

Epoxy matrix composites filled with micro-sized LD sludge: wear characterization and analysis

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Abstract. Owing to the very high cost of conventional filler materials in polymer composites, exploring the possibility of using low cost minerals and industrial wastes for this purpose has become the need of the hour. In view of this, the present work includes the development and the wear performance evaluation of a new class of composites consisting of epoxy and micro-sized LD sludge. LD sludge or the Linz-Donawitz Sludge (LDS) are the fine solid particles recovered after wet cleaning of the gas emerging from LD convertors during steel making. Epoxy composites filled with different proportions (0, 5, 10, 15 and 20 wt %) of LDS are fabricated by conventional hand lay-up technique. Dry sliding wear trials are performed on the composite specimens under different test conditions as per ASTM G 99 following a design of experiment approach based on Taguchi's orthogonal arrays. The Taguchi approach leads to the recognition of most powerful variables that predominantly control the wear rate. This parametric analysis reveals that LDS content and sliding velocity affects the specific wear rate more significantly than normal load and sliding distance. Furthermore with increase in LDS content specific wear rate of the composite decreases for a constant sliding velocity. The sliding wear behavior of these composites under an extended range of test conditions is predicted by a model based on the artificial neural network (ANN).

Keywords: Epoxy composites; LD Sludge; Characterization; Sliding Wear; Artificial Neural Network (ANN)

1. Introduction

Polymers have found wide range of applications in various engineering fields due to their high strength and low density as compared to monolithic and metal alloys. For weight sensitive uses, polymers are the most suitable materials because of their lightweight, but their high cost sometimes becomes the bounding factor for many applications. Therefore easily available and low cost fillers are often used to reduce the cost of the element. Examination of the effect of such filler addition is necessary to ensure that the mechanical properties of the composites are not affected adversely by such addition. Till date large numbers of materials are used as fillers in epoxy composites [1]. The main objective of addition of fillers in to polymer can therefore be branched into two basic categories; first, to reduce the price of the element and second, to enhance the thermal, mechanical or tribological properties. There have been various reports on the use of materials such as alumina and silica mixed



into polypropylene [2-3] and polyethylene [4-5]. But very few attempts have indeed been made to utilize low cost minerals and industrial wastes in preparing particulate-reinforced polymer composites. However, this is emerging as a subject of extensive research in recent years.

An important aspect of particulate reinforced polymer composites is that the properties of the materials can be varied by controlling the filler percent and polymer combinations. A proper combination of polymer matrix and reinforcing filler material can lead to the formation of a composite with enhanced properties comparable to or even better than those of conventional metallic materials [6]. These composites materials are desired because of their high corrosion resistance, low density, low cost and ease of fabrication [7-8]. The low-cost filler like red mud has been used in some earlier studies with different polymer matrices such as polyester [9], polypropylene [10] and nylon [11] to study the wear and mechanical properties. Patnaik *et al.* [13-14] studied erosion wear response of the glass-fiber reinforced fly ash filled polyester composites using Taguchi's design-of-experiment method. Many attempts have also been made in the past on the utilization of blast furnace and steel slags [12]. Padhi *et al.* [15] studied the tribo-performance analysis of blast furnace slag filled with polymer composites while Pati *et al.* [16] studied the utilization of LD slag in erosion resistant coatings.

It has been found that about 2 to 4 tonnes of wastes are being generated per tonne of steel produced. The various solid wastes in the form of sludges and slags that are produced from steel industry are, blast furnace flue dust and sludge, blast furnace slag (BFS), LD converter slag, LD sludge etc. LD sludge particles are the fine solid particles recovered after wet cleaning of the gas emerging from LD converters during steel making. The LDS in solid lump form collected from Rourkela Steel Plant contains average particle size of about 70-100 μm with a true density of 1.5 g/cm^3 . Detailed characterization of LDS is reported by Das *et al.* [12]. They found that LDS comprised of FeO, Fe₂O₃, CaO, MgO, SiO₂, Al₂O₃ and small quantity of P and MnO.

Although few attempts have been made on the utilization of LD sludge [12] in the past, no work has so far been reported on tribo-mechanical performance of LDS produced from steel plant. In view of this, the present work includes the investigation of dry sliding wear behaviour using a pin-on-disc set up for epoxy filled with LDS with an objective to find ways to use these locally available inexpensive industrial wastes as a substitute for highly expensive fillers for wear resistance applications. Taguchi technique and Artificial Neural Network (ANN) have been used in an integrated manner in this work for parametric analysis of the wear process and to predict the sliding wear response of epoxy-LDS composites within and beyond the experimental limits.

2. Experimental details

2.1. Composite Fabrication

Linz-Donawitz sludge (LDS) collected from Rourkela steel plant, India is sieved and particles with a size range of 70–100 μm are obtained. Composites are prepared by mixing these particles with low temperature curing epoxy resin (LY556) which is used as the matrix phase. Prior to LDS addition hardener (HY951) is properly mixed with epoxy resin in 10:1 ratio by weight as recommended. Epoxy resin has density of 1.1 g/cm^3 and modulus of 3.42 GPa. To prepare 10mm diameter and 250mm length cylindrical samples, glass tubes are used, which are uniformly coated with wax at the inner surface. The dough (epoxy resin mixed with LDS) is then poured in to the glass tubes. Five samples are made with different compositions (0, 5, 10, 15 and 20 wt %) of LDS. The tubes are kept at room temperature for 24 hours and then the glass tubes are broken and samples are collected.

2.2. Sliding Wear Test

Wear tests are carried out under dry sliding conditions to evaluate the performance of these composites in a pin-on-disc type friction and sliding wear test rig (supplied by DUCOM) as per ASTM G 99. The composites slide against a disc made up of hardened steel (EN-32, hardness 72 HRC, surface roughness 0.6 μm Ra). Samples are fitted to the holder and sliding rpm is set to the desire value before applying the load. All other parameters like sliding velocity, track radius and normal load

are set before starting the test. In a pin-on-disc test the sample is held stationary and the steel disc is rotated at a required speed while a lever mechanism is used to apply the normal load. A series of tests using Taguchi's L_{25} orthogonal array are conducted with five sliding velocities of 63, 125, 190, 250, and 315 cm/s under five different normal loadings of 5, 10, 15, 20, and 25 N. After each trial, the specimen is cleaned with acetone and a precision electronic balance with accuracy ± 0.1 mg is used to measure the weight loss from the composite and the specific wear rate ($\text{mm}^3/\text{N}\cdot\text{m}$) is then expressed on 'volume loss' basis as:

$$W_s = \frac{\Delta m}{\rho t} * V_s F_n \quad (1)$$

where Δm is the mass loss in the test duration (g), ρ is the density of the composite (g/mm^3), t is the test duration (s), V_s is the sliding velocity (m/s), and F_n is the average normal load (N).

2.3. Experimental Design

Design of experiment (DOE) is a powerful tool used for analyzing and modelling the influence of control factors on performance output. The selection of the control factors is the most vital stage in the design of experiment. In the initial stage a number of factors are considered and subsequently the less important factors are eliminated leaving behind only the more important factors. Table 1 gives the various operating conditions under which wear tests are carried out. Four parameters, viz., sliding velocity, normal load, sliding distance and filler content each at five levels, are considered in this study in accordance with L_{25} orthogonal array design. The experiments are conducted at room temperature. Use of Taguchi L_{25} reduced the number of experiments from $4^5 = 1024$ conventional runs to just 25 runs thereby saving a lot of experimental time and cost. The signal to noise (S/N) ratio for minimum wear rate can be expressed as 'smaller is the better' characteristic and can be calculated as the logarithmic transformation of the loss function as shown below.

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

where, y is the observed data and n is the number of observations. For minimization of wear rate, with the above S/N ratio, 'Lower is better' (LB) characteristic is suitable.

Table 1. Levels of the variables (control factors) used in the experiment

Control factors	Levels					Units
	I	II	III	IV	V	
Sliding velocity (A)	63	125	190	250	315	cm/sec
Normal load (B)	5	10	15	20	25	N
Sliding distance (C)	500	1000	1500	2000	2500	m
LDS content (D)	0	5	10	15	20	wt %

2.4. Prediction Using Artificial Neural Network

Artificial Neural Network (ANN) is an approach motivated by the biological neural system and has been used to solve a wide variety of issues in distinct fields. It finds various applications in the field of medical science, classification and control of dynamic system, image and speech recognition etc. To develop a functional model, the multiple-layered networks with differential transfer functions are trained by using the back-propagation ANN. In the present study ANN serves as a very helpful tool for predicting the specific wear rate as it is very difficult to get a mathematical formulation for the wear behaviour. This proposed approach not only yields a sufficient understanding of the effects of process parameters but also produces an optimal parameter setting to ensure that the composites exhibit the best wear performance characteristics [17].

Table 2. Taguchi Experimental design (L_{25} orthogonal array)

Test Run	Sliding Velocity A (cm/sec)	Normal Load B (N)	Sliding Distance C (m)	LDS Content D (wt %)	Sp. Wear Rate (10^{-9} mm ³ /N-m)	S/N Ratio (dB)
1	63	5	500	0	1.612	-4.14730
2	63	10	1000	5	1.325	-2.44432
3	63	15	1500	10	0.873	1.17972
4	63	20	2000	15	0.786	2.09155
5	63	25	2500	20	0.688	3.24823
6	125	5	1000	10	1.053	-0.44857
7	125	10	1500	15	0.982	0.15777
8	125	15	2000	20	0.788	2.06948
9	125	20	2500	0	1.736	-4.79099
10	125	25	500	5	1.442	-3.17931
11	190	5	1500	20	0.864	1.26973
12	190	10	2000	0	1.823	-5.21573
13	190	15	2500	5	1.518	-3.62544
14	190	20	500	10	1.192	-1.52553
15	190	25	1000	15	1.118	-0.96884
16	250	5	2000	5	1.615	-4.16345
17	250	10	2500	10	1.316	-2.38512
18	250	15	500	15	1.214	-1.68437
19	250	20	1000	20	0.986	0.12246
20	250	25	1500	0	2.012	-6.07256
21	315	5	2500	15	1.396	-2.89771
22	315	10	500	20	1.117	-0.96106
23	315	15	1000	0	2.226	-6.95050
24	315	20	1500	5	1.834	-5.26799
25	315	25	2500	10	1.528	-3.68247

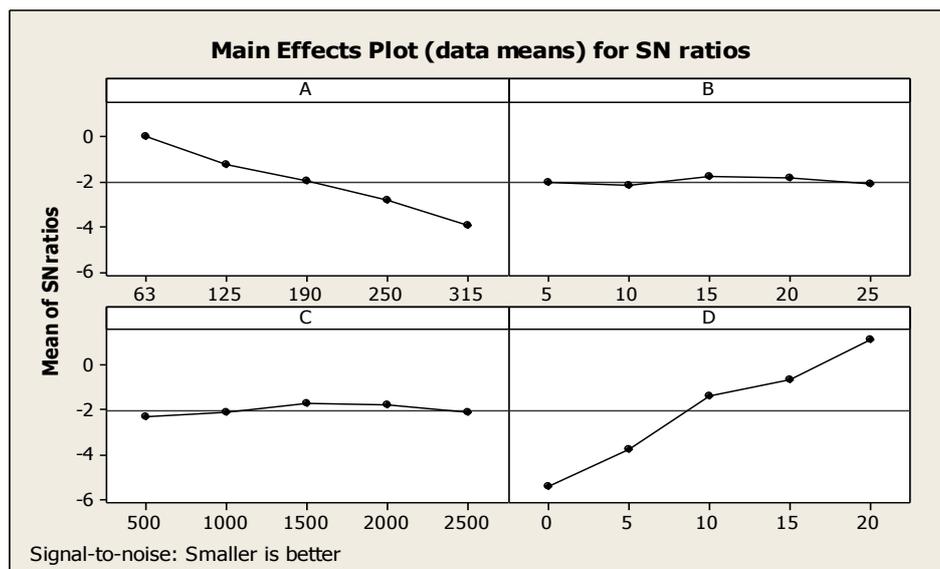
**Figure 1.** Effect of control factors on sliding wear rate of Epoxy-LDS composites

Table 3. Response table for S/N Ratios (Epoxy-LDS composites)

Level	A	B	C	D
1	-0.01442	-2.07746	-2.29951	-5.43542
2	-1.23832	-2.16969	-2.13795	-3.73610
3	-2.01316	-1.80222	-1.74667	-1.37239
4	-2.83661	-1.87410	-1.78013	-0.66032
5	-3.95195	-2.13099	-2.09020	1.14977
Delta	3.93752	0.36747	0.55285	6.58518
Rank	2	4	3	1

Table 4. Comparison of Experimental Results with ANN Predicted Values

Test run	Specific wear rate (10^{-9} mm ³ /N-m)		Error %
	Experimental	ANN Predicted	
1	1.612	1.610	0.074
2	1.325	1.210	9.481
3	0.873	0.922	5.375
4	0.786	0.798	1.583
5	0.688	0.775	11.312
6	1.053	1.060	0.728
7	0.982	0.863	13.783
8	0.788	0.785	0.328
9	1.736	1.548	12.087
10	1.442	1.406	2.539
11	0.864	0.830	4.065
12	1.823	1.816	0.363
13	1.518	1.426	6.450
14	1.192	1.232	3.273
15	1.118	1.036	7.892
16	1.615	1.638	1.410
17	1.316	1.321	0.426
18	1.214	1.194	1.602
19	0.986	1.061	7.070
20	2.012	1.951	3.077
21	1.396	1.309	6.621
22	1.117	1.129	1.114
23	2.226	2.145	3.754
24	1.834	1.892	3.068
25	1.528	1.724	11.412

3. Results and discussion

3.1. Dry Sliding Wear Analysis

The specific wear rates calculated on the basis of the test results using equation (1) and their corresponding S/N ratios obtained by Taguchi L_{25} orthogonal array for all the 25 experiments are presented in Table 2. The overall mean of the S/N ratio is found to be -1.593 dB for epoxy based composites reinforced with LD sludge. The analyses are completed using commercial software specifically used for design of experiment applications known as MINITAB 14. From the S/N ratio output, it is observed that among all the four factors considered for the analysis, the specific wear rate is mostly affected by the LDS content in the epoxy composite followed by the sliding velocity, sliding distance and normal load. The analysis shows that at sliding velocity (A) of 63 cm/s, normal load (B) of 15 N, sliding distance (C) of 1500 m and LDS content (D) of 20 wt%, the specific wear rate is minimum as evident from Figure 1. The relative effects of the control factors on specific wear rate are evident from the response table (Table 3).

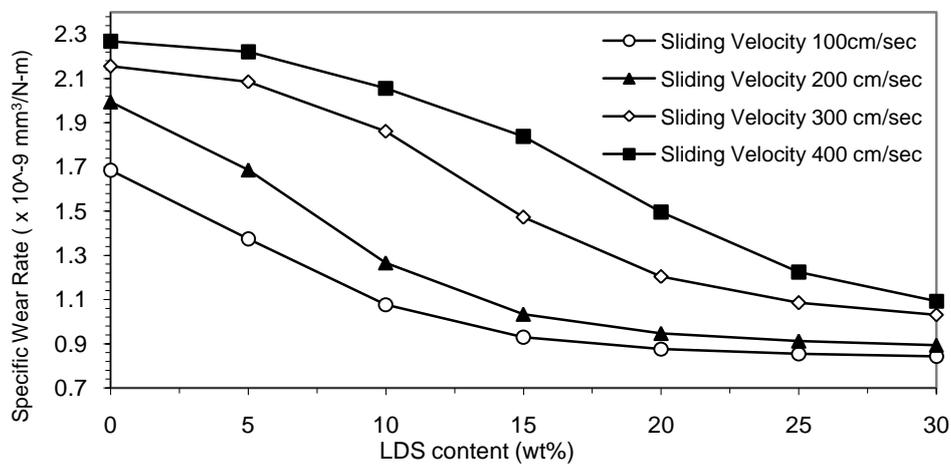


Figure 2. ANN prediction of variation in specific wear rate with LDS content for epoxy composite

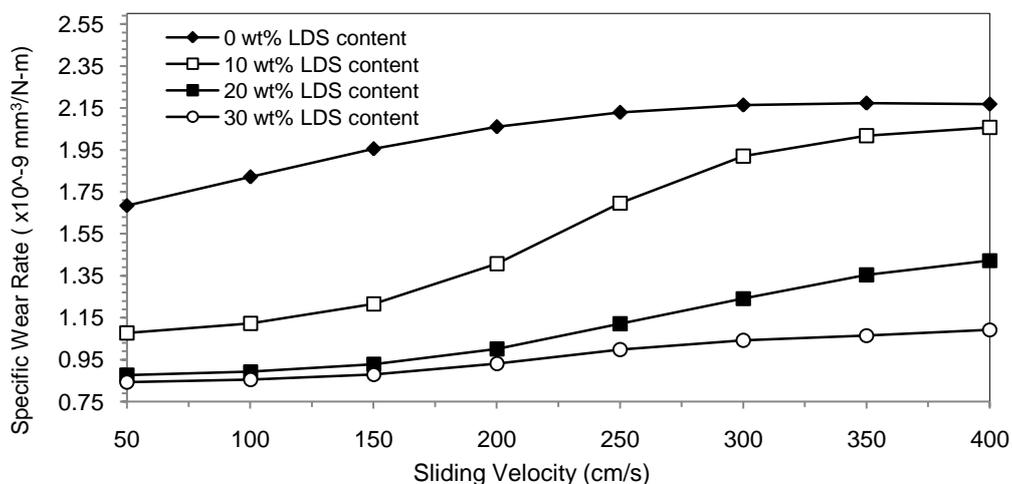


Figure 3. ANN prediction of variation in specific wear rate with sliding velocity for Epoxy composites

3.2. Artificial Neural Network for Prediction

Artificial Neural Network (ANN) is a very convenient tool to predict the output and input pattern of nonlinear problems such as sliding wear. In the present analysis LDS content, sliding velocity, sliding distance and normal load are taken as the four input parameters. The values of input parameters are normalized so that they lie in between 0 and 1. The output layer which represents the specific wear rate has one neuron. A number of networks are explored by varying the values of input training parameters and the one with least error criterion is finally selected for training. A comparison between the experimental values and the predicted values is performed and the results are shown in Table 4 with their corresponding error percentages.

The effects of two most influencing factors on the specific wear rate i.e. LDS content and sliding velocity are shown in Figure 2 and Figure 3 respectively, in which the simulated specific wear rates for various tests using ANN are presented. With increase in sliding duration, formation of wear debris of different sizes and shapes take place between the specimen and the counter surface. It is justifiable that with increase in sliding velocity with or without applied load, the epoxy resin softens due to generation of frictional heat. As a result the brittle LDS particle having sharp edges can easily tear the matrix phase of the composite and eventually get aligned along the sliding direction. The wear behaviour of the composites again depends on shape, size, hardness and brittleness of the LDS particles. Furthermore wear resistance of composites is enhanced due to presence of higher filler volume. Benefits of filler content presence are increased hardness, strength, and decrease in polymerization shrinkage, thermal expansion and contraction, water sorption, softening, staining and finally improved wear resistance. Increasing the filler content offers characteristics like bulk curing with less polymerization shrinkage, decreased wear and peackability to the composite resin. LDS filled epoxy composites show a decrease in the wear rate with the increase in the LDS content, which acts as obstacle to shear deformation during the sliding conditions. With a rise in the concentration of LD sludge the declining trend in the wear rate is significant but up to a limit.

4. Conclusions

This work suggests the use of LD sludge, an industrial waste as a potential filler material in the making of polymer composites. The compatibility of LDS particles in epoxy resin is found to be fairly good. Incorporation of LDS has resulted in substantial improvement in the wear resistance of neat epoxy. This work shows that dry sliding wear characteristics of these composites can be successfully analyzed using Taguchi experimental design. The analysis reveals that LDS content and the sliding velocity are the most predominant control factors affecting the wear rates of the composites. This work further shows that artificial neural networks have been gainfully employed for the prediction of wear response of these composites in a parameter space wider than the experimental domain.

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