hybrid composites tabulated in Table 3 to develop the regression equations.

Figure 8 Contour plot for wear volume against filler loading and siC grit size (see online version for colours)



In the present study general multiple linear regression analysis was adopted to come out with a quantitative relationship between the response (wear volume loss) and the predictors (% filler loading, abrading distance, applied load and SiC abrasive particle size)

The general linear regression model obtained from the regression analysis of the hybrid composites is given by equation (2).

Wear volume =
$$4.925 + 1.811 \times 10^{-1} \times P$$
) + $(5.637 \times 10^{-2} \times D)$ + $(4.228 \times 0^{-1} \times L)$
- $(4.404 \times 10^{-2} \times G)$ - $(1.197 \times 10^{-2} \times P \times D)$
- $(2.160 \times 10^{-1} \times P \times L)$ + $(1.190 \times 10^{-2} \times P \times G)$
- $(7.638 \times 10^{-3} \times D \times L)$ - $(2.29 \times 10^{-4} \times D \times G)$
+ $(7.074 \times 10^{-3} \times L \times G)$ + $(3.472 \times 10^{-3} \times P \times D \times L)$
+ $(6.1 \times 10^{-5} \times P \times D \times G)$ + $(1.377 \times 10^{-3} \times P \times L \times G)$
+ $(8.5 \times 10^{-5} \times D \times L \times G)$ - $(4.9 \times 10^{-5} \times P \times D \times L \times G)$,

where

D: abrading distance

L: load

G: SiC grit size

P: wt.% of oMMT.

A very good correlation between wear volume predicted by regression analysis and that observed from experimental results (difference of less than 1% between the two) was observed.

5 Conclusions

The following are the conclusions drawn from the current study:

- 1 Addition of oMMT to C-E composites has resulted in enhancement of abrasion resistance. The minimum specific wear rate of 0.047 × 10⁻¹⁰ (mm³/Nm) and maximum specific wear rate of 0.193 × 10⁻¹⁰ (mm³/Nm) was observed in the case of 5 wt.% oMMT filled C-E composite and unfilled C-E respectively. The nanosized particulates have resulted in large interfacial contact area with the matrix leading to superior interface bonding. The surface treatment of reinforcements has supplemented the enhancement.
- 2 Higher oMMT loading (5 wt.%) has showed improved abrasion resistance compared to both unfilled as well as lower wt.% oMMT filled composites. Hence, C-E composites with 5 wt.% oMMT addition can be recommended for applications involving abrasion.
- 3 The abrading distance and oMMT filler loading, applied load and the abrasive size influenced the specific wear rate.
- 4 The optimum parameter set to minimise wear loss as obtained by Taguchi analysis is
 - a filler loading of 5 wt.% oMMT
 - b abrading distance of 50 m
 - c applied load of 5 N
 - d SiC abrasive size of 35 μm.

It was confirmed from the experimental results that, for the above values of the test parameters, lowest wear volume was recorded.

- From ANOVA, the influence of each parameter on wear loss was evaluated and is as follows: The factor with the highest% contribution to wear volume loss is the abrading distance (52.71%), followed by filler loading (24.65%) and the applied load (18.02%) and SiC particle size (2.74%).
- 6 The mathematical model developed using regression analysis ensured a very good correlation with the experimental results.

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