DRY SLIDING WEAR RESPONSE OF
FLY ASH FILLED
POLY-ETHER-ETHER KETONE COMPOSITES

A Project Report Submitted in partial fulfilment of the Requirements for the Degree of

B. Tech.

(Mechanical Engineering)

By

ALOK KUMAR SAHOO

Roll No. 107ME022

National Institute of Technology, Rourkela

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Under the supervision of

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Rourkela - 769008

CERTIFICATE

This is to certify that the work in this thesis entitled **Dry Sliding Wear Response of Fly Ash Filled Poly-Ether-Ether-Ketone Composites** by **Alok Kumar Sahoo**, has been carried out under my supervision in partial fulfilment of the requirements for the degree of **Bachelor of Technology** in **Mechanical Engineering** during session 2010 – 2011 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/ Institute for the award of any degree or diploma.

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ABSTRACT

This paper reports the development and wear performance evaluation of a new class of poly-ether-ether-ketone **(***PEEK) based composites filled with fly ash, an industrial waste. Fly ash is generated in large amounts from furnaces in coal based thermal power plants worldwide during power generation. The fly ash particles of average size 100 micron are reinforced in PEEK resin to prepare particulate filled composites of three different compositions (0, 7.5 and 15 wt% of fly ash). Dry sliding wear trials are conducted following a well-planned experimental schedule based on design of experiments (DOE) using a standard pin-on-disc test set-up. Significant control factors predominantly influencing the wear rate are identified. Effect of fly ash content on the wear rate of PEEK composites under different test conditions is studied. The results of the experiments are compared with that of results obtained from wear test of* TiO_2*-PEEK composites under same experimental conditions. An Artificial Neural Networks (ANN) approach taking into account training and test procedure to predict the dependence of wear behavior on various control factors is implemented. This technique helps in saving time and resources for large number of experimental trials and predicts the wear response of fly ash filled PEEK composites within and beyond the experimental domain.*

CHAPTER 1

INTRODUCTION

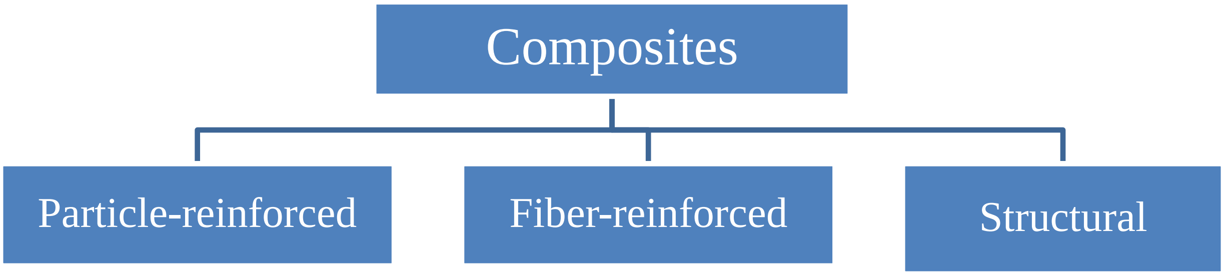
This work involves fabrication of a polymer (PEEK) composite reinforced with Fly ash particles and its subsequent response to dry sliding wear. A quick and brief knowledge of the following helps one to get acquainted with the current work and its objectives:

1.1 Composites

Composites are functional materials with specific properties, properties which cannot be obtained individually from either of metals/alloys, polymers or ceramics. A composite is usually a multi-phase material that demonstrates a significant proportion of essential properties of the constituent phases such that a desirable combination of properties is achieved. The constituent phases are often chemically dissimilar and separated by a distinct interface. Composites find applications in aerospace, sports, transportation industries, etc.

Wood and *Bone* are examples of naturally occurring composites.

The fly ash-PEEK composite is a *two-phase* composite where PEEK is the *matrix* and fly ash particles constitute the *dispersed phase*. The matrix is usually referred to the phase that is continuous and surrounds the dispersed phase. A simple classification of the composites based on the type of reinforcements is given below.



The fabricated composite is a *large particle-reinforced polymer matrix* composite since fly ash in particle form is reinforced in PEEK which is the polymer resin in liquid form.

1.2 Polyether Ether Ketone (PEEK):

PEEK is a semi-crystalline colourless organic polymer. It is a thermoplastic that has desirably very good mechanical and chemical resistance properties that are retained to high temperatures. It is highly resistant to thermal degradation as well as attack by both organic and aqueous environments. It has superior resistance to wear and dynamic fatigue. Certain physical characteristics of PEEK are given below:

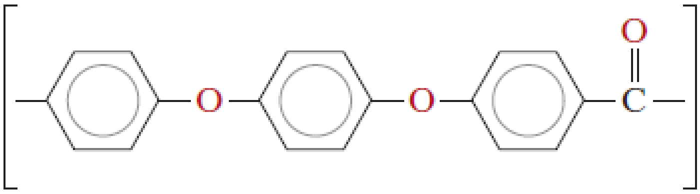
Density = 1.31 gm/cc

Modulus of elasticity = 3.6 GPa

Thermal conductivity = 0.25 W/m.K

Melting Point ~ 343ºC

The chemical structure of PEEK has the following repeating unit:



1.3 Fly ash

It is one of the residues generated in combustion of pulverized coal. It is an industrial waste and is also one of the numerous substances that cause air, water and soil pollution. The combustion of powdered coal in thermal power plants produces fly ash. The high temperature of burning coal turns the clay minerals present in the coal powder into fused fine particles. . It is abrasive and refractory in nature. Chemical analysis of fly ash shows the presence of SiO_2 (52–66%), Fe_2O_3 (6–8%), Al_2O_3 (21–27%), Ca (6%) and Ti, P, Mg, Na, S, K (< 1%).The chemical properties of the fly ash are largely influenced by the type of the coal burned (i.e., anthracite, bituminous, and lignite). Approximate 100 million tonnes of fly ash is generated in India's thermal power plant per annum. Although fly ash management has seen considerable improvement over the past few years, still its utilization level is very less. Due to increasing environmental concern and growing magnitude of the problem it has become imperative to manage fly ash. Fly ash has a number of useful applications that serve to utilize some of the large amounts being produced. It is used extensively worldwide as an extender in cement in concrete. Other outlets for fly ash include the treatment of acid mine drainage, production of zeolites as a supplementary feedstock for cement production and application as bricks and as a filler in paint. But the potential of fly ash as filler material in composite making has not been explored so far.

A picture of fly ash used in fabrication of fly ash - PEEK composite

1.4 Wear

Wear is defined as progressive loss of material volume from a solid surface due to the damage caused as a result of relative motion between the surface and an external surface/substance in contact.

Wear is usually classified into the following categories:

- *Adhesive wear*
- *Abrasive wear*
- *Surface fatigue*
- *Fretting wear*
- *Erosive wear*

The wear test (dry sliding wear test) performed on the fabricated composite is a type of Adhesive wear. Adhesive wear involves material transfer from one surface to another due to direct contact and plastic deformation. Dry sliding wear involves sliding of one surface over other under the application of a load normal to the plane of motion.

CHAPTER 2

LITERATURE REVIEW

In recent past, there has been a shift in trends of engineering and scientific research from conventional materials to hybrid/composite materials. It has been observed that by incorporating hard filler particles into polymer based composites; synergistic effects may be achieved in the form of higher modulus and reduced material cost (Pukanszky, 1995; Acosta et.al, 1986; Gregory et.al, 2003).The inclusion of such particulate fillers into polymers for commercial applications is primarily aimed at the cost reduction and stiffness improvement (Rothon, 1997; Rothon 1999). However, study 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. Available references show that a large number of materials being used as fillers in polymers (Katz et. al, 1997; Zhenyu et. al, 2008; Chang et.al 2005; Satapathy et. al, 2008). Many kinds of polymer–matrix composites reinforced with particulates have a wide range of industrial applications such as heaters, electrodes (Jang, 1994), composites with thermal durability at high temperature (Jung-il et. al, 2004) etc. Ceramic filled polymer composites have also been the subject of extensive research in recent years and consequently, a number of reports are available on the use of ceramics such as Al_2O_3, SiC etc. as particulate fillers (Patnaik et. al, 2009).Now a days size of the particle being reduced and studies are being done to study the effect of particulate in composite (W.J. Cantwell et.al 1994; M. Imanaka et.al 2001; H. Wang et.al 2002). Use of industrial wastes like Red Mud as a filler has been explored previously owing to its ease of availability and low cost and its usage reduces the burden on the environment (Satapathy, et. al, 2009). But the potential of fly ash as a filler material in PEEK matrix has not been reported so far.

Wear as a mode of failure has generated a lot of interest due to its effect on the reliability of components and the costs involved in replacement of worn out parts. A lot of time is wasted which also results in loss of productivity. Wear process in composites has been observed to depend significantly on the characteristics of the filler materials (Khedkar Jaydeep et. al, 2002). Thus a sliding wear analysis has been carried out in the current work in order to determine the effect of micro-sized fly ash particle content on the specific wear rate. Study reveals that sliding wear in composites also depends on speed, load and the distance of sliding. Wear is said to occur in an interacting environment since a change in either load, sliding distance, filler content and speed or a combination of any of the parameters is found to change the wear rate (Suresha S. et.al, 2010).Research shows that plastic-elastic damage of composite depends upon particle size. A ductile interphase is considered in the frame of incremental damage theory to analyse the above dependence (Yang Hui et.al 2011). In order to design the composites for specific functions, a proper correlation between wear behaviour and the parameters affecting it needs to be done. The analysis of the effect of characteristic parameters on the wear behaviour has been successfully conducted by employing the design of experiments strategy by Taguchi’s experimental technique (Patnaik et.al, 2009; Basavarajappa S. et.al, 2005; Ross P. J. 1993; Taguchi G. 1993; Chauhan S.R. et.al, 2009). Taguchi’s parameter design can optimize the performance characteristics through the setting of design parameters and reduce the sensitivity of the source of variation (Basavarajappa S. et. al, 2007; Roy K. R. 1990). This is carried by the efficient use of experimental runs to the combinations of variables to be studied. This technique is a powerful tool for acquiring the data in a controlled way and to analyze the influence of process parameters over some specific parameters, which is unknown function of these process variables. The crucial stage in the plan of experiments is selection of factors which have effects on the process. Taguchi technique creates a standard orthogonal array to consider the effect of several factors on the target value and defines the plan of experiments. The experimental results are analyzed by using analysis of means and variance of the influence of factors (Taguchi G. 1993).

A novel technique like artificial neural networks (ANN) which is inspired by the biological neural system and has been used to solve a wide variety of problems in diverse fields is used to analyse and predict the wear response under different test conditions (Zhang et. al, 2003). Review of past research suggests that artificial neural networks has been successfully employed in the prediction of wear behaviour to a set of inputs within and beyond the experimental domain which thereby helps in saving time and resources for a large number of experimental trials (Kranthi Ganguluri et. al, 2010; Satapathy A et. al, 2010). A set of experimental data is used as a training set for the ANN function. Another set of data can be used for testing the function to obtain the required accuracy, after which the prediction for a random set of data can be done.

The objectives of this work are outlined as follows:

1. Fabrication of a set of matrix composites with and without ceramic fillers.
2. Parametric appraisal of dry sliding wear process of unfilled and fly ash filled PEEK composites using Taguchi experimental design.
3. Prediction of wear rate at different operating conditions using ANN.

CHAPTER 3

EXPERIMENTAL DETAILS

3.1 Fabrication of composite

Fly ash particles with average size 100 micro-meter, collected from NSPCL, Rourkela are reinforced in PEEK (Poly ether ether ketone) to prepare the composite. Two percent (2%) cobalt napthalate (as accelerator) is mixed thoroughly in PEEK followed by 2% methyl-ethyl-ketone peroxide (MEKP) as hardener resin prior to reinforcement. The composite are cast by conventional hand-lay-up technique in glass tubes so as to get cylindrical specimens (dia. 9 mm, length 120 mm). Before casting the glass tubes are lined with wax for easy removal. The castings are left to cure at room temperature for about 24 h after which the tubes are broken and samples are released. Composites of three different compositions C1, C2 and C3 (filled with 0, 7.5, and 15 wt% of fly ash) are made. Specimens of suitable dimension are cut using a diamond cutter for further physical characterization and wear test. A similar process is employed for the fabrication of TiO_2 reinforced PEEK composite.

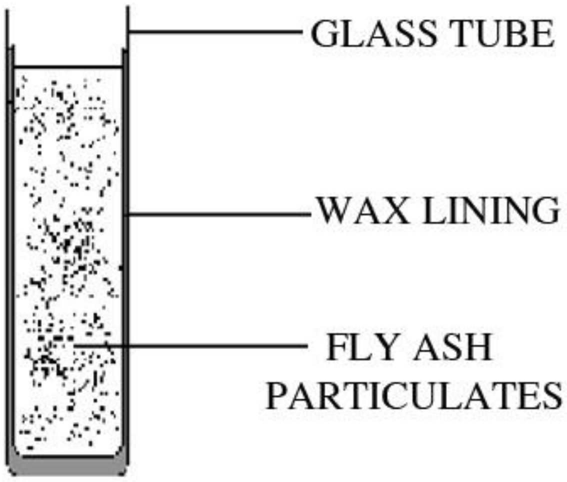


Fig. 1 Hand lay-up technique

3.2 Sliding Wear Test

To evaluate the performance of these composites under dry sliding condition, wear tests are carried out in a pin-on-disc type friction and wear monitoring test rig (supplied by DUCOM) as per ASTM G99. The counter body is a disc made of hardened ground steel (EN-32, hardness 72 HRC, surface roughness 0.6 m Ra). The specimen is held stationary and the disc is rotated while a normal force is applied through a lever mechanism. A series of tests are conducted with three sliding velocities of 165, 250, and 335 cm/s under three different normal loadings of 5, 10 and 15 N. The material loss from the composite surface is measured using a precision electronic balance with accuracy ±0.1 mg and the specific wear rate (mm^3/ N-m) is then expressed on 'volume loss' basis as:

$$W_s = \triangle m / (\rho V_s t F_n) \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where,

$\triangle m$ is the mass loss in the test duration (gm)

ρ is the density of the composite (gm/mm^3)

t is the test duration (s)

V_s is the sliding velocity (cm/s)

F_n is the average normal load (N).

The specific wear rate is defined as the volume loss of the specimen per unit sliding distance per unit applied normal load.

Fig. 2 Wear measuring test rig

3.3 Scanning Electron Microscopy

The worn surfaces of the specimens are examined directly by scanning electron microscope JEOL JSM-6480LV. The worn samples are mounted on stubs with silver paste. To enhance the conductivity of the samples, a thin film of platinum is vacuum evaporated onto them before the photomicrographs are taken.

3.4 Experimental Design

Design of experiment is a powerful analysis tool for modeling and analyzing the influence of various control factors on performance output. The most important step in the design of experiment is the selection of the control factors. Therefore, a number of factors are included so that non-significant variables can be identified at the earliest opportunity. The wear tests are carried out under operating conditions given in Table 1. Four parameters, viz., sliding velocity, fly ash content, normal load and sliding distance each at three levels, are considered in this study in accordance with L_9 (3^4) orthogonal array design.

Each of the experimental observations is transformed into a signal-to-noise (S/N) ratio. There are several S/N ratios available depending on the type of characteristics. The S/N ratio for minimum erosion rate coming under *smaller-is-better* characteristic, which can be calculated as logarithmic transformation of the loss function as shown below.

Smaller is the better characteristic:

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

Where n is the number of observations, and y is the observed data.

Smaller-is-better characteristic, with the above S/N ratio transformation, is suitable for minimization of erosion rate.

Symbols	**Control Factors**	Level			
		I	II	III	Units
Factor A	Sliding Velocity	165	250	335	cm/sec
Factor B	Normal Load	5	10	15	N
Factor C	Filler content	0	7.5	15	wt%
Factor D	Sliding distance	1200	1600	2000	m

Table 1 Control factors and their selected levels for dry sliding wear test

CHAPTER 4

RESULTS & DISCUSSION

4.1 Dry Sliding Wear Test Results

The specific wear rates obtained for all the 9 test runs along with the corresponding S/N ratio are presented in below table.

Test Run	Sliding Velocity (cm/sec) **A**	Load (N) **B**	Filler (fly ash) content (wt %) **C**	Sliding Distance (m) **D**	Specific Wear Rate (mm^3/ N-m) **Ws**	S/N ratio (dB)
1	165	5	0	1200	1.920	-5.66602
2	165	10	7.5	1600	1.527	-3.67678
3	165	15	15	2000	1.265	-2.04181
4	250	5	7.5	2000	1.844	-5.31522
5	250	10	15	1200	1.586	-4.00606
6	250	15	0	1600	1.901	-5.57964
7	335	5	15	1600	1.911	-5.62521
8	335	10	0	2000	2.275	-7.13963
9	335	15	7.5	1200	2.176	-6.75318

Table 2 Specific wear rates of fly ash filled composite obtained for different test conditions with S/N ratios

From this table 2, the overall mean for the S/N ratio of the wear rate of Fly ash filled composite is found to be – 5.0893 dB and for TiO_2 filled composite it is -3.4174 dB (from table 3). This is done using the software MINITAB 14 specifically used for design of experiment applications. The S/N ratio response analysis shows that among all the factors, sliding velocity is the most significant factor followed by filler content and normal load while the sliding distance has the least or almost no significance on wear rate of the particulate filled composites under this investigation. The analysis of the results leads to the conclusion that factor combination of A_1, B_3, and C_3 gives the minimum specific wear rate.

Test Run	Sliding Velocity (cm/sec) **A**	Load (N) **B**	Filler (TiO_2) content (wt %) **C**	Sliding Distance (m) **D**	Specific Wear Rate (mm^3/ N-m) W_s	S/N ratio (dB)
1	165	5	0	1200	1.622	-4.20102
2	165	10	7.5	1600	1.145	-1.17611
3	165	15	15	2000	1.026	-0.22295
4	250	5	7.5	2000	1.448	-3.21537
5	250	10	15	1200	1.186	-1.48169
6	250	15	0	1600	1.501	-3.52761
7	335	5	15	1600	1.691	-4.56287
8	335	10	0	2000	2.075	-6.34036
9	335	15	7.5	1200	2.002	-6.02928

Table 3 Specific wear rates of TiO_2 filled composite obtained for different test conditions with S/N ratios

The effects of individual control factors as obtained from the Taguchi analysis are shown in Figure 1. The S/N ratio response are given in Table 4, from which it can again be concluded that among all the factors, sliding velocity is the most significant factor followed by filler content and normal load while the sliding distance has the least or almost no significance on wear of the particulate composite.

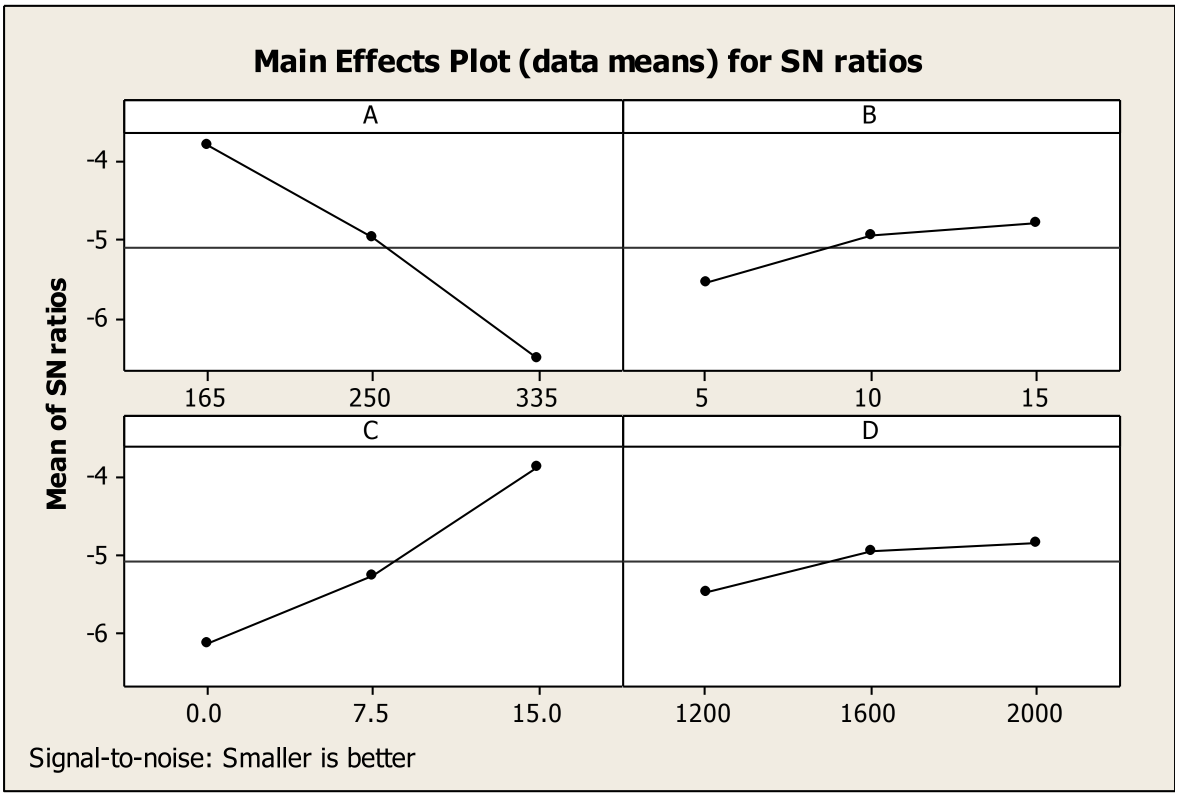


Fig. 3 Effect of Control Factors on Specific Wear Rate for fly ash filled composite

Level	**A**	**B**	**C**	**D**
1	-3.795	-5.535	-6.128	-5.475
2	-4.967	-4.941	-5.248	-4.832
3	-6.506	-4.792	-3.891	-4.832
Delta	2.711	0.744	2.237	0.643
Rank	1	**3**	**2**	**4**

Table 4 Response Table of fly ash filled composite for Signal to Noise Ratios (Smaller is better)

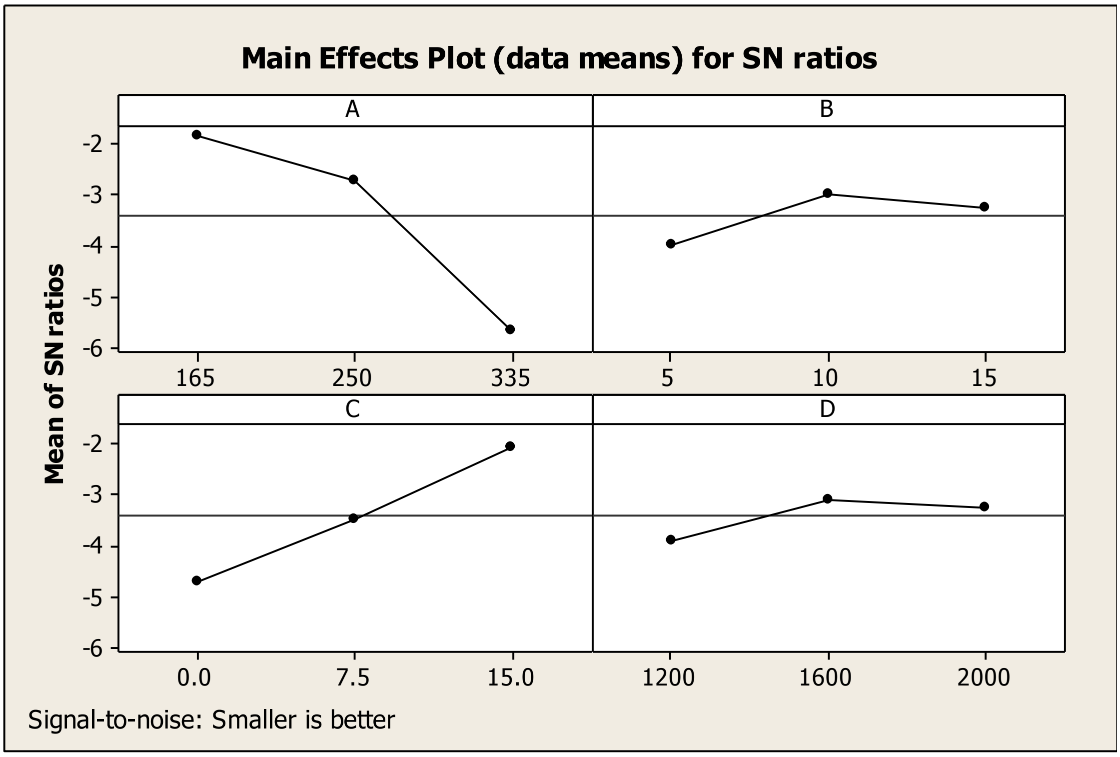


Fig. 4 Effect of Control Factors on Specific Wear Rate for TiO_2 filled composite

Level	A	B	C	D
1	-1.867	-3.993	-4.690	-3.904
2	-2.742	-2.999	-3.474	-3.089
3	-5.644	-3.260	-2.089	-3.260
Delta	3.777	0.994	2.600	0.815
Rank	**1**	**3**	**2**	**4**

Table 5 Response Table of TiO_2filled composite for Signal to Noise Ratios (Smaller is better)

4.2 Factor Settings for Minimum Specific Wear Rate of Fly Ash – PEEK Composites

In this study, an attempt is made to derive a predictive correlation in terms of the significant control factors for determination of specific wear rate of these fly ash filled PEEK composites. The single-objective function requires quantitative determination of the relationship between erosion rates with combination of control factors. In order to express, erosion rate in the form of a mathematical model the following correlation is suggested.

$$Ws = K_0 + K_1 \times A + K_2 \times B + K_3 \times C \qquad (3)$$

Here, W_s is the performance output term and K_{i} (i = 0, 1…3) are the model constants. The constants are calculated using non-linear regression analysis with the help of SYSTAT 7 software and the following relations are obtained:

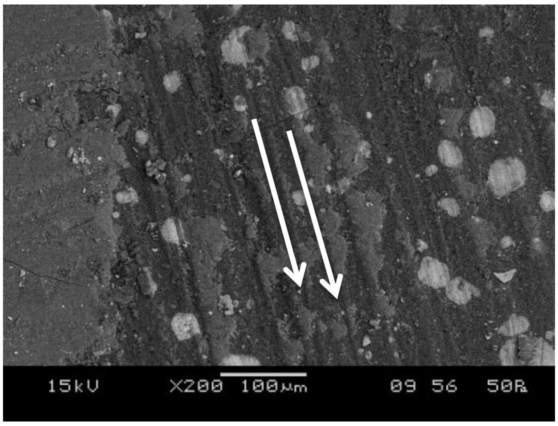
$$Ws = 1.347 \ + \ 0.003 \times A - 0.011 \times B - 0.03 \times C \qquad (4)$$

(r^2=0.999)

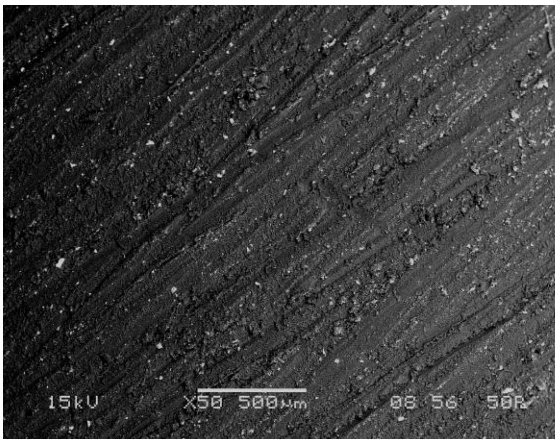
The correctness of the calculated constants is confirmed as high correlation coefficients (r^2) in the tune of 0.999 is obtained for Eq. (3) and therefore, the model is quite suitable to use for predictive purpose.

4.3 Surface Morphology

The morphology of the worn surface of the PEEK composite with 7.5 wt% fly ash is illustrated in Figure 5. This micrograph is taken after 20 minutes of test duration with a sliding velocity of 165 cm/s under a normal load of 10 N. It can be seen that there is a plastic flow of the matrix material in the sliding direction which is indicated by the arrows (Figure 5a). It is understandable that with increase in applied load and/or sliding velocity, PEEK softens due to frictional heat generation. As a result, the fly ash particles, which are brittle in nature and have sharp edges, easily tear the matrix and gradually get aligned along the sliding direction, as seen in Figure 5(b). These particles by virtue of their size, shape, brittleness and high hardness influence modify the wear behavior of the composites. Longer duration of sliding results in scars on the particulate bodies as well and also results in formation of wear debris of different sizes and shapes.



(a)



(b)

Fig. 5 SEM micrographs of the worn composite surfaces

4.4 Comparison of TiO_2 filled composite with fly ash filled composite

TiO_2 is a well-known filler material which has high abrasive strength and its composite has already been used in many industrial applications. Although it has good filler characteristics the cost of titania is very high and it is not readily available. This motivates to explore the wear characteristics of fly ash as cheap replacement filler. Table 6 compares the specific wear rate for titania with fly ash filled composite for different test runs. It is found that under same experimental conditions the titania filled composite shows better resistance to wear than fly ash filled composite.

Test Run	Sliding Velocity (cm/sec) **A**	Load (N) **B**	Filler content (wt %) **C**	Sliding Distance (m) **D**	Specific Wear Rate (mm^3/ N-m) **Ws (Fly ash)**	Specific Wear Rate (mm^3/ N-m) **Ws (Titania)**
1	165	5	0	1200	1.920	1.622
2	165	10	7.5	1600	1.527	1.145
3	165	15	15	2000	1.265	1.026
4	250	5	7.5	2000	1.844	1.448
5	250	10	15	1200	1.586	1.186
6	250	15	0	1600	1.901	1.501
7	335	5	15	1600	1.911	1.691
8	335	10	0	2000	2.275	2.075
9	335	15	7.5	1200	2.176	2.002

Table 6 Comparison of specific wear rate between TiO_2 filled composite with fly ash filled composite for different test runs

Figure 6 compares the specific wear rate of titania filled composite with fly ash filled composite for different test runs graphically. It is seen that although titania shows better wear behaviour as a filler material, the wear rates obtained for fly ash are comparable with that of titania. Thus, fly ash can be considered as feasible replacement for titania as a filler material.

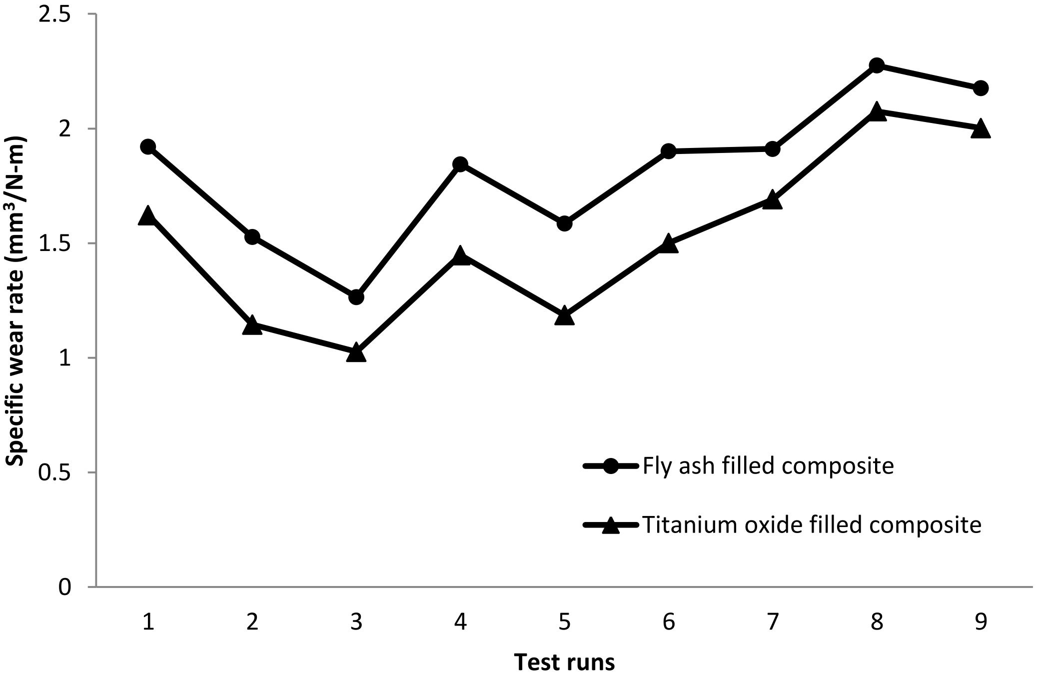


Fig. 6 Comparison of sp. wear rate of fly ash & titania filled composites for different test runs

4.5 Analysis using ANN

Wear process is considered as a non-linear problem with respect to its variables: either materials or operating conditions. To obtain minimum wear rate, appropriate combinations of operating parameters have to be planned. In this work, a statistical method, responding to the constraints, is implemented to correlate the operating parameters. This methodology is based on artificial neural networks (ANN), which is a technique that involves database training to predict input-output evolutions. In the present analysis, the sliding velocity, filler content and normal load are taken as the three input parameters. As already described, each of these parameters is characterized by one neuron and consequently the input layer in the ANN structure has three neurons. Different ANN structures with varying number of neurons in the hidden layer are tested at constant cycles, learning rate, error tolerance, momentum parameter, noise factor and slope parameter. Based on least error criterion, one structure, shown in Table 7, is selected for training of the input-output data. A software package NEURALNET for neural computing using back propagation algorithm is used as the prediction tool for specific wear rate of the composite samples under various test conditions. The three-layer neural network having an input layer (I) with three input nodes, a hidden layer(H) with ten neurons and an output layer (O) with one output node used in this work is shown in Figure 7.

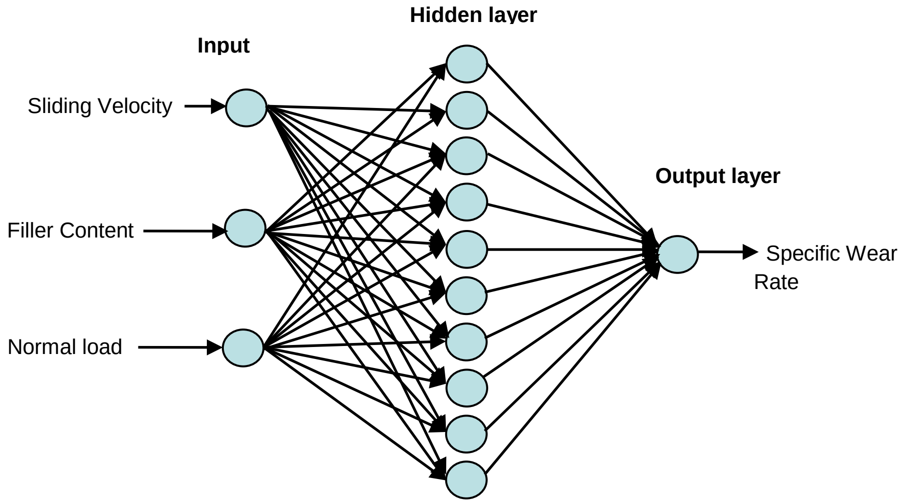


Fig. 7 The three layer neural network

Input parameters for training	**Values**
No. of input layer neurons (I)	3
No. of hidden layer neurons (H)	10
No of output layer neurons (O)	1
No. of epochs	10^7
Slope parameter (£)	0.6
Learning rate (β)	0.5
Momentum parameter (α)	0.7
Noise factor (NF)	0.7
Error tolerance	0.01

Table 7 Input parameters selected for training in NEURALNET

The error tolerance limit was chosen as 5% and with the above selected input parameters the prediction results were found to lie well within the limit.

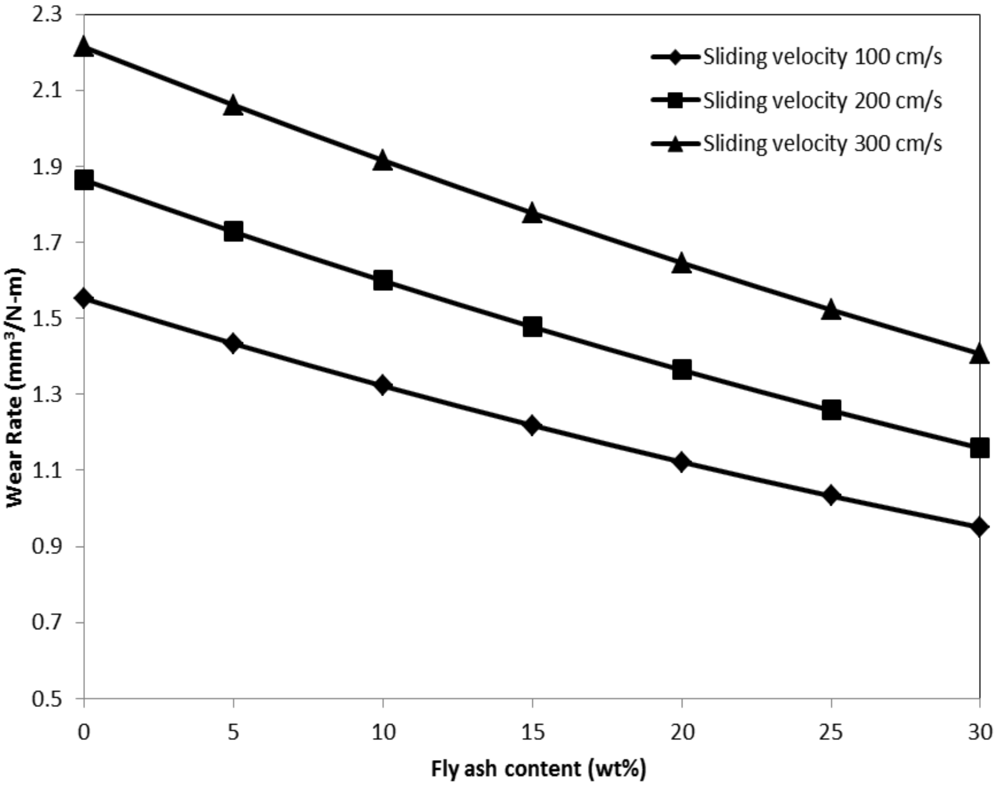


Fig. 8 Variation of specific wear rate with fly ash content at different sliding velocities

Keeping normal load constant at 10N, the simulated specific wear rates indicating the effects of varying filler (fly ash) contents and sliding velocities are presented in Figure 8. It is interesting to note that while the specific wear rate decreases almost exponentially with the increase in fly ash content in the composite, indicating an improvement in the wear resistance of the composite, it increases with increase in the sliding velocity. The presence of fly ash particles seems to have helped in restricting the mass loss from the composite surface due to sliding wear.

CHAPTER 5

CONCLUSION

Conclusions

Dry sliding wear characteristics of these composites can be experimented following a design-of-experiment approach. This study reveals that fly ash possesses good filler characteristics as it improves the sliding wear resistance of the composite. Artificial neural network (ANN) technique is successfully applied in this investigation to predict and simulate the wear response of the composites under various test conditions within and beyond the experimental domain. The predictions of wear rates as functions of filler content and testing conditions thus prove a remarkable capability of well-trained neural networks for modeling concern.

Scope for future work

This work leaves a wide scope for future research. The composites of similar nature can be tested for wear modes other than sliding. Thermoset polymers such as epoxy or polyester can be used as the matrix material and potential of industrial wastes other than fly ash can be explored.

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Dry Sliding Wear Response of LD Slag Filled Poly-ether-ether-ketone Composites

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Abstract

This work reports the development and wear performance evaluation of a new class of poly-ether-ether-ketone **(***PEEK) based composites filled with an industrial waste called LD slag (LDS). LDS is generated in great amounts from LD furnaces in steel plants worldwide during steel making. The slag particles of average size 100 μm are reinforced in PEEK resin to prepare particulate filled composites of three different compositions (0, 7.5 and 15 wt% of LDS). Dry sliding wear trials are conducted following a well planned experimental schedule based on design of experiments (DOE) using a standard pin-on-disc test set-up. Significant control factors predominantly influencing the wear rate are identified. Effect of LDS content on the wear rate of PEEK composites under different test conditions is studied. An Artificial Neural Network (ANN) approach taking into account training and test procedure to predict the dependence of wear behavior on various control factors is implemented. This technique helps in saving time and resources for large number of experimental trials and predicts the wear response of LDS filled PEEK composites within and beyond the experimental domain.*

Keywords: Polymer composites, PEEK, LD Slag, sliding wear, Artificial Neural Networks, Design of Experiment

Introduction

Significant quantities of sludge and slag are generated as waste material or byproduct every day from steel industries. Steel plant slags mainly include blast furnace slag and steel melting slag known as Linz–Donawitz converter slag (LDS). The main chemical components of typical LDS samples generated in steel plant are Fe, CaO, and SiO_2. LD slag can be utilized in many areas such as soil conditioners, fertilizers, and recovery of metal values etc. (Das et. al, 2007). But the potential of this slag as filler material in composite making has not been explored so far. It has been observed that by incorporating hard filler particles into polymer based composites, synergistic effects may be achieved in the form of higher modulus and reduced material cost (Pukanszky, 1995; Acosta et.al, 1986; Gregory et.al, 2003). The inclusion of such particulate fillers into polymers for commercial applications is primarily aimed at the cost reduction and stiffness improvement (Rothon, 1997; Rothon 1999). However, study 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. Available references suggest a large number of materials being used as fillers in polymers (Katz et. al, 1997; Zhenyu et. al, 2008; Chang et.al 2005; Satapathy et. al, 2008). Various kinds of polymers and polymer–matrix composites reinforced with metal particles have a wide range of industrial applications such as heaters, electrodes (Jang, 1994), composites with thermal durability at high temperature (Jung-il et. al, 2004) etc. Ceramic filled polymer composites have also been the subject of extensive research in recent years and consequently, a number of reports are available on the use of ceramics such as Al_2O_3, SiC etc. as particulate fillers (Patnaik et. al, 2009). But the potential of LDS as a filler material in PEEK matrix has not been reported so far. This work investigates and analyses the sliding wear response of these PEEK composites filled with micro-sized LDS particles using a novel technique like artificial neural networks (ANN) which is inspired by the biological neural system and has been used to solve a wide variety of problems in diverse fields (Zhang et. al, 2003).

Experimental Details

Sliding Wear Test

To evaluate the performance of these composites under dry sliding condition, wear tests are carried out in a pin-on-disc type friction and wear monitoring test rig (supplied by DUCOM) as per ASTM G 99. The counter body is a disc made of hardened ground steel (EN-32, hardness 72 HRC, surface roughness 0.6 m Ra). The specimen is held stationary and the disc is rotated while a normal force is applied through a lever mechanism. A series of tests are conducted with three sliding velocities of 165, 250, and 335 cm/s under three different normal loadings of 5, 10 and 15 N. The material loss from the composite surface is measured using a precision electronic balance with accuracy ±0.1 mg and the specific wear rate (mm^3/ N-m) is then expressed on 'volume loss' basis as:

$$\mathbf{Ws = \Delta m / (\rho\ t\ Vs\ Fn)} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where Δm is the mass loss in the test duration (gm), ρ is the density of the composite (gm/mm^3), t is the test duration (s), Vs is the sliding velocity (m/s), and Fn is the average normal load (N). The specific wear rate is defined as the volume loss of the specimen per unit sliding distance per unit applied normal load.

Scanning electron microscopy

The worn surfaces of the specimens are examined directly by scanning electron microscope JEOL JSM-6480LV. The worn samples are mounted on stubs with silver paste. To enhance the conductivity of the samples, a thin film of platinum is vacuum evaporated onto them before the photomicrographs are taken.

Experimental Design

Design of experiment is a powerful analysis tool for modeling and analyzing the influence of control factors on performance output. The most important stage in the design of experiment lies in the selection of the control factors. Therefore, a number of factors are included so that non-significant variables can be identified at the earliest opportunity. The wear tests are carried out under operating conditions given in Table 1. Four parameters, viz., sliding velocity, LDS content, normal load and sliding distance each at three levels, are considered in this study in accordance with L_9 (3^4) orthogonal array design.

Each of the experimental observations is transformed into a signal-to-noise (S/N) ratio. There are several S/N ratios available depending on the type of characteristics. The S/N ratio for minimum erosion rate coming under *smaller-is-better* characteristic, which can be calculated as logarithmic transformation of the loss function as shown below.

Smaller is the better characteristic: $\frac{S}{N} = -10\log\frac{1}{n}\left(\sum y^2\right)$ …….. (2)

Where n is the number of observations, and y is the observed data. *Smaller-is-better* characteristic, with the above S/N ratio transformation, is suitable for minimization of erosion rate.

Results and Discussion

Dry Sliding Wear Test Results

The specific wear rates obtained for all the 9 test runs along with the corresponding S/N ratio are presented in Table 2. From this table, the overall mean for the S/N ratio of the wear rate is found to be – 4.36078 dB. This is done using the software MINITAB 14 specifically used for design of experiment applications. The S/N ratio response analysis shows that among all the factors, sliding velocity is the most significant factor followed by filler content and normal load while the sliding distance has the least or almost no significance on wear rate of the particulate filled composites under this investigation. The analysis of the results leads to the conclusion that factor combination of A_1, B_3, and C_3 gives the minimum specific wear rate. The effects of individual control factors as obtained from the Taguchi analysis are shown in Figure 1. The S/N ratio response are given in Table 3, from which it can again be concluded that among all the factors, sliding velocity is the most significant factor followed by filler content and normal load while the sliding distance has the least or almost no significance on wear of the particulate composite.

Surface Morphology

The morphology of the worn surface of the PEEK composite with 7.5 wt% LDS is illustrated in Figure 2. This micrograph is taken after 20 minutes of test duration with a sliding velocity of 165 cm/s under a normal load of 10 N. It can be seen that there is a plastic flow of the matrix material in the sliding direction which is indicated by the arrows (Figure 2a). It is understandable that with increase in applied load and/or sliding velocity, PEEK softens due to frictional heat generation. As a result, the LDS particles, which are brittle in nature and have sharp edges, easily tear the matrix and gradually get aligned along the sliding direction, as seen in Figure 2b. These particles by virtue of their size, shape, brittleness and high hardness influence modify the wear behavior of the composites. Longer duration of sliding results in scars on the particulate bodies as well and also results in formation of wear debris of different sizes and shapes.

Analysis using ANN

Wear process is considered as a non-linear problem with respect to its variables: either materials or operating conditions. To obtain minimum wear rate, appropriate combinations of operating parameters have to be planned. In this work, a statistical method, responding to the constraints, is implemented to correlate the operating parameters. This methodology is based on artificial neural networks (ANN), which is a technique that involves database training to predict input-output evolutions. In the present analysis, the sliding velocity, filler content and normal load are taken as the three input parameters. As already described, each of these parameters is characterized by one neuron and consequently the input layer in the ANN structure has three neurons. Different ANN structures with varying number of neurons in the hidden layer are tested at constant cycles, learning rate, error tolerance, momentum parameter, noise factor and slope parameter. Based on least error criterion, one structure, is selected for training of the input-output data. A software package NEURALNET for neural computing using back propagation algorithm is used as the prediction tool for specific wear rate of the composite samples under various test conditions. The three-layer neural network having an input layer (I) with three input nodes, a hidden layer (H) with ten neurons and an output layer (O) with one output node used in this work is shown in Figure 3.

The simulated specific wear rates indicating the effects of varying filler (LDS) contents and sliding velocities are presented in Figure 4. It is interesting to note that while the specific wear rate decreases almost exponentially with the increase in LDS content in the composite, indicating an improvement in the wear resistance of the composite, it increases with increase in the sliding velocity. The presence of LDS particles seems to have helped in restricting the mass loss from the composite surface due to sliding wear.

Conclusions

Dry sliding wear characteristics of these composites can be experimented following a design-of-experiment approach. This study reveals that LDS possesses good filler characteristics as it improves the sliding wear resistance of the composite. Artificial neural network (ANN) technique is successfully applied in this investigation to predict and simulate the wear response of the composites under various test conditions within and beyond the experimental domain. The predictions of wear rates as functions of filler content and testing conditions thus prove a remarkable capability of well-trained neural networks for modeling concern.

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Tables and Figures

Table 1 Control factors and their selected levels for dry sliding wear test

Symbols	**Control Factors**	Level			
		I	II	III	Units
Factor A	Sliding Velocity	165	250	335	cm/sec
Factor B	Normal Load	5	10	15	N
Factor C	Filler content	0	7.5	15	wt%
Factor D	Sliding distance	1200	1600	2000	m

Table 2 Specific wear rates obtained for different test conditions with S/N ratios

Test Run	Sliding Velocity (cm/sec) **A**	Load (N) **B**	Filler (LDS) content (wt %) **C**	Sliding Distance (m) **D**	Specific Wear Rate (mm^3/ N-m) **W_s**	S/N ratio (dB)
1	165	5	0	1200	1.8000	-5.10545
2	165	10	7.5	1600	1.3470	-2.58735
3	165	15	15	2000	1.0851	-0.70940
4	250	5	7.5	2000	1.6443	-4.31962
5	250	10	15	1200	1.4362	-3.14430
6	250	15	0	1600	1.7619	-4.91963
7	335	5	15	1600	1.8957	-5.55539
8	335	10	0	2000	2.1475	-6.63866
9	335	15	7.5	1200	2.0576	-6.26722

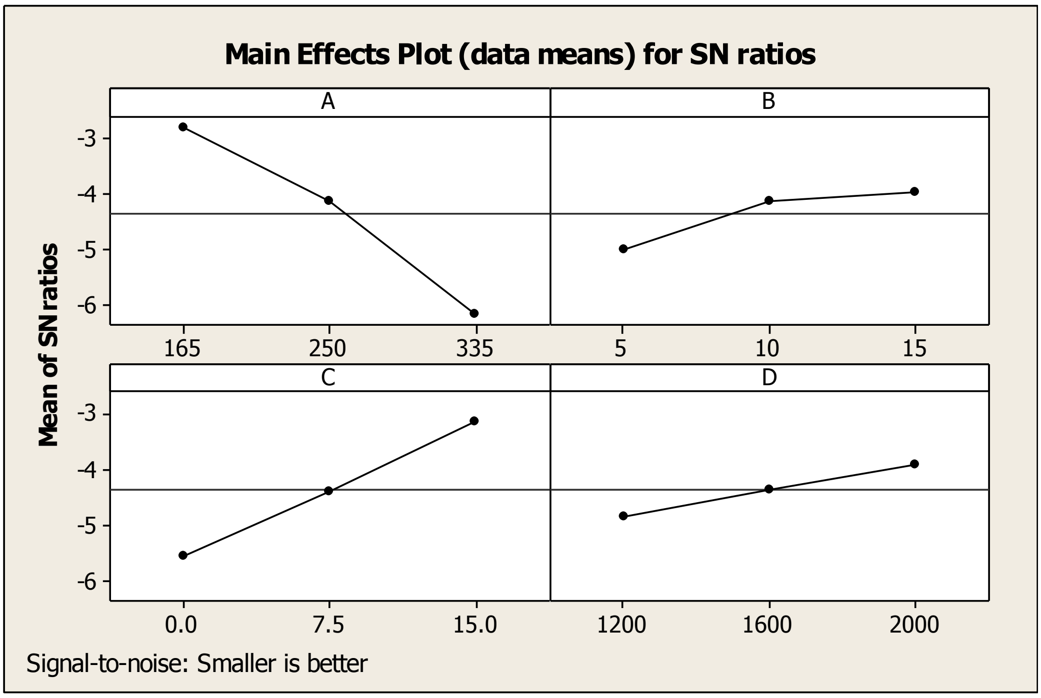
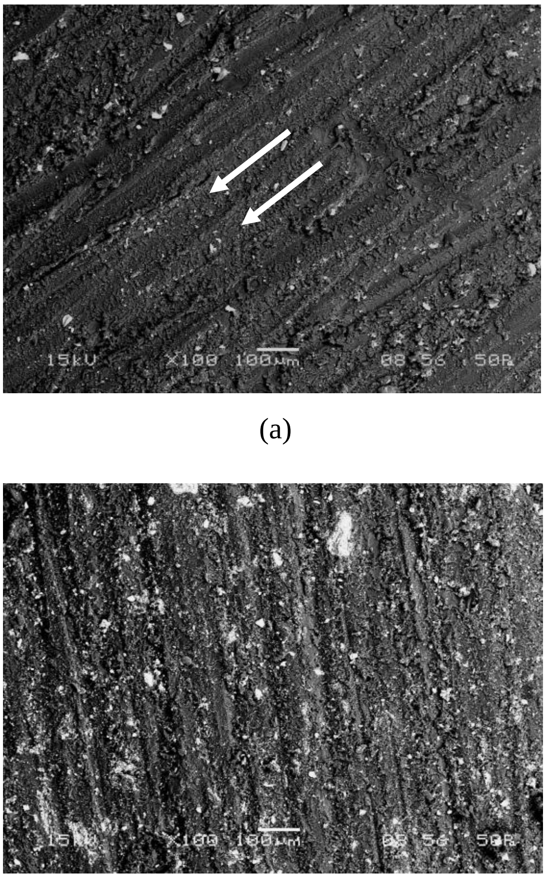


Figure 1 Effect of Control Factors on Specific Wear Rate

Table 3 Response Table for Signal to Noise Ratios (Smaller is better)

Level	A	B	C	D
1	-2.801	-4.993	-5.555	-4.839
2	-4.128	-4.123	-4.391	-4.354
3	-6.154	-3.965	-3.136	-3.889
Delta	3.353	1.028	2.418	0.950
Rank	**1**	**3**	**2**	**4**



(a)

(b)

Figure 2 SEM micrographs of the worn composite surfaces

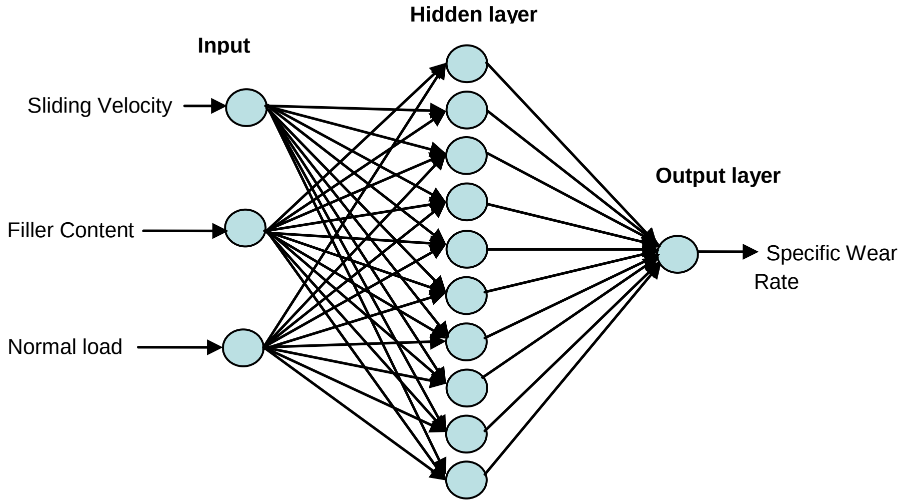


Figure 3 The three layer neural network

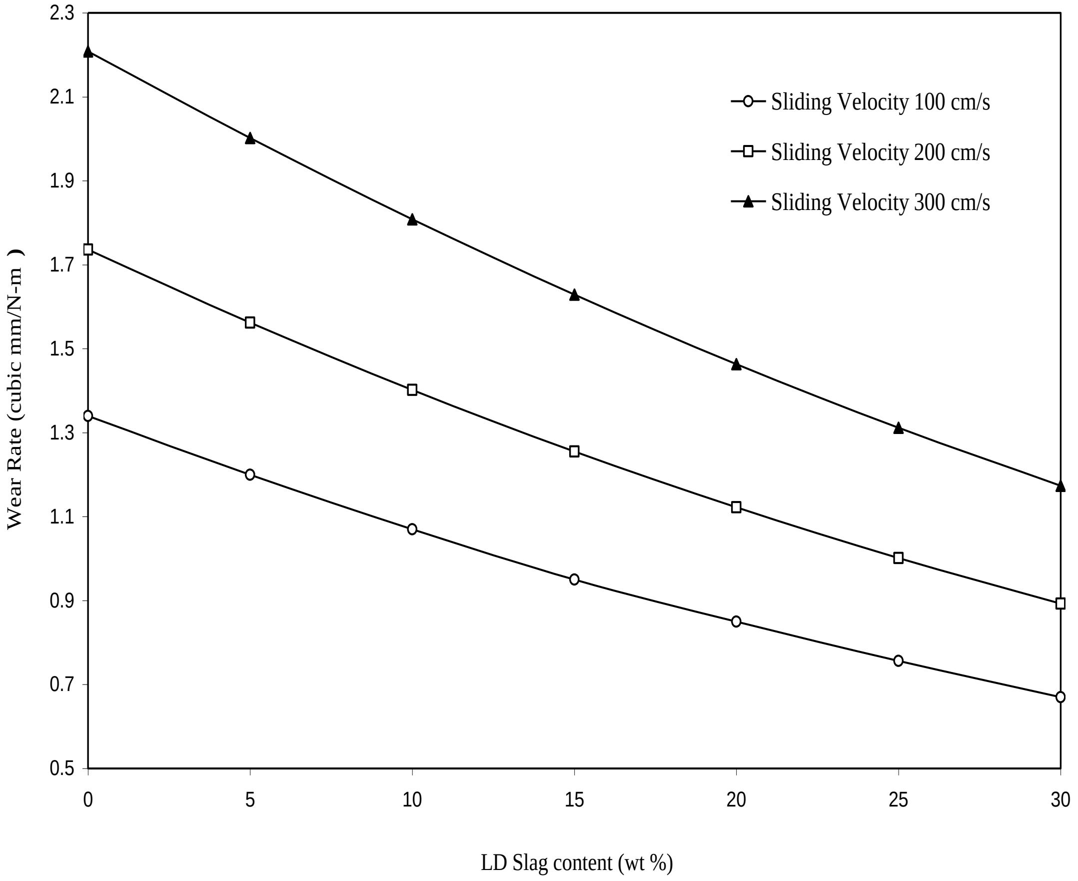


Figure 4 Variation of specific wear rate with LD slag content at different sliding velocities