



Effect of Fillers on Erosive Wear behavior of Polyoxymethylene/ Polytetrafluoroethylene Blend and their Composites: A Statistical Approach

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ABSTRACT

In this research article, erosion wear behavior of polyoxymethylene (POM)/polytetrafluoroethylene (PTFE) blend and their composites reinforced with short glass fiber (SGF), filled with ceramic fillers such as silicon carbide and alumina (Al_2O_3) in micro/nano scale, along with molybdenum disulfide and perfluoropolyether in nanoscale as solid lubricants have been investigated. Experiments were carried out on solid particle erosion test rig. The tribological parameters considered in this study include impact velocity (30, 35, and 40 m/s), impingement angle (30°, 45°, and 60°) and particle size of the silica (212, 425, and 600 μm) used as erodent. The erosion test reveals that POM/PTFE blend demonstrated utmost resistance to erosion wear followed by POM/PTFE nano- and micro-hybrid composites, while POM/PTFE reinforced with SGF demonstrated the least resistance in the study group. The weight loss due to erosion was found to be increased with increase in impact velocity and decrease in erodent particle size. The prepared composites demonstrated maximum weight loss due to erosion at 30° impingement angle. Statistical analysis of erosion wear data was carried out by Taguchi design of experiment followed by analysis of variance to establish the inter-dependence of erosion wear operating parameters in the study.

Key words: Polyoxymethylene/polytetrafluoroethylene blend, Short glass fiber and ceramic fillers, Erosion, Taguchi design of experiments.

1. INTRODUCTION

Solid particle erosion is a dynamic process which causes material removal from a target surface due to the impingement of fast moving solid particles [1]. Solid particle erosion reveals negative results such as wear of components, surface roughing, macroscopic scooping appearance, surface degradation, and reduction in functional life of the structure. Hence, solid particle erosion has been considered as a serious problem as it is responsible for many failures in engineering applications [2]. Polymer matrix composites (PMCs) are finding increased application under conditions in which they may be subjected to solid particle erosion at applications such as pipe lines carrying sand slurries in petroleum refining, helicopter rotor blades, pump impeller blades, high-speed vehicles, and aircrafts operating in desert environments [3].

Erosion resistance of PMCs used in many applications has become an important material property, particularly in the selection of alternative materials [4]. Further, the

erosive wear behavior is different for thermoset and thermoplastic based composites. The failure mode in thermoset composites is a complex process involving matrix micro-cracking, fiber-matrix debonding, fiber breakage, and material removal [5-7]. However, Thermoplastic composites behave differently. The higher matrix toughness allows substantial plastic deformation which absorbs a great extent of the impact energy [5]. The matrix is uniformly grooved due to microcutting and microplothing, which results in maximum material removal at oblique impact, viz. 30°. Therefore, it becomes imperative to study solid particle erosive wear behavior of PMCs in various operating conditions [8].

Many researchers [9-18] have evaluated the wear resistance of PMCs in solid particle erosive wear. In common they revealed that the operating parameters such as fiber and filler type, concentration, length and orientation, impingement angle, impact velocity, erodent mass flow rate, erodent size and the interfacial

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bonding between fiber and matrix have influence on the erosion behavior. To achieve the desired material characteristics for a particular application, it is important to know how the PMCs wear performance changes with the parameters. In a study conducted by Suresh *et al.*, [9] solid particle erosion behavior of polyetherketone (PEK) reinforced by short glass fibers (SGFs) with varying fiber content (0-30 wt.%) was investigated. Here, the authors evaluated the erosion behavior by considering different impact angles (15-90°) and impact velocities (25-66 m/s) using silica sand particles as erodent. The results revealed that PEK polymer and their composites exhibited maximum erosion rate at 30° impact angle indicating ductile erosion behavior. The erosion rates of PEK composites increased with increase in the amount of glass fiber (GF). Rattan and Bijwe [10] reported the erosion wear behavior of polyetherimide (PEI) and their composites using silica particles as erodent striking at a constant impact velocity and at different angles of impingement. Even though the mechanical properties of PEI improved substantially by carbon fabric reinforcement, they found that the erosion resistance of the material deteriorates by a factor of about four to six times at all the angles of impingement. In spite of the fact that PEI is not a very ductile polymer (elongation to fracture, 60%), they found that it exhibits maximum wear at 15°, which is a characteristic of ductile and semi-ductile mode of failure. Sari *et al.* [11] reported the erosive wear performance of carbon fiber (CF) reinforced polyetheretherketone (PEEK) and GF reinforced PEEK composites. They revealed that both composites exhibited semi-ductile erosive wear behavior with a maximum wear at the contact angle of 45°. The weight loss of CF-reinforced composites is higher than that of GF-reinforced composites. Bagci *et al.* [12] studied a solid particle erosion behavior of glass fabric reinforced polyester matrix composite materials, in which a remarkable increase in the erosion rate and correlated with erodent sizes used in the tests. Moreover, changes in erodent particle size bring about more effects on erosive wear rate than changes in the impact velocities. Tewari *et al.* [13] studied the solid particle erosion behavior of unidirectional CF and GF reinforced epoxy composites and evaluated for both impingement angle and fiber orientations and found that these two had a significant influence on erosion. Tilly [14-16] has investigated the influence of velocity, impact angle, particle size, weight of abrasive quartz particles impacted, and type of reinforcement in plastic materials such as nylon, polypropylene, and epoxy resins.

The important factors affecting the erosion rates are influenced by matrix, hardness of the fibers, and bond strength of fibers and matrix [17]. Patnaik and Tejyan [18] reported the erosive wear performance of viscose fiber based needle-punched nonwoven fabric mats (designated as VS200, VS400, and VS600)

reinforced composites. They implemented Taguchi design of experiment (DOE) and analysis of variance (ANOVA) to demonstrate the effects of various factors on the erosion rate. The results demonstrated that impact velocity, fiber content, and impingement angle are the operative factors influencing the erosion rate of viscose fiber based needle-punched nonwoven reinforced composites.

Research studies have been made on the erosive wear of composites on fiber-reinforced polymer and filler-reinforced-systems. The effect of fillers is considered more as modification of the matrix and less as reinforcement, probably because of the low percentage of fillers. As a result, the effect of particulate fillers on erosion characteristics of hybrid composites has hardly received any research attention. There is no clear understanding of the mechanism of erosion and how the properties of the constituents and the interface affect the erosion behavior of these composites. A possibility that the incorporation of both particles and fibers in polymer could provide a synergism in terms of improved properties and tribological performance has not been adequately explored so far. However, incorporation of fibers/fillers also leads to changes in the nucleation and crystallization processes of the crystalline matrices [19], thus affecting the performances of blends and their composites. Hence, this study reports the erosive wear performance of polyoxymethylene (POM) matrix alloyed with polytetrafluoroethylene (PTFE) reinforced with SGF and filled with ceramic fillers such as silicon carbide (SiC), aluminum oxide (Al₂O₃), molybdenum disulfide (MoS₂), and perfluoropolyether (PFPE). Further, Taguchi DOE and ANOVA approach are implemented to establish the interdependence of operating parameters, namely fiber/filler content, impact velocity, impingement angle, and particle size of the erodent.

2. EXPERIMENTAL DETAILS

2.1. Materials and Methods

In the present research, POM, purchased from DuPont, is used as a matrix material and PTFE purchased from DuPont as an alloying filler selected to form a polymer blend. The average particle size of PTFE was about 5-15 μm. Silane-treated SGF, purchased from Fine Organics, Mumbai, were used as reinforcement. The average diameter of the SGFs was approximately 12 μm with an average fiber length of about 4 mm. SiC procured from Carborundum India Ltd, Chennai, and micro-aluminum oxide (m-Al₂O₃), purchased from Triveni Chemicals, Gujarat, are used as ceramic fillers and the average particle size was about 5-10 μm. Nano-aluminum oxide (n-Al₂O₃), procured from Triveni Chemicals, Gujarat, with average particle size of 70 nm was also used as ceramic filler. MoS₂, purchased from M/s Omkar specialty chemicals Ltd, Thane, with average particle size of about 5-10 μm

was used as solid lubricating filler. Further, PFPE, procured from Nye Lubricants, USA, with average particle size of about 200 nm was also used as solid lubricant.

Before compounding, the polymer granules and fillers were dried at 80°C for 10 h in an oven. Selected compositions were mixed and extruded in Barbender co-rotating twin-screw extruder. The granules of the extrudates were pre dried in an air circulated oven at 80°C for 10 h and injection molded in a microprocessor-based injection molding machine fitted with a master mold containing the cavity for the tests of erosive wear specimens. The temperatures maintained in three zones of the barrel were 200°C, 235°C, and 260°C, respectively. Polymer blends and their composites prepared for the present experimental work is as shown in Table 1.

2.2. Erosion Testing

Erosion testing is carried out as per the ASTM G-76 using air jet erosion test rig. This basically consists of a sand hopper, conveyer, nozzle, setting for angle of impact, and specimen control panel. The erodent is fed into the hopper which then flows through the nozzle where it attains high speed with the help of carrier gas at high pressure. Velocity of the erodent can be varied by adjusting the air pressure. The erodent used is of angular quartz particles of 212 µm size. The composite specimen of size, 40 mm × 40 mm × 2.5 mm is rigidly placed on specimen holder. The erodent with a feed rate of 9 g/min and a particle velocity of 30 m/s was calculated by using a double disc method. In the present study, dry silica sand (angular) of different particle sizes (212, 425, and 600 µm) is used as erodent. Each sample is cleaned in acetone, dried, and weighed to an accuracy of ±0.1 mg using a precision electronic balance. It is then eroded in the test rig for 3 min and weighed again to determine the weight loss. The process is repeated until the erosion rate attains a constant value called steady-state erosion rate. The ratio of this weight loss to the weight of the eroding particles causing the loss is then computed as a dimensionless incremental erosion rate. The conditions under which the erosion tests were carried out are listed in Table 2.

2.3. Statistical Analysis of Wear Data

Taguchi experimental design is a powerful analysis tool for modeling and analyzing the influence of control factors on performance output. The control factors under which erosion tests were carried out are given in Table 3. Four parameters namely, material, impact velocity, impingement angle, and erodent size, each at three levels are considered in this study in accordance with the L₂₇ orthogonal array design. In Table 4, each column provides a test parameter and each row gives a test condition which is a combination of the parameter levels. Four parameters each at three levels would require 3⁴=81 runs in a full factorial experiment. However, Taguchi's factorial experiment approach reduces it to 27 runs, which is a great advantage. The experimental observations are transformed into signal-to-noise (SN) ratios. The SN ratio for the minimum erosion rate (maximum erosion resistance) is indicated by a smaller value, which is a better characteristic [20]. This can be calculated as a logarithmic transformation of mass-loss due to erosion rate as shown in equation:

$$\frac{S}{N} = -10 \log \frac{1}{n} (\sum y^2) \quad (1)$$

Where "n" is the number of observations, and "y" is the observed data. The test conditions used for the experimentation are listed in Table 3.

ANOVA is performed to find out the significant process parameters in terms of percentage contribution. The technique does not directly analyze the data, but rather determines the variability (variance) of data and finds out the significant process parameters [21]. The confirmation experimental procedure and the corresponding mathematical equation to predict the parameters used in the present research study are discussed by Hemanth *et al.* [22] in our earlier research article.

Figure 1 gives the erosion loss of POM/PTFE blend and their composites as a function of the impingement angle. The erosion loss of micro and combined micro and nano-filler reinforced POM/PTFE hybrid composites considerably increases with increase in impingement angle from 30° to 45° and then drastic decrease in erosion loss with increase in impingement angle (>45°) results. On the other hand, in the range

Table 1: Constituents of the polymer material composite system for present study.

Composites	Designation	POM	PTFE	SGF	SiC	m/n-Al ₂ O ₃	MoS ₂	PFPE
POM+PTFE	1A	80	20	-	-	-	-	-
POM+PTFE+SGF	2A	68	12	20	-	-	-	-
POM+PTFE+SGF+SiC+m-Al ₂ O ₃ +MoS ₂	3A	60	10	17.5	5	5/0	2.5	-
POM+PTFE+SGF+SiC+n-Al ₂ O ₃ +PFPE	4A	60	10	17.5	5	0/2.5	-	5

PTFE=Polytetrafluoroethylene, SGF=Short glass fibers, POM=Polyoxymethylene, SiC=Silicon carbide, MoS₂=Molybdenum disulfide, PFPE=Perfluoropolyether

Table 2: Test parameters considered for routine experiments.

Erodent	Silica sand
Erodent size (µm)	212, 425, 600
Impingement angle (°)	15, 30, 45, 60, 75 and 90
Impact velocity (m/s)	30, 35, 40 and 45

Table 3: Control factors and levels used in the experiment.

Control factors	Levels		
	1	2	3
Material	1A	3A	4A
Velocity (m/s)	30	35	40
Erodent size (µm)	212	425	600
Impingement angle (°)	30	45	60

of impingement angles selected in this study, for POM/PTFE blend with SGFs (2A), the erosion loss was highest. Edit as in general, the wear properties do always describe the whole tribological system rather than a material property alone. Such systems always consist of a counterpart, the specimen composition, a medium in between (e.g., lubricant), the environment and the stress conditions over a certain time range. Depending on the tribological conditions, the mechanisms involved in the erosion process may change significantly; especially the topography of the sample with different erosion parameters, Table 3 summarizes the test parameters selected for the further erosion test in the next stage of design of experiments.

3. RESULTS AND DISCUSSION

3.1. Erosive Wear Routine Experiments

Material property alone. Such systems always consist of a counterpart, the specimen composition, a

Table 4: Experimental plan using the L₂₇ orthogonal array and performance data.

Material	Velocity (m/s)	Erodent (µm)	Angle (°)	Wear loss (g)	SN ratio (dB)
1A	30	212	30	0.0206	33.72266
1A	30	425	45	0.0060	44.43697
1A	30	600	60	0.0012	58.41638
1A	35	212	45	0.0351	29.09386
1A	35	425	60	0.0058	44.73144
1A	35	600	30	0.0089	41.01220
1A	40	212	60	0.0330	29.62972
1A	40	425	30	0.0135	37.39332
1A	40	600	45	0.0204	33.80740
3A	30	212	30	0.0274	31.24499
3A	30	425	45	0.0050	46.02060
3A	30	600	60	0.0046	46.74484
3A	35	212	45	0.0341	29.34491
3A	35	425	60	0.0060	44.43697
3A	35	600	30	0.0163	35.75625
3A	40	212	60	0.0422	27.49375
3A	40	425	30	0.0132	37.58852
3A	40	600	45	0.0242	32.32369
4A	30	212	30	0.0236	32.54176
4A	30	425	45	0.0077	42.27019
4A	30	600	60	0.0033	49.62972
4A	35	212	45	0.0412	27.70206
4A	35	425	60	0.0080	41.93820
4A	35	600	30	0.0118	38.56236
4A	40	212	60	0.0549	25.20855
4A	40	425	30	0.0099	40.08730
4A	40	600	45	0.0281	31.02587

SN=Signal-to-noise

medium in between (e.g., lubricant), the environment and the stress conditions over a certain time range. Depending on the tribological conditions, the mechanisms involved in the erosion process may change significantly, especially the topography of the sample with different erosion parameters such as size of erodent, shape, impact velocity, and impingement angle. Hence, to account the said erosion parameters, Table 3 gives the test parameters selected for the further erosion tests in the next stage of DOE.

3.2. Statistical Analysis of Wear Data

The wear data were analyzed using MINITAB software, specifically used for the DOE applications. Table 4 presents the plan of experiments with wear losses using L_{27} orthogonal array. SN ratio has been calculated using equation (1), is presented in Table 5. The overall mean for the SN ratio of the wear losses was found to be 37.4876 dB. Figures 2 and 3 graphically denote the influence of the process parameters on the wear losses. The wear control parameter settings with maximum SN ratio value deliver the optimal quality with least variation. The graph shown in Figure 2 depicts the variation of the SN ratio when the control factor setting was varied from one level to the other.

The power wear loss was at the higher SN ratio values in the response graph (Figure 2). From the graph, it is clear that control factor combination of A1, B1, C2, and

D3 provides least wear loss. Thus minimum wear loss for the developed composite materials is attained when the filler content (A) and velocity (B) are at lowermost level, while erodent particle size (C) is at the moderate level and impingement angle (D) is at higher most level.

The SN ratio response is displayed in Table 6. The delta (maximum-minimum) values of A, B, C, and D are 2.59, 10.05, 12.55, and 5.80, respectively. The strongest influence on the wear loss was exerted by factor C, followed by factors B, D, and A, respectively.

The interaction effects of control parameters plot for SN ratio is portrayed in Figure 3. The wear control parameters do not interact when the lines are parallel and strong interactions arise when the lines cross in the interaction plots. Observation of Figure 3 reveals strong interactions between fiber/filler content versus velocity: Fiber/filler content versus erodent particle size and fiber/filler content versus impingement angle. In order to justify the interactions, statistical analysis (ANOVA) was carried out.

3.3. ANOVA and Control Parameters

Statistical design termed ANOVA is employed to cater individual percentage contribution of control factors influencing the wear behavior of PMCs. These measure the quality characteristics of control factors. The ANOVA for SN ratio results are listed in Table 7. In the present study, ANOVA analysis was carried out for a level of significance of 5% (for level of confidence 95 %). The last column of the Table 7 indicates the order of significance among control factors and interactions. It could be noted from the Table 7 that the control factors *material* ($p=0.2610$) has static influence of 2.224%, velocity ($p=0.0020$)

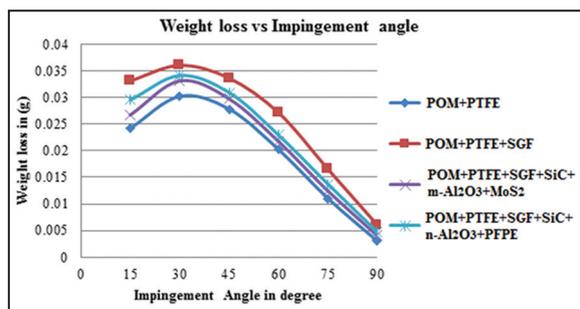


Figure 1: Depicts the weight loss of the polyoxymethylene/polytetrafluoroethylene composite versus impingement angle.

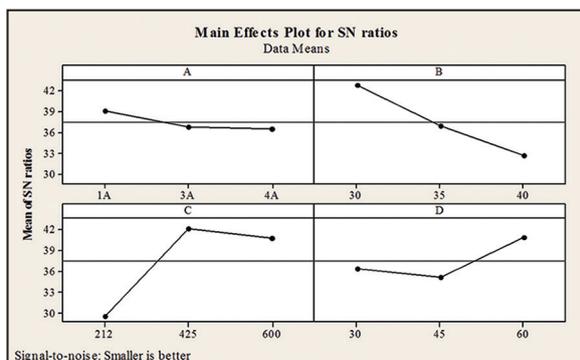


Figure 2: Main effects plot for signal-to-noise ratio of erosion test.

Table 5: Confirmation test for erosive wear loss.

Levels	Optimal process parameter		Improvement in the result (%)
	Prediction $A_1B_1C_2D_3$	Experimental $A_1B_1C_2D_3$	
SN ratio (dB)	59.3492	60.000	1.085
Wear loss (g)	0.00105	0.0010	4.762

SN=Signal-to-noise

Table 6: Response table for SN ratio.

Level	Material	Velocity	Erodent	Angle
1	39.14	42.78	29.55	36.43
2	36.77	36.95	42.10	35.11
3	36.55	32.73	40.81	40.91
Delta	2.59	10.05	12.55	5.80
Rank	4	2	1	3

SN=Signal-to-noise



Figure 3: Operating parameters interaction plot for signal-to-noise ratio.

Table 7: Analysis of variance for SN ratio.

Source	DF	Seq SS	Adj MS	P	P(%)
Material	2	37.00	18.50	0.261	02.224
Velocity	2	458.57	229.28	0.002	27.566
Erodent	2	857.31	428.65	0.000	51.536
Angle	2	166.38	83.19	0.023	10.002
Material*velocity	4	8.82	2.21	0.928	00.530
Material*erodent	4	35.34	8.83	0.562	02.124
Material*angle	4	34.65	8.66	0.570	02.083
Error	6	65.46	10.91		03.935
Total	26	1663.53			100.000

S=3.30308, R²=96.06%, R²(adj)=82.95%, DF=Degrees of freedom, Seq SS=Sequential sum of squares, Adj MS=Adjusted mean squares, P=Test statistics, P(%)=Percentage of contribution, SN=Signal-to-noise

has an influence of 27.566%, erodent (p=0.0000) has greater static influence of 51.536% and impingement angle (p=0.023) has an influence of 10.002 % on wear loss of the material system under study. However, the interaction between material versus velocity (p=0.928), material versus erodent (p=0.562) and material versus impingement angle (p=0.570) has an influence of 0.53%, 2.124%, and 2.083%, respectively, which show less importance to the contribution on wear loss.

The present analysis shows that erosive wear control parameters such as velocity, erodent and impingement angle have both statistical and physical importance (% contribution is greater than error) in the erosive wear behavior of POM/PTFE blend and their composites. However, interactions have statistical significance but do not have physical significance, since error percentage (3.935%) is more than percentage contribution of these

interactions, which is obvious from the ANOVA results.

3.4. Confirmation Experiment

The last step in the DOE, is the confirmation experiment. This is carried out to validate the inferences drawn during the analysis phase as stated by Roy [23] and Ross [24]. The estimated SN ratio for wear loss using the optimum level of parameters can be calculated with the help of the predictive equation as discussed in our earlier work [22].

The results of experimental confirmation using optimal wear parameters and comparison of the predicted wear loss with the actual wear loss using the optimal wear loss parameters are shown in Table 5. The enhancement in SN ratio from the preliminary level to optimal level is 1.085%. The wear loss is decreased by 4.762%. Hence, the wear loss are improved by using Taguchi method.

4. CONCLUSIONS

Based on the experimental work carried out, the following conclusion can be drawn.

POM + PTFE blend showed better resistance to erosion wear compared to other composites. The presence of particulate fillers in these composites did not improve their erosion wear resistance. From the statistical analysis, it can be concluded that among all the factors, erodent size is most significant control factor followed by velocity, impingement angle, and material. Material has the least effect on erosion rate of all the composites. Large abrasive particles lead to a decrease in wear. A marked decrease in erosion rate was observed as the erodent size increased from 212 to 425 μm. This study starts a new approach for utilization of fiber/filler reinforced thermoplastic blend based hybrid composites. These reinforced polymer

composites have sufficient potential for applications in highly erosive environments.

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