

# Mechanical and Slurry Abrasive Wear Behaviour of E-Glass Fiber Reinforced Composite Materials

Vinod Kumar Gautam<sup>1</sup>, Saurabh Kumar Singh<sup>2</sup> & Anil Kumar<sup>3</sup>

<sup>1</sup>Department of Mechanical Engineering, Malaviya National Institute of Technology, Jaipur

<sup>2</sup>Department of Mechanical Engineering, Indian Institute of Technology, Delhi 110016

<sup>3</sup>Department of Mechanical Engineering, M.M.M.U.T. Gorakhpur 273010

**Abstract:** E-glass fiber reinforced vinylester composites are fabricated in three different (55, 60 and 65) wt. % of a vinylester resin matrix. The mechanical characterization of these composites is performed. The slurry abrasive wear behaviour of fabricated composites has been conducted under different operating conditions. Slurry abrasive wear characteristics of these fabricated composites are successfully analyzed using Taguchi's experimental design scheme. The results obtained from these experiments are also validated against existing microscopic models of Ratner-Lancaster and Wang. It is observed that quite good linear relationships is held between specific wear rate and reciprocal of ultimate strength and strain at tensile fracture of these composites which is an indicative that the experimental results are in fair agreement with these existing models. Out of all the composites fabricated it is found that tensile strength of E-glass fiber reinforced composites increases because of interface strength enhancement. The morphology of worn composite specimens has been examined by scanning electron microscopy to understand the wear mechanisms.

## 1. Introduction

Composites are materials constituting of two or more than two chemically distinct ingredients [5], having a distinct interface separating them on a micro-scale. There are one or more than one discontinuous phases, therefore, mixed in a continuous phase to form a composite. In most of the situations the discontinuous phase which is reinforcement, usually harder and stronger than the continuous phase. This continuous phase is termed as the matrix. The matrix material can be metallic, polymeric or ceramic. When the matrix material is a polymer composite, then the composite is called as polymer matrix composite (PMC) [4]. The fiber reinforced polymer (FRP) consist of fibers of high strength and modulus bonded to a matrix with separate interface between them. In this form, both matrix and fibers retain their chemical and physical properties. Generally, fibers are the principal load bearing elements/members while the matrix places them at the desired location and orientation, and acts

as a load transfer medium between them, and also protects them from the degradation due to the harsh environmental. In past years, because of fairly good strength, low density, and high performance/cost ratios with rapid clean processing, tremendous growth in the developments and applications of fiber reinforced thermo-setting polymer composites such as epoxy, polyester and vinyl ester have [2] been observed. Polymer and their composites are widely used in various industrial applications such as ship panel, bearing material, seals, gears, cams, wheels, automotive bodies, clutches and transmission belts. Therefore, the mechanical and Tribological behaviour of these composite materials should be studied systematically. In the conditions where the wear performance in non-lubricated environments is a key parameter for material selection, polymer composites are widely used in mechanical components and also these components are used in various types of structure, where wear is the dominant factor for consideration. Among different types of wear, slurry abrasive wear situation [3] occurs in numerous equipment such as vanes and marine equipment. Literature shows that many researchers used Carbon fiber, glass fiber, aramide and graphite fiber are most common fibers used in polymer matrix composite. Literature shows that short fiber reinforcement polymer matrix composite led to the deterioration in the abrasive wear resistance, while the other reinforcement improved the abrasion resistance of the polymer matrix composites. Three body abrasive wear behaviour of polymer matrix composites have been reported by lots of researchers.

## 2. Experimental details

### 2.1. Panel preparation

E-glass fibers are reinforced with vinyl ester resin to prepare the fiber reinforced composites A1, B1, B2 and B3. The composition and designation of the composites prepared for this study are listed in Table 1. The vinyl ester resin mixed with E-glass fiber in three different percentages (55 wt. %, 60 wt. % and 65 wt. %). The marble dust is used as filler material (5wt%, 10 wt.% and 15 wt. %) in this study.

Composite slabs are prepared using conventional hand-layup technique. The E-glass fibers are mixed thoroughly in the vinyl ester resin. The low temperature curing vinyl ester resin and corresponding hardener are mixed in a ratio of 10:1 by weight as recommended by the supplier. The vinyl ester resin and the hardener are supplied by Sankar dies & chemicals, New Delhi, India. The E-glass fiber and the vinyl ester resin possess Young's modulus of 72.5 GPa and 3.5 GPa respectively and a density of 2.54 g/cm<sup>3</sup> and 1.23 g/cm<sup>3</sup> respectively. Each ply of fiber is of dimension 300 \* 300 mm<sup>2</sup>. A wooden mold having dimensions of 310 \* 310 \* 40 mm<sup>3</sup> is used. A silicon mold releasing spray is used to facilitate easy removal of composites from the mold after curing. The casting of each composite is cured under a load of about 40 kg for 24 hours before it is removed from the wooden mold. After this the cast is post cured in the air for another 24 hours after removing out of the wooden mold. Sample of suitable dimensions are cut using a diamond cutter for physical/mechanical characterization and slurry abrasive wear testing. Utmost care has been taken to maintain uniformity and homogeneity of the fabricated polymer composites.

Table 1. Designation and detailed composition of the composites

| Designations | Compositions  |
|--------------|---|
| A1           | E-glass fiber (30 wt. %) + Vinyl ester (70 wt.%)                          |
| B1           | E-glass fiber (30 wt. %) + Vinyl ester (65 wt.%) + marble dust (5 wt.%)   |
| B2           | E-glass fiber (30 wt. %) + Vinyl ester (60 wt. %) + marble dust (10 wt.%) |
| B3           | E-glass fiber (30 wt. %) + Vinyl ester (55 wt.%) + marble dust (15 wt.%)  |

**2.2. Slurry abrasive wear test**

To evaluate the performance of polymer composites under three body slurry abrasion condition, wear tests are carried out as per ASTM G-105 & B-611 using the slurry abrasion test rig supplied by MAGNUM ENGINEERS Ltd. The rubber wheel (Diameter 178mm \* 12.7mm, Hardness A-50). Wet sand/rubber wheel abrasion test involves the abrading of standard test specimen with grit of control size & composition. The abrasive (slurry mixture) comes in contact between the test specimen & a rotating rubber rim of shore hardness (A 50). The test specimen is pressed against the rotating rubber wheel at a specified force by means of a lever arm while a controlled & continuous flow of grit abrades the surface of the specimen to be tested. The test duration & force applied by the lever arm is varied. The specimens are weighed before and after the test to calculate the weight loss during testing.

Due to a wide difference in material density, slurry abrasion is reported on volume loss basis as.

$$W = \frac{\Delta M}{\rho} * S * N \dots\dots\dots (1)$$

Where, ΔM is the mass loss during the testing in grams (gm), ρ is the density of the composite (gm/cm<sup>3</sup>), S is the sliding distance (m) and N is the normal load (Newton). The specific wear rate is defined as the volume loss of the specimen per unit sliding distance(S) per unit applied normal load (N).

**2.3. Mechanical characterization**

The density of composite materials was determined by a buoyancy test (Archimedes Principles) according to (ASTM- 3800). Several samples were taken from each material type. The dry weight of each sample was measured on an electronic balance. Then the samples were weighed, and then submerged in water. If one knows the density of the matrix, filler, water, the density of composite can be calculated with the following equations:

$$\rho_c = \frac{W_{air}}{(W_{air} - W_{water})} * \rho_{water} \dots\dots\dots (2)$$

where,  
W<sub>air</sub> is the weight of the composites in the air (gm)  
W<sub>water</sub> is the weight of the composites in the water (gm)

ρ<sub>water</sub> is the density of the water  
ρ<sub>c</sub> is the density of the composites(gm/cc)

Volume fraction of void is calculated based on the theoretical and experimental density by using the following equation:

$$V_f = \frac{\rho_t - \rho_e}{\rho_t} * 100 \dots\dots\dots (3)$$

Micro-hardness of the sample is calculated using a Rockwell-hardness tester equipped with a steel ball indenter (1/1600) indenter by applying a load of 50 Kgf. The tensile test is performed on flat specimens on Digital Tensometer machine. The flexural and impact strength test is also conducted for the study the mechanical behaviour of composite. The low velocity instrumented impact tests are carried out on composite specimens. The tests are done as per ASTM D 256 using an impact tester. At the last, the wear surfaces of the composite due to the slurry abrasion testing are examined by scanning electron microscope Nova Nano FE-SEM 450 (FEI) which provides ultra-high resolution characterization & analysis giving precise, true nano-meter scale informations.

**2.4. Experimental design**

The Taguchi method is a commonly used technique for optimizing selected design parameters. Taguchi method provides accurate, systematic and efficient result for experimentation to determine

optimum settings of design parameters for better performance, optimum quality at optimized cost [26–29]. Since our aim is to obtain the best possible results with considering all level of parameter, we optimize the number of experiment to minimize the cost and also time during various testing, which is also an important requirements Exhaustive literature review reveals that parameters viz., wheel speed, normal load, filler contents and abrasive size largely influence the slurry abrasive wear characteristics of fabricated polymer composites. Thus, the impact of four parameters are studied using  $L_{16}$  orthogonal design [30]. The parameter settings and control factors for the slurry abrasive wear test (given in Tables 2 and 3) present the selected levels for various control factors.

Table 2. Parameter setting for wear test

|                 |          |
|-----------------|----------|
| Control factors | Symbol   |
| Wheel speed     | Factor A |
| Normal Load     | Factor B |
| Erodent Size    | Factor C |
| Filler contents | Factor D |

**Table 3**  
Levels of various control factors

| Control Factors    | Levels |     |     |     | Units         |
|--------------------|--------|-----|-----|-----|---------------|
|                    | I      | II  | III | IV  |               |
| A: Wheel speed     | 100    | 150 | 200 | 250 | RPM           |
| B: Normal Load     | 5      | 10  | 15  | 20  | N             |
| C: Erodent Size    | 106    | 150 | 212 | 300 | $\mu\text{m}$ |
| D: Filler contents | 0      | 5   | 10  | 15  | Wt.%          |

The plan of the experiments is as follows: the first column is assigned to wheel speed (A), the second column to Normal load (B), third column to Erodent size (C) and the fourth column is assigned to Filler contents (D) respectively and the remaining columns are used to estimate experimental errors. In practice, these factors can be assigned arbitrarily to any of the arrays columns, provided that all combinations are included. After assigning appropriate level settings, S/N: signal-to-noise ratio is needed to evaluate experiment results. In S/N analysis, the smaller the S/N ratio, better experimental results.

$$\eta = -10 \log (\text{M.S.D}) \text{ ----- (4)}$$

Where,  
M.S.D. is the mean square deviation for the slurry abrasive wear rate.

There are three categories of quality characteristics, i.e. smaller-the-better, higher-the-better, and nominal-the-better. For optimal performance, smaller-the-better characteristic for slurry abrasive wear rate must be taken. The mean-square deviation (M.S.D.) for smaller-the-better characteristic can be expressed as [30]

$$\frac{S}{N} = -10 \log \frac{1}{n} (y^2) \text{ ----- (5)}$$

Where n is the number of observation and y is the observed data.

ANOVA is performed to find out the significant process parameters for the experiment. With the help of S/N ratio and ANOVA analysis, we will find the optimum combination of process parameters. At the end, a confirmation experiment is conducted to verify the optimal process parameters obtained from the design parameter.

### 3. Results and discussion

#### 3.1. Mechanical Properties

In the present research work, the theoretical and measured densities of Bidirectional E-glass fiber–epoxy and chopped E-glass reinforced vinylester composites, along with the corresponding volume fraction of voids are presented in Table 4. It is found that the composite density calculated theoretically from weight fractions using the equation-2 are not equal to the experimentally density values, as expected. It is evident from Table 4 that the density of E-glass fiber reinforced vinylester composites increase with the increasing vinylester wt.%.

Table 4. Composite designations and their experimental and theoretical densities.

| Composite Designations | Experimental density ( $\text{g/cm}^3$ ) | Theoretical Density ( $\text{g/cm}^3$ ) | Volume fraction of voids (%) |
|------------------------|--|---|------------------------------|
| A1                     | 2.12                                     | 2.23                                    | 4.93                         |
| B1                     | 2.23                                     | 2.35                                    | 5.11                         |
| B2                     | 2.32                                     | 2.48                                    | 6.45                         |
| B3                     | 2.45                                     | 2.62                                    | 6.49                         |

#### 3.2. Micro-hardness

The measured micro-hardness values of all the four composites are presented in Fig.3.1. It is observed that marble dust addition shows a rapid change in the hardness value of the composites. The composite B2 and B<sub>3</sub> with marble dust content is found to exhibit some improvement in hardness compared to the unfilled composite A1. The mean hardness of marble dust is generally high like any other oxide ceramic and so with their inclusion, the composite hardness is increases. The low or marginal effect of these particulate fillers on composite micro-

hardness may be due to the presence of pores and voids. Here the hardness is seen to have improved with increase in marble dust content.

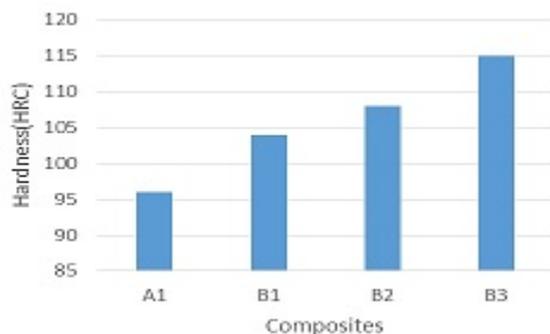


Figure 3.1 Variation of hardness of the different composites

### 3.3. Tensile and flexural strength

These variations of composites A1, B1, B2 and B3 in tensile and flexural strengths are shown in Figure 3.2 Gradual increase in both the tensile and flexural strength is noticed. Similar observations have been already made for fiber reinforced thermoplastic composites. However, it may be mentioned that both these strength properties of the composites are important for marine industry as well as for other structural applications.

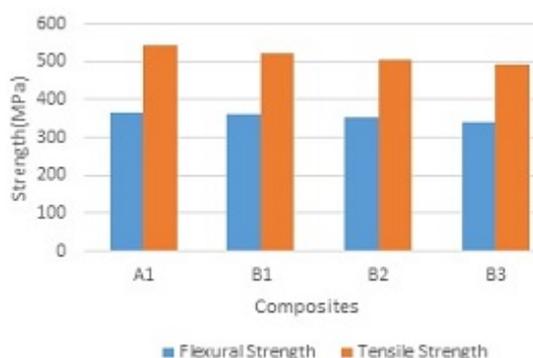


Fig.3.2 Comparison of Flexural and Tensile Strength of composites

### 3.4. Thermo-gravimetric analysis (TGA)

Thermal analysis is an important technique in the characterization of polymeric materials. During the fabrication of new products from polymers, knowledge of the thermal stability of their components is essential. Thermo gravimetric data provide the different stages of thermal breakdown, weight of the materials in each stage. The degradation studies of materials are usually intended to provide a data base for industrial practices in order to ascertain the working conditions that could prevent the degradation of the materials. The TGA

plot depicted in Figure 4.3 provides the information about the thermal stability of fabricated composite under this study.

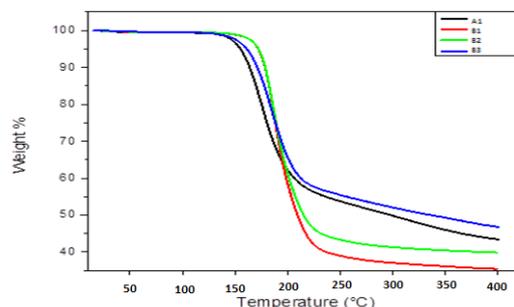


Fig.3.3. TGA behaviour of fabricated composites

### 3.4 Dynamic Mechanical Analysis (DMA)

The storage moduli ( $E'$ ) of the investigated composites are plotted in Figure 3.4. As the temperature increases by continuous heat supply, the  $E'$  magnitude is observed to drop steeply till 55 °C. Further there is a slight increment in storage modulus value after this temperature range. The modulus of 10 % filled composite is found to be highest in the transition regime while 15 % filled composite shows the lowest modulus. Glass transition regime is observed in the temperature range of 30 °C to 55 °C. Storage modulus values for glass fiber reinforced vinyl ester composites show a linear trend with slight decrease in the value of storage modulus with the change in temperature (room temperature to 30°C). This shows glassy regime in region 1, i.e., from room temperature to 30 °C. Region 2 shows a sharp decline in the values of storage modulus with the increase in the values of temperature (30°C to 55 °C), i.e., the material changes from glassy to rubbery transition regime in the temperature range of 30°C to 55°C; due to a sharp drop in the value of storage modulus, the material has lost its usefulness as a structural material.

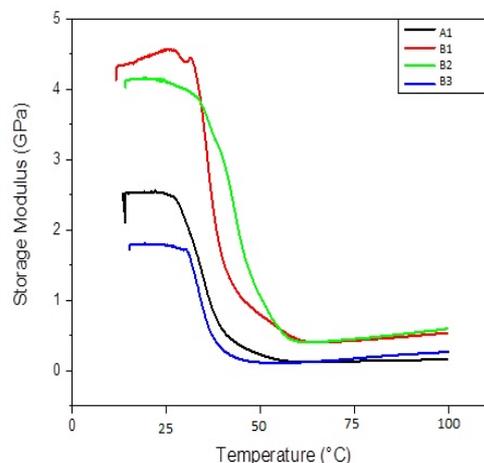


Fig.3.4 Storage modulus ( $E'$ ) Vs Temperature

### 3.5. Loss Modulus (E'') Vs Temperature

Figure 3.5 shows the value of loss modulus with the increase in the value of temperature. The peak of the loss modulus curve denotes the glass transition temperature (T<sub>g</sub>). Figure denotes that the glass transition temperature increases slightly up to 10 % of the filler content while 15 % composite samples show minimum value of (T<sub>g</sub>) equal to 34 °C. The value of T<sub>g</sub> is found to be maximum for the 10 % filler content and it is equal to 43 °C.

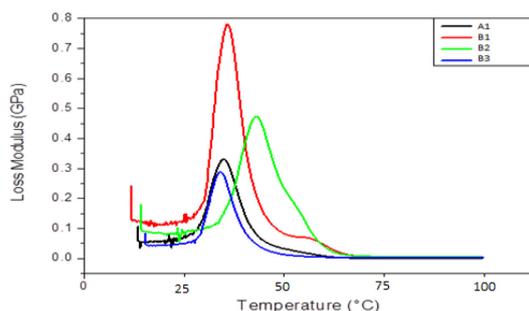


Fig. 3.5 Loss Modulus (E'') Vs Temperature

### 3.6. Slurry Abrasion Test Results and Taguchi analysis

A possible reason for the semi-ductile abrasion behaviour exhibited by the vinyl ester based polymer composites in the present investigation is that the glass fibers used as reinforcements for vinyl ester matrix are a typical brittle material. Their abrasion is caused mostly by damage mechanism such as micro-cracking. Such damage is supposed to increase with the increase of kinetic energy loss of the load and erodent size of the sand particles. In the present study, the peak abrasion rate shifts to a larger value of erodent size (150 μm) and it is clearly due to the brittle nature of glass fibers. So although vinyl ester composite is a ductile material, the presence of fibers makes the composite relatively more sensitive to impact energy. This explains the semi-ductile nature of the glass-vinyl ester composites with respect to solid particle abrasion.

#### Abrasion Rate and Taguchi Analysis using Marble dust as a Filler

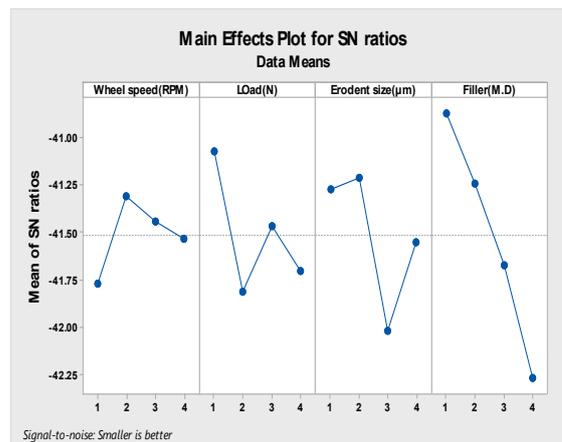


Figure 3.6. Effect of control factors Vs S/N ratio of glass vinyl ester & marble dust filled composites

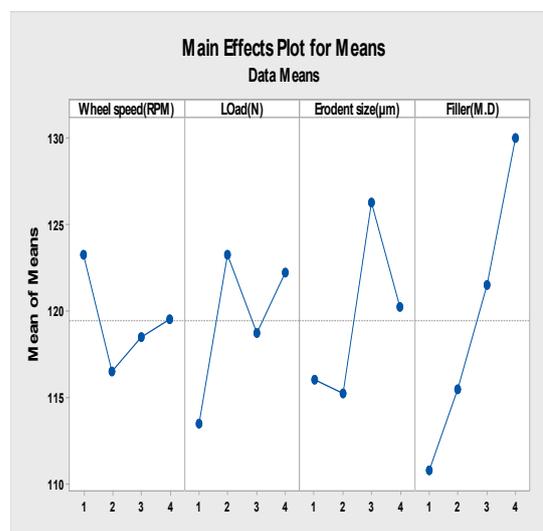


Fig. 3.7. Effect of control factor Vs Means of Means for marble dust filled composites

In the main effect of control factors on abrasion rate plot if the line for a particular factor is close to horizontal in nature, then the factor has no significant effect. On the other hand, a factor for which the line has the more inclination will have the most significant effect.

Table 5. Experimental design using L16 orthogonal array glass vinyl ester composites A1, B1, B2 and B3.

| Sl. No | Wheel Speed | Load(N) | Erodent Size (μm) | Abrasion Rate (gm/Kg) | S/N Ratio (db) |
|--------|-------------|---------|-------------------|-----------------------|----------------|
| 1      | 100         | 5       | 106               | 106                   | -40.5061       |
| 2      | 150         | 5       | 150               | 119                   | -41.5109       |
| 3      | 200         | 5       | 212               | 129                   | -42.2118       |

|    |     |    |     |     |          |
|----|-----|----|-----|-----|----------|
| 4  | 250 | 5  | 300 | 139 | -42.8603 |
| 5  | 100 | 10 | 150 | 110 | -40.8279 |
| 6  | 150 | 10 | 106 | 125 | -41.9382 |
| 7  | 200 | 10 | 300 | 108 | -40.6685 |
| 8  | 250 | 10 | 212 | 123 | -41.7981 |
| 9  | 100 | 15 | 212 | 130 | -42.2789 |
| 10 | 150 | 15 | 300 | 126 | -42.0074 |
| 11 | 200 | 15 | 106 | 112 | -40.9844 |
| 12 | 250 | 15 | 150 | 106 | -40.5061 |
| 13 | 100 | 20 | 300 | 108 | -40.6685 |
| 14 | 150 | 20 | 212 | 123 | -41.7981 |
| 15 | 200 | 20 | 150 | 126 | -42.0074 |
| 16 | 250 | 20 | 106 | 121 | -41.6557 |

From Table 5, the overall mean for the S/N ratio of the abrasion rate for marble dust filled composites is found to be -42.8603 db.

The analysis was made using the popular software specifically used for design of experiment applications known as MINITAB 17. Before any attempt is made to use this simple model as a predictor for the measures of performance, the possible interactions between the control factors must be considered [23]. Thus factorial design incorporates a simple means of testing for the presence of the interaction effects.

Table 6. RESPONSE TABLE FOR SIGNAL TO NOISE RATIOS (smaller is better)

| Level | Load (N) A | Wheel Speed (Rpm) | Erodent size (µm) | Filler (M.D) D |
|-------|------------|-------------------|-------------------|----------------|
| 1     | -41.77     | -41.07            | -41.27            | -40.87         |
| 2     | -41.31     | -41.81            | -41.21            | -41.24         |
| 3     | -41.44     | -41.47            | -42.02            | -41.68         |
| 4     | -41.53     | -41.71            | -41.55            | -42.27         |
| Delta | 0.46       | 0.74              | 0.81              | 1.40           |
| Rank  | 4          | 3                 | 2                 | 1              |

Table 7. Results of the confirmation for abrasion wear rate for composites A1, B1, B2 and B3

| Level     | Optimal control parameters                                  |   | Error (%) |
|-----------|---|---|-----------|
|           | Prediction  | Experimental  |           |
| S/N Ratio | D <sub>1</sub> C <sub>2</sub> B <sub>3</sub> A <sub>4</sub> | D <sub>1</sub> C <sub>2</sub> B <sub>3</sub> A <sub>4</sub> | 2.32      |

### 3.7. ANOVA and the effect of Factors

In order to find out statistical significance of various factors like wheel speed (A), Load (B), erodent size (C) and filler contents (D) on abrasion rate, analysis of variance (ANOVA) is performed on experimental data. Table 8. show the results of the ANOVA with the abrasion rate of vinyl ester reinforced glass fiber filled with marble dust powder polymer composites taken in this investigation. The last column of the table indicates percentage contribution of the control factors and their interactions on the performance output. From Table 8, it can be observed for the Marble dust filled Glass-vinylester composites that filler content (p=0.014), erodent size (p = 0.057) and load (p=0.080) have considerable influence on abrasion rate. It means the filler contents (p=0.014) is the most significant factor and the wheel speed (p=0.222) has the negligible influence on performance output.

Table 8. ANOVA table for abrasion rate (for Marble dust filled composites)

| Source            | DF | Adj SS  | Adj MS | F-Value | P-Value |
|-------------------|----|---------|--------|---------|---------|
| Wheel speed (RPM) | 3  | 96.19   | 32.06  | 2.66    | .222    |
| Load(N)           | 3  | 232.69  | 77.56  | 6.43    | .080    |
| Erodent size (µm) | 3  | 305.69  | 101.90 | 8.45    | .057    |
| Filler (M.D)      | 3  | 827.19  | 275.73 | 22.86   | .014    |
| Error             | 3  | 36.19   | 12.06  |         |         |
| Total             | 15 | 1497.94 |        |         |         |

\*\*DF: degree of freedom, ##Seq SS: sequential sum of squares, §Adj. SS: extra sum of squares <sup>SS</sup> Seq MS: sequential mean squares, \*\*\*F: F-test, ###P: percent contribution.

The analytical and experimental investigation into the abrasion behaviour of glass fiber reinforced vinyl ester composites leading to the following major conclusions:

1. Solid particle abrasion characteristics of these composites can be successfully analysed using Taguchi experimental design scheme.

2. The results indicate that filler content, erodent size and load are the significant factors affecting the abrasion wear rate.

3. The Result shows that Marble dust filled composite has lest p value over the composite without filler, which indicates that marble dust as a filler is more influencing factor.

The abrasion wear values obtained experimentally also suggest that the glass fiber reinforced vinyl ester composites exhibit semi-ductile abrasion for low wheel speed and load. However, for relatively high

wheel speed, they present a ductile abrasion response.

#### 4. Conclusion

By incorporating of these particulate fillers into the glass-fiber reinforced vinyl ester, synergistic effects, as expected were achieved in the form of modified mechanical properties and improved slurry abrasion wear resistance. Inclusion of glass fiber in vinyl ester without using filler, minor improved the load bearing capacity but improved the shear strength and the ability to withstand flexural strength of the glass vinyl ester composites. But with the incorporation of particulate fillers, the tensile strengths of the composites were found to be less. There can be two reasons for this decline in tensile strength of these particulate filled glass vinyl ester composites compared to the unfilled one. One possibility is that the chemical reaction at the interface between the filler particles and the matrix may be too weak to transfer the tensile stress. Hardness values have been found to have much improved in the marble dust filled composites. The reduction in tensile strength and the improvement in hardness with the incorporation of fillers under the action of a tensile force the filler-matrix interface is vulnerable to the bonding depending on interfacial bond strength and this may lead to a break in the composite. But in case of hardness test, a compression or pressing stress is in action. So the polymeric matrix phase and the filler phase would be pressed together and touch each other more tightly.

Sand particles impact on the fibres and cause fibres to break because of the formation of cracks perpendicular to their length. These cracks are presumably caused by fibre-bending stresses due to the impact of erodent particles on the unsupported fibers.

Further damage results when the interfaces between the broken fibers and the matrix resin are degraded until the fibers are removed by subsequent impacts.

#### 5. References

[1]. Yousif BF, Nirmal Umar, Wong KJ. Three-body abrasion on wear and frictional performance of treated betelnut fibre reinforced epoxy (T-BFRE) composite. *Mater Des* 2010;31:4514–21.

[2]. Chand N, Neogi S. Mechanism of material removal during three-body abrasion of FRP composite. *Tribol Lett* 1998;4:81–5.

[3]. Mishra P, Acharya SK. Anisotropy abrasive wear behavior of bagasse fiber reinforced polymer composite. *Int J Eng. Sci Technol* 2010;2(11):104–12.

[4]. Stokes VK. Random glass mat reinforced thermoplastic composites Part II. Characterization of the tensile strength. *Polym Compos* 1990;11(6):354–67.

[5]. American society for testing and materials (ASTM). In: Standard test method for apparent

interlaminar shear strength of parallel fiber composites by short beam method, ASTM D 2344-84. West Conshohocken (PA): Annual book of ASTM standards, ASTM; 1984. p. 15–7.

[6]. Rajesh JJ, Bijwe J, Tewari US. Friction and wear studies of short glass fiber reinforced polyetherimide composites. *Wear* 2002;252:769–76.