A Study on Erosion Wear Behavior of Iron-Mud / Glass Fiber Reinforced Epoxy Composite

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Abstract. The current study investigates the erosion wear behavior of Iron-Mud / Glass Fiber Reinforced Epoxy Composite. It highlights the possible utilization of iron mine wastes for developing a new class of wear resistant hybrid polymer composites. In the present research, the composites were fabricated through hand-layup process by reinforcing woven glass fibers in epoxy polymer filled with different weight proportions of iron-mud. It was found in general that wear resistance of the fabricated composites improved with the fillers addition. The experimental runs were performed according to the Box-Behnken design of experiment methodology and ANOVA tests were carried out to determine statistical significance and percentage contribution of the control factors. The response surface methodology (RSM) optimization revealed the minimum erosion value of 1.5049 mm³/kg at the optimal parametric combination of 20% iron mud content, 70 m/s erodent velocity, 12 g/min erodent discharge rate and 90° impinging angle. Further validation tests showed a close relationship with experimental and theoretical data.

1. Introduction
Polymer composites are slowly replacing traditional materials as viable alternative products in many engineering applications such as automobiles, sports goods, marine, space and aeronautical industry-related applications. They are widely used as a superior tribo-engineering material considering their numerous advantages like wear resistance, excellent strength to weight ratio and stiffness to weight ratio as compared to monolithic metal alloys. Despite these advantages, there is a downward trend in use because of its high cost and unstable properties for development of composites. To be economical, utilization of low cost and abundantly available fillers are a viable option. Inclusion of fillers has dual purpose: firstly, to have superior mechanical and tribological properties, and secondly, making the component economical. Therefore, Tailor-made materials can be manufactured accordingly for specific engineering requirements through appropriate selection of fillers, fibres, matrix and processing techniques.

One of the alternatives is the use of iron-mines wastes as filler materials. Overburden and topsoil aggregates (iron-mud) as a major solid waste generated during iron mining and ore processing (mainly laterite) has a severe storage and reclamation problems with adverse environmental effects. Few researchers have studied the utilization of overburden in construction, Linz-Donawitz slag and iron-ore-tailings as fillers in polymer composites [1 - 4]. The possibility of inclusion of iron-mud in fiber
reinforced polymer composites (FRPCs) that could provide a synergism in terms of enhanced performance like superior wear resistance has not been addressed so far.

Amongst the various wear mechanisms, such as, adhesion, abrasion, erosion, corrosion, or fatigue, industries encounter erosion wear for the failure of significant number of engineering components out of which some are made from FRPCs. Solid particle erosion (SPE) is the natural wear of the surface of the body as a result of the mechanical interaction between solid particulate matter and surface, e.g., erosion during pipes carrying airborne solids, fly ash affected boiler tubes and gas turbine blades [5-9]. To decrease or control such erosion wear, appropriate selection of materials during design and complete knowledge of effect variables on the decay rate has become highly essential. In order to reduce the replacement-cost and material wastage of these worn spares, attention need to being given to investigate the tribological properties. A review of literature emphasized the influence of SPE. Few researchers indicated fillers like alumina, red mud, SiC, granite, fly ash, copper slag, LD-slag integrated into the polymer matrix, reduces the rate of material loss significantly during erosion [10-19]. Miyazaki and Takeda identified the influence of fiber weight fraction, fiber direction, filler weight fraction on the SPE characteristics [20]. Tilly and Sage studied the effect of weight, velocity, particle size, and impact angle of erodents on carbon-fiber-reinforced nylon, nylon, polypropylene, epoxy polymer and GF-reinforced plastic [21]. Barkoula, Karger-Kocsis and Patnaik et al. studied the effect of erodent velocity, particle size, and impact angle of impinging abrasives during experimental conditions on erosion resistance of polymer matrix composites [22-24]. However, a recent review by More et.al. suggests investigation of SPE on polymer composite is not available to the same degree as for ceramics or metals till date [25].

![Figure 1](image)

**Figure 1.** (a) Schematics of air jet-type erosion testing machine; (b) Eroded specimen after air erosion

Present work is an attempt to find a possible use of this abundant overburden (mines waste), which might gainfully be employed as a particulate filler in polymers for developing erosion wear resistant composites. It reports on developing a new class of hybrid polymer composites and investigates its tribological performance. The erosion wear experiments are conducted as per the Box-Behnken design approach under controlled laboratory conditions. The quadratic model was developed for enhanced analysis and prediction of wear behavior and to assess the damage due to wear for the minimum
erosion value at the optimal parametric combination. Further, ANOVA was performed to investigate the most significant control factors and their interactions.

2. Materials and Methods

2.1. Materials

Present fabricated composite used Epoxy-56L (Chemical Name-Bisphenol-A-Diglycidyl-Ether) as raw material for matrix material which can be cured at room temperature. Epoxy is used because of its high rigidity and superior wear and thermal properties, satisfactory corrosion resistance to alkali and acid and less volumetric shrinkage during curing exhibiting excellent dimensional stability in the electronic and coating industries [26 - 29].

Table 1. Typical attributes of materials.

| Property               | Glass fiber | Epoxy | Iron-mud |
|------------------------|-------------|-------|----------|
| Elastic modulus (GPa)  | 72.5        | 3.42  |          |
| Density (g/cc)         | 2.59        | 1.19  | 2.8      |

Plain weave E-glass fibers were used as reinforcement material. Iron-mud particles (collected from OCL India Ltd., Rajgangpur, Odisha, India) with a size range of 75-150 µm dried completely in sunshine and an oven at 100 °C were used as the filler material. Table 1 and Table 2 shows the typical attributes of materials used and chemical composition of iron-mud respectively.

Table 2. Composition of Iron-mud.

| Constituent             | Percentage |
|-------------------------|------------|
| Fe₂O₃                   | 71.35%     |
| Al₂O₃                   | 9.11%      |
| SiO₂                    | 8.39%      |
| CaO                     | 1.87%      |
| Traces of MgO and TiO₂  | 0.03%      |
| Loss on ignition        | 9.25%      |

2.2. Fabrication of Composite

Epoxy resin (56L) and its hardener (MH91) from Marshal Polymer Ltd were manually mixed by a mechanical stirrer to enhance the dispersion of the filler particulates in the polymer matrix at a 10:1 ratio as per the weight recommendation. Composites with different weight percentages (0, 5, 10, 15, and 20) of iron-mud content, with 50% weight fraction of GF fabricated by conventional hand lay-up process under a light compression mold (stainless steel of 250 mm × 250 mm × 3.5 mm dimensions, load applied 25 kg) for proper curing. The fabricated composites were further cured for 2 hours in a hot air oven at 100 °C. To ease the removal of the composite from the mold, silicon spray was used as the releasing agent. Specimens of suitable dimensions were prepared as per the ASTM standard using a diamond cutter for further investigations.

3. Results and Discussion

3.1. Box-Behnken Design of Experiment

Statistical software Minitab 18 was used for the analysis of design matrix. For modelling and optimization of input parameters (composites based upon iron mud content (A), erodent velocity (B), erodent discharge rate (C), impinging angle (D)) and its output response erosion value (E_v) were performed by using Box-Behnken design of experiment (DOE). Three levels of factor has been considered as outlined in table 3.
Table 3. Factor levels Box-Behnken design of experiment.

| Independent variables | Symbols | Units | Coded and actual levels |
|-----------------------|---------|-------|-------------------------|
| Iron-Mud content      | A       | %     | Low (-1) Mean (0) High (1) |
| Erodent Velocity      | B       | m/s   | 70 110 150               |
| Erodent discharge rate| C       | gm/min| 4 8 12                   |
| Impinging angle       | D       | degree| 30 60 90                 |

3.2. Air-Erosion Test

Wear test was carried out by using DUCOM TR-470 solid particle erosion tester (Refer Figure 1a) as per ASTM G76 standard. Composite laminates specimen dimensions of 25 mm × 25 mm × 3.5 mm (Length × Width × Thickness) were used (Refer Figure 1b). Nozzle (Tungsten Carbide, 1.5 x 50 mm long), erodent (Aluminum Oxide (Al₂O₃) 50 μm), stand-off distance (10 mm), test duration (5 min) used were kept same throughout all the tests. As per Box-Behnken DOE, 29 numbers of randomly chosen experiments had been performed to minimize the error of the experimental process which includes five centre points as reported in Table 4. Erosion value was calculated from the data of loss in volume per kg of erodent.

Table 4. Box-Behnken experimental design and results.

| Experiment | A (%) | B(m/s) | C(gm/min) | D(degree) | E,(mm³/kg) |
|------------|-------|--------|------------|------------|------------|
| 1          | 10    | 110    | 8          | 60         | 86.1357    |
| 2          | 10    | 110    | 12         | 90         | 19.7863    |
| 3          | 0     | 150    | 8          | 60         | 217.264    |
| 4          | 20    | 110    | 12         | 60         | 81.2222    |
| 5          | 0     | 70     | 8          | 60         | 11.6336    |
| 6          | 10    | 70     | 4          | 60         | 37.6886    |
| 7          | 0     | 110    | 12         | 60         | 69.9189    |
| 8          | 10    | 110    | 4          | 30         | 88.8778    |
| 9          | 10    | 150    | 12         | 60         | 199.468    |
| 10         | 10    | 70     | 8          | 90         | 11.4354    |
| 11         | 20    | 150    | 8          | 60         | 248.349    |
| 12         | 20    | 110    | 8          | 90         | 59.9535    |
| 13         | 0     | 110    | 8          | 90         | 51.1376    |
| 14         | 20    | 110    | 8          | 30         | 89.9441    |
| 15         | 10    | 70     | 12         | 60         | 6.3481     |
| 16         | 10    | 150    | 8          | 30         | 219.824    |
| 17         | 10    | 150    | 4          | 60         | 249.187    |
| 18         | 10    | 110    | 12         | 30         | 81.2011    |
| 19         | 0     | 110    | 4          | 60         | 98.3804    |
| 20         | 20    | 70     | 8          | 60         | 19.7787    |
| 21         | 10    | 70     | 8          | 30         | 23.6079    |
| 22         | 10    | 110    | 8          | 60         | 85.7198    |
| 23         | 0     | 110    | 8          | 30         | 76.4794    |
| 24         | 10    | 110    | 8          | 60         | 71.6257    |
| 25         | 10    | 110    | 4          | 90         | 59.0588    |
| 26         | 10    | 150    | 8          | 90         | 131.484    |
| 27         | 10    | 110    | 8          | 60         | 76.2843    |
| 28         | 20    | 110    | 4          | 60         | 93.5478    |
| 29         | 10    | 110    | 8          | 60         | 71.8755    |
3.3. ANOVA and its Statistical Analysis

The Table 5 shows the value of degree of freedom (DF), adjusted sum of squares (AdjSS), adjusted sum of mean squares (AdjMS), Fishers value (F) and probability value (P) for erosion value. It was found that R-square (98.43) is close to 100% and is in close relationship with the observational data. The predictability of model is good which was concluded from the values of "Pred R-Squared" and "Adj R-Squared". As the difference between "Pred R-Squared" (0.9171) and "Adj R-Squared" (0.9686) is less than 0.2, they are in reasonable agreement.

In the quadratic model (Refer equation (1)), a negative coefficient refers to antagonistic effect. Among all the significant variables, iron-mud content (A) contributed significantly as compared to other factors towards increasing the wear resistant property of the composites.

\[ E_{vE}^{0.5} = -1.50 - 0.115A + 0.0247B - 0.252C + 0.1845D + 0.00341A^2 + 0.000449B^2 - 0.0012C^2 - 0.001114D^2 + 0.000329A^2B + 0.00561A^2C - 0.000389B^2A - 0.00588C^2D \]  

(1)

| Source            | DF | Adj SS   | Adj MS   | F-Value | P-Value |
|-------------------|----|----------|----------|---------|---------|
| Model             | 14 | 360.201  | 25.729   | 62.68   | 0.000   |
| Linear            | 4  | 343.001  | 85.750   | 208.92  | 0.000   |
| A                 | 1  | 0.879    | 0.879    | 2.14    | 0.166   |
| B                 | 1  | 314.330  | 314.330  | 765.81  | 0.000   |
| C                 | 1  | 10.363   | 10.363   | 25.25   | 0.000   |
| D                 | 1  | 17.429   | 17.429   | 42.46   | 0.000   |
| Square            | 4  | 13.106   | 3.277    | 7.98    | 0.001   |
| A*A               | 1  | 0.752    | 0.752    | 1.83    | 0.197   |
| B*B               | 1  | 3.354    | 3.354    | 8.17    | 0.013   |
| C*C               | 1  | 0.003    | 0.003    | 0.01    | 0.938   |
| D*D               | 1  | 6.519    | 6.519    | 15.88   | 0.001   |
| 2-Way Interaction | 6  | 4.094    | 0.682    | 1.66    | 0.203   |
| A*B               | 1  | 0.069    | 0.069    | 0.17    | 0.688   |
| A*C               | 1  | 0.201    | 0.201    | 0.49    | 0.495   |
| A*D               | 1  | 0.005    | 0.005    | 0.01    | 0.910   |
| B*C               | 1  | 0.958    | 0.958    | 2.33    | 0.149   |
| B*D               | 1  | 0.872    | 0.872    | 2.12    | 0.167   |
| C*D               | 1  | 1.989    | 1.989    | 4.85    | 0.045   |
| Error             | 14 | 5.746    | 0.410    |         |         |
| Lack-of-Fit       | 10 | 5.093    | 0.509    | 3.12    | 0.142   |
| Pure Error        | 4  | 0.654    | 0.163    |         |         |
| Total             | 28 | 365.948  |          |         |         |

Figure 2 (a) shows the normal probability plot of residuals for erosion value. Residuals being near to straight line indicates that the experimental data are reliable and normally distributed. The statistically significant model parameters are shown in Pareto chart (Refer Figure 2 (b)). The model terms B, C, D, D' and B' were highly significant with a probability of 95%. The Interaction CD was significant (p-values less than 0.05).

Figure 3 shows the interaction effects between the erodent discharge rate (C) and impinging angle (D), respectively, for erosion value due to erosion wear in the form of a 3-D response surface and the corresponding contour plots.
3.4. Optimization of developed model

The iron mud content, erodent velocity, erodent discharge rate, impinging angle are the factors upon which wear performance depends. In order to determine the erosion value of composites, the

Figure 2. (a) Normal probability plot of residuals for erosion rate; (b) Pareto Chart for Standardized effect for erosion rate as response, α=0.05; (c) Optimisation plot of erosion value.

Figure 3. Erosion value versus C and D (a) 3D surface plot; (b) Contour plot.
independent variables were analyzed with an aim of reducing wear rate. Figure 2 (c) represents the optimization plot for which erosion value can be minimized. The minimum erosion value 1.5049 mm$^3$/kg is possible when 20% iron mud content, 70 m/s erodent velocity, 12 g/min erodent discharge rate and 90° impinging angle with desirability of 1. The present study considers an arbitrary set of factor combination as shown in Table 6 and the erosion value was found to be close to the predicted value.

Table 6 Confirmation experiment response values.

| Model         | Process Parameters   | Response | Error |
|---------------|----------------------|----------|-------|
| Experimental  | Iron-mud content (A) | Erodent Velocity (B) | Erodent discharge rate (C) | Impingement angle (D) | Erosion value (E$_V$) | %     |
| Box-Behnken   | 15                   | 110      | 8     | 90    | 51.94056              | ---    |
|               |                      |          |       |       | 47.2092               | 9.2    |

4. Conclusions
Following major conclusions may be drawn from the present research:

- Iron-mud/glass fiber filled hybrid composite was successfully fabricated from the iron mine waste using hand layoff technique successfully with various iron mud % as filler materials.
- Implementation of Box-Behnken design of experiment based on the response surface methodology for the analysis of erosion wear characteristics was done successful. The steady-state erosion wears performance analysis reveals that the inclusion of iron mud has enhanced the erosion wear resistance of composite. The predicted optimum value for minimum erosion value is 1.5049 mm$^3$/kg for a composite sample having 20% iron mud content at 70 m/s erodent velocity, 12 g/min erodent discharge rate and 90° impinging angle.
- The error associated with the experimental results and the predictive model for erosion wear is within the range of 0-10% as presented in the confirmation tests. Conversely, by increasing the number of experimental runs, the error can be further reduced.
- The fabricated composites under the current study will find potential application for various components, e.g. structures used in desert conditions, false ceiling, low-cost housing, pipes carrying coals and dust, partition board, industrial fan, etc. Further, by using other potential fillers, the current investigation can be extended to a new range of hybrid composites.

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