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## Optimization of dry sliding wear behavior of epoxy nanocomposites under different conditions

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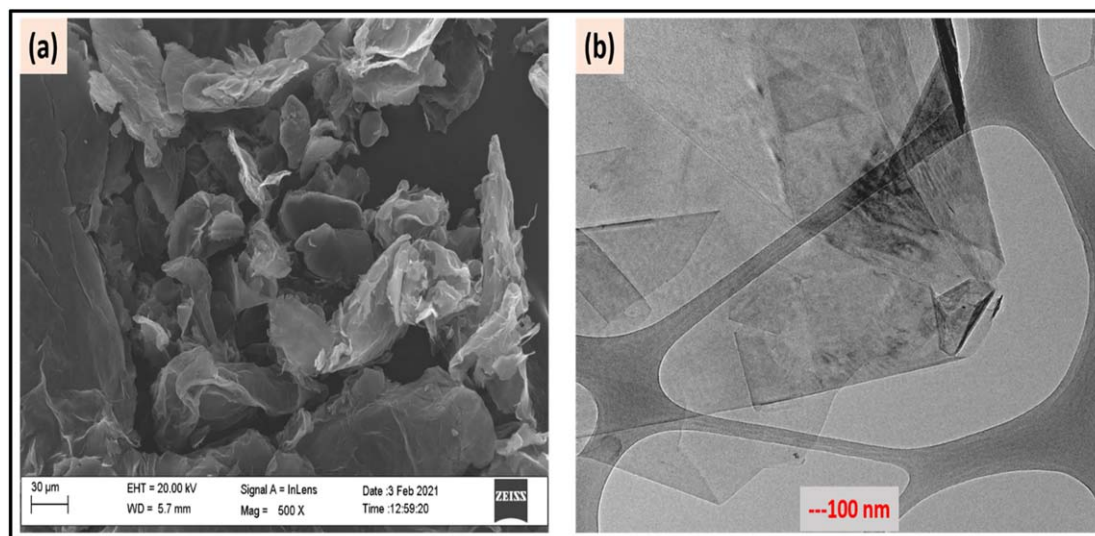
**Keywords:** nanocomposite, graphene nanoplatelets, sliding wear, epoxy, hand layup

### Abstract

This paper evaluates the wear properties of epoxy nanocomposites containing GNP (Graphene Nanoplatelets). In this research, variation of GNP (0, 0.1, 0.25, and 0.5 wt%) were used to make nanocomposites. The hand layup technique was used for the fabrication of different composites. The Taguchi method is used to optimize the wear test and its related characteristics. Analysis of variance is used to understand the correlation between input variables and response measurements. Load (0.5, 1.0, 1.5, and 2.0 kg) and time (4, 6, 8, and 10 min) are the main variables for exploring wear characteristics for this study. It has been discovered that minimum wear in terms of mass loss and friction coefficient are minimum at 0.5 wt% GNP. Scanning electron microscopy (SEM), energy dispersive x-ray analysis (EDX), and RAMAN spectroscopy were used to characterize the wear mechanism. The result shows that the optimized value is closer to the experimental value.

## 1. Introduction

Polymer nanocomposites (PNC) are regarded as one of the most promising groups of materials among today's advanced materials. Nano reinforced materials exhibit a great combination of properties with multiple design possibilities. As a result of their great potential and applicability in various sectors, these polymer composites are in high demand in both academia and industry [1–3]. Several PNCs have been devised and produced at the nanoscale in the recent two decades to develop new and enhanced electrical, mechanical, and wear properties. Apart from their broad applications, these materials now have applications in energy, electronic, and medical devices [4, 5]. Epoxy resin has proven to be a significant engineering polymer in this context, having been employed in various applications like adhesives and structural elements in the aerospace industry [6]. Much work has been done to overcome the disadvantages and expand its applicability to other possible sectors. Carbon nanofillers such as graphite, carbon nanotubes (CNT), and carbon nanofibers that conduct electricity and heat could improve the mechanical, electrical, wear, and thermal properties of these materials [7, 8]. Many engineering applications needing minimal friction and wear require epoxy nanocomposite materials with outstanding tribological characteristics. One of the most promising ways to solve the epoxy matrix's significant dry wear problem is by adding a carbon nanofiller [9, 10]. Epoxy nanocomposites are widely employed in various sliding circumstances, including structural components and automotive and marine vessels [11–13]. Carbon-based filler and fiber-based composites are being explored as potential anticipated materials for various tribological applications due to their superior mechanical properties [14]. Adding GNP to the GFRP composites reduced wear volume loss from a tribological standpoint. Furthermore, a sufficient amount of GNP (1 wt%) considerably improved the tribo-mechanical performance of the composite materials [15]. The tribological properties of epoxy composites are explored using carbon nanotubes and graphene oxide. [16]. Carbon nanocage filler is used with Epoxy to make nanocomposite. At 0.25 wt% optimum wear properties have been



**Figure 1.** (a) SEM & (b) TEM image of GNP.

achieved, and the wear rate is reduced by 21.8%. [3]. The findings showed that one-dimensional Carbon is one of the most effective fillers for high-temperature Epoxy-based friction materials [17]. Silicon carbide (SiC) and graphite (Gr) are used to make epoxy composites. SiC/Gr containing epoxy composites show the lowest and highest wear resistance [18]. In this work, a combination of  $\text{Al}_2\text{O}_3$  (2 wt%) and GNP (0.5 wt%) in the Epoxy shows the least wear and friction coefficient compared to other varieties [19]. According to these investigations, functional fillers have a considerable impact on wear characteristics. Many various wear models determined wear as a function of filler, average load, and other factors [20–22]. The conventional and Taguchi approaches can both be used to look into the impact of specific factors on single response situation optimization. Multiple response optimization is also necessary in tribological circumstances. Each response's optimization is calculated individually to apply the technique described above to solve optimization issues involving multiple responses [23, 24]. Wang *et al* [25] studied the effect of  $\text{ZrO}_2$  nanoparticle sizes ranging from 10 to 100 nanometers. They find that smaller nano size particle has great impact on tribological behavior of polymer composites. Xing *et al* [26] used different size varied from 120 and 510 nm nano silica particles for comparison of wear properties of epoxy composites. They found the similar trend of result, that smaller size shows better impact on wear. Use of nano particles in place of micro gives better wear performance of composites. It is due to reduction in abrasiveness and angularity of nano particles compare to micro particles. Sometimes these nano particles act as a lubrication and polishing agent between mating surfaces. This is the responsible factor for reduction in friction and wear of composites. Smaller particles, in general, appear to contribute more to the enhancement of tribological characteristics than bigger particles. Sometimes nano filler used above certain limits may decrease the wear characteristics due to agglomeration and non-uniform dispersion of particles in composites [27].

Polymer composites containing different nanofiller have become famous in a variety of tribological applications. Carbon nanofiller has more advantages over inorganic nanofiller. It is due to the self-lubrication property, which gives low friction, high wear resistance, and good corrosion resistance [1, 4]. Polymer composites are used in various applications where observation of the tribo mechanism is essential. So, a better understanding of the tribology of polymer composites is equally important for the different tribological applications of these materials. That is the way to promote these materials in tribology field applications. This paper deals with GNP as a nanofiller to make epoxy nanocomposites for understanding the wear characteristics for different applications.

This work aims to determine the wear behavior of epoxy nanocomposites containing different weight percent of GNP. Analysis of variance (ANOVA) and Taguchi's method is used to optimize the result and determine the best parameters for various output such as mass loss (M) and coefficient of friction (COF). GNP reinforced epoxy nano composites could become more popular as a result of the current research. Taguchi's design' for studying wear qualities dramatically decreases the number of experiments. It provides a regression equation for predicting a mass loss. The results may increase the application of GNP-filled epoxy nanocomposites in tribology fields.

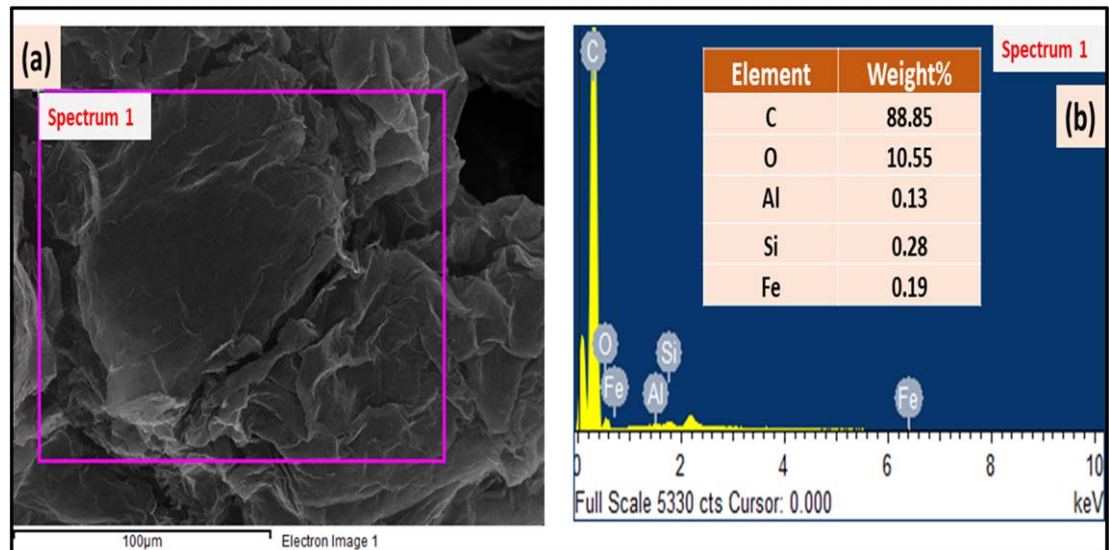


Figure 2. (a) EDX image & (b) Element mapping of GNP.

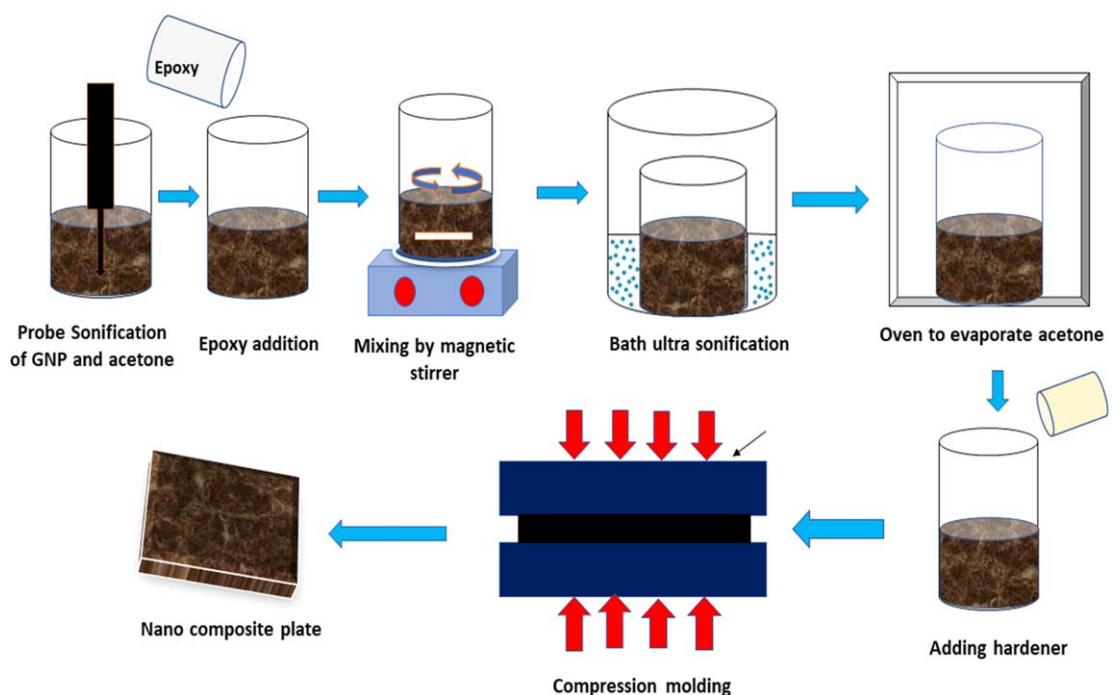


Figure 3. Diagram of nanocomposite fabrication.

## 2. Experiment

### 2.1. Materials and Method

Epoxy (LY-556) and hardener (HY-951) were purchased from Go Green, India. GNP was used as a nanofiller procured from Platonic nanotech, India. Epoxy was taken as per requirement in a glass beaker and put in the muffle furnace (75 °C) for 30 min. Figures 1(a) and (b) show GNP's SEM and TEM images. Figure 2 shows the EDX image and element present in GNP.

Preheating is required to remove moisture and increase the viscosity of Epoxy. GNP is also heated for one hour to remove moisture content. After heating, GNP is taken as per requirement and mixed with acetone using a probe sonicator for 20 min to get proper dispersion of nanofiller. Epoxy was poured into the acetone and filler solution. A magnetic stirrer and bath ultrasonicator are used for proper mixing and dispersion of nano particles in Epoxy. The stirrer operated at 600 rpm for 30 min at 70 °C. Temperature is provided to magnetic stirrer for

**Table 1.** Composites and their shore hardness.

| Materials             | Shore-D Hardness |
|-----------------------|------------------|
| Epoxy (E-0)           | 76               |
| 0.1% GNP/Epoxy (E-1)  | 78               |
| 0.25% GNP/Epoxy (E-2) | 80               |
| 0.5% GNP/Epoxy (E-3)  | 82               |

**Table 2.** Input variables.

| Variables             | Level 1 | Level 2 | Level 3 | Level 4 | Units |
|-----------------------|---------|---------|---------|---------|-------|
| Nano particle wt% (C) | 0       | 0.1     | 0.25    | 0.5     | %     |
| Load (P)              | 0.5     | 1.0     | 1.5     | 2.0     | kg    |
| Time (T)              | 4       | 6       | 8       | 10      | min   |

**Table 3.** L16 orthogonal array for experimental Design.

| Exp.no. | Nano particle wt% (C) | Load (P) (kg) | Time (T) (min) | Mass loss (M) (mg) | COF  |
|---------|-----------------------|---------------|----------------|--------------------|------|
|         | 0.00                  | 0.5           | 4              | 64                 | 0.52 |
|         | 0.00                  | 1.0           | 6              | 78                 | 0.54 |
|         | 0.00                  | 1.5           | 8              | 89                 | 0.56 |
|         | 0.00                  | 2.0           | 10             | 99                 | 0.57 |
|         | 0.10                  | 0.5           | 6              | 65                 | 0.48 |
|         | 0.10                  | 1.0           | 4              | 71                 | 0.46 |
|         | 0.10                  | 1.5           | 10             | 76                 | 0.49 |
|         | 0.10                  | 2.0           | 8              | 83                 | 0.51 |
|         | 0.25                  | 0.5           | 8              | 63                 | 0.43 |
|         | 0.25                  | 1.0           | 10             | 66                 | 0.45 |
|         | 0.25                  | 1.5           | 4              | 71                 | 0.46 |
|         | 0.25                  | 2.0           | 6              | 72                 | 0.47 |
|         | 0.50                  | 0.5           | 10             | 54                 | 0.39 |
|         | 0.50                  | 1.0           | 8              | 57                 | 0.41 |
|         | 0.50                  | 1.5           | 6              | 59                 | 0.42 |
|         | 0.50                  | 2.0           | 4              | 60                 | 0.43 |

complete vaporization of acetone from the mixture. In a bath ultrasonicator, ultrasonic waves are used for 30 min at 60 °C to get a homogeneous mixture of Epoxy and GNP. The mixture was cooled at room temperature. Hardener was taken in another beaker in the ratio of 10:1 w.r.t to Epoxy. Hardener was poured into the mixture at room temperature. Then the mixture was poured into the die. The upper mold is placed over the lower mold and then compressed using a compression molding machine for 24 h [28] shown in figure 3. Table 1 shows the shore hardness of composites.

## 2.2. Wear test using design of experiments (Taguchi method)

The tests were carried out using different parameters listed in table 2. The design of experiments is used to make an L16 array for given inputs shown in table 3 [20, 23, 24, 29–32]. According to that table, the experiments were conducted using the pin-on-disc machine. Specimens were cut from sheets according to ASTM G99 [24]. The size of the specimen is 30 mm × 10 mm. For an accurate reading, the test was carried out three times. The operating parameters were used for the wear test: variable load: 0.5, 1.0, 1.5, and 2 kg, times of 4, 6, 8, and 10 min were used with the constant sliding velocity of 300 rpm. Specimen weight is measured before and after the test using an electronic balance machine. The difference in weight is used to calculate the wear loss. A lower value suggests more excellent performance is referred to as ‘smaller is better.’ [23]. ANOVA is used to analyze the impact of input parameters on the evaluation of tribological characteristics such as mass loss and friction coefficient. Nova Nano SEM 450 (FEI) and RAMAN spectroscopy setup were used to understand the worn surface structure.

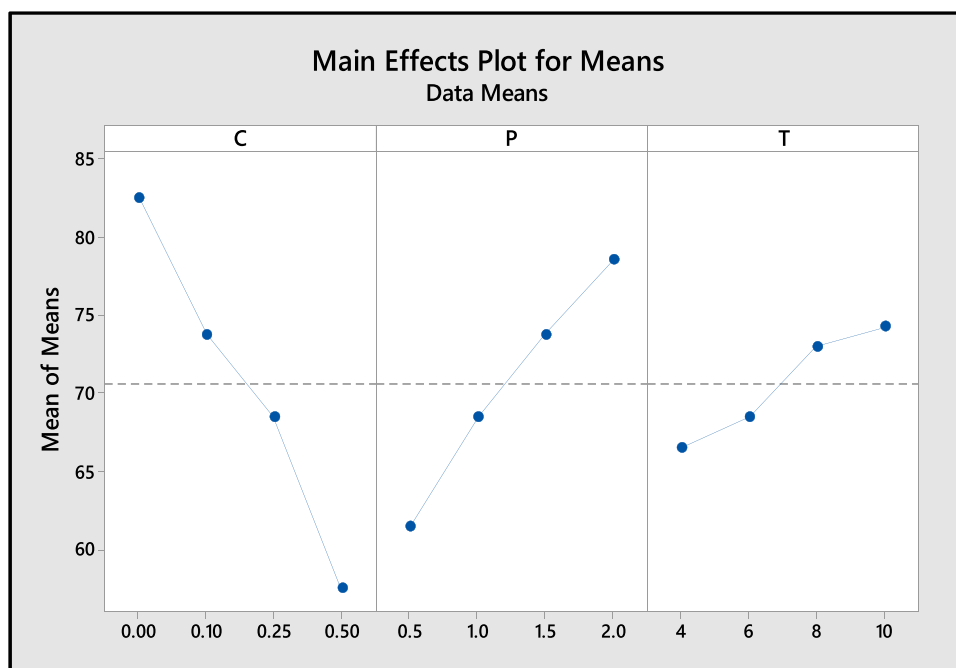


Figure 4. Main effect plot for mass loss.

Table 4. Analysis of variance table (M).

| Source  | DF | Seq SS  | Contribution | Adj SS  | Adj MS    | F-Value | P-Value    |
|---------|----|---------|--------------|---------|-----------|---------|------------|
| C       | 3  | 1680.53 | 74.03%       | 1680.53 | 560.178   | 142.09  | 0.000      |
| P       | 3  | 557.03  | 24.54%       | 577.03  | 185.677   | 47.10   | 0.007      |
| T       | 3  | 8.77    | 0.39%        | 8.77    | 2.924     | 0.74    | 0.565      |
| Error   | 6  | 23.66   | 1.04%        | 23.66   | 3.943     |         |            |
| Total   | 15 | 2269.99 | 100.00%      |         |           |         |            |
| S       |    |         | R-sq         |         | R-sq(adj) |         | R-sq(pred) |
| 1.98558 |    |         | 98.96%       |         | 97.39%    |         | 92.59%     |

Table 5. Analysis of variance table (COF).

| Source    | DF | Seq SS   | Contribution | Adj S.S. | Adj MS    | F-Value | P-Value    |
|-----------|----|----------|--------------|----------|-----------|---------|------------|
| C         | 3  | 0.039069 | 89.62%       | 0.039069 | 0.013023  | 178.60  | 0.000      |
| P         | 3  | 0.003819 | 8.76%        | 0.003819 | 0.001273  | 17.46   | 0.002      |
| T         | 3  | 0.000269 | 0.62%        | 0.000269 | 0.000090  | 1.23    | 0.378      |
| Error     | 6  | 0.000437 | 1.00%        | 0.000437 | 0.000073  |         |            |
| Total     | 15 | 0.043594 | 100.00%      |          |           |         |            |
| S         |    |          | R-sq         |          | R-sq(adj) |         | R-sq(pred) |
| 0.0085391 |    |          | 99.00%       |          | 97.49%    |         | 92.86%     |

### 3. Results and discussion

#### 3.1. Analysis of variance (ANOVA) analysis

In the Taguchi method, the set of input parameters has been achieved by considering an orthogonal array. This set of parameters gives the least possible experiment for optimization. ANOVA analysis provides information about the impact of input factors on the output results. ANOVA table shows the percentage contribution of all the input values to desirable outcomes. The impact and importance of different factors on the results are analyzed in this analysis [30]. Tables 4 and 5 show the percentage contribution of every factor which affects the wear characteristics. From table 4, it is evident that weight percent of GNP and load are the main factors that influence the wear characteristics of epoxy nanocomposites. P-values of these input elements are less than 0.05 in our case. It is informed that all the factors contribute to the M and COF. GNP (74.03%) and load (24.54%) influence M of wear tests more than the time factor. ANOVA shows that the nanofiller significantly affects M and

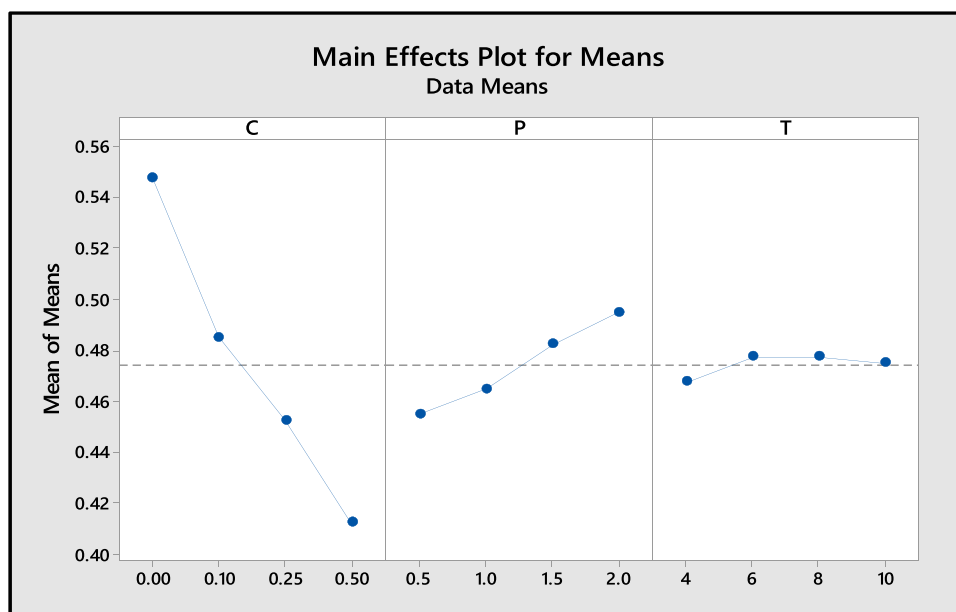


Figure 5. Main effect plots for COF.

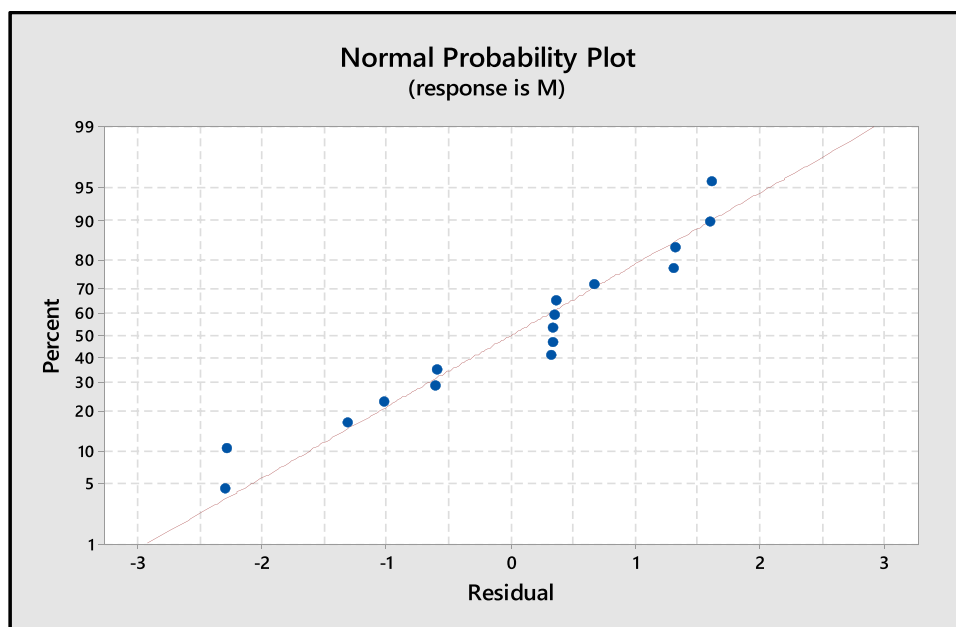


Figure 6. Probability plot for M.

COF [23, 30]. For COF, the least substantial factor is also time, with a p-value of 0.378. The nanoparticle weight variation contributes the most to mass loss, accounting for 74.03 percent of the total. At the same time, the other factors show less effect. A similar trend has been demonstrated for COF also (referred to table 5). Two characteristics, the percent of GNP and load, are significant at the 5% significance level, whereas the remaining components are insignificant. Obviously, for Epoxy nanocomposites, the effect of nanoparticles is more than that of others. So, it is confirmed that wear loss is reduced with increasing GNP percentage. The excellent bonding between matrix and filler significantly affected wear. It was observed that particularly at 0.5 percent GNP shows greater wear resistance than others. GNP has a lubrication nature which is responsible for the reduction in COF.

According to analysis and from the table, it is determined that the weight percentage of nanofiller impact a major effect on both outputs (M and COF). For mass loss and COF, nanofiller concentration gives 74.03% and 89.62% contribution respectively. The second parameter load also has a significant impact (24.34% and 8.76%)

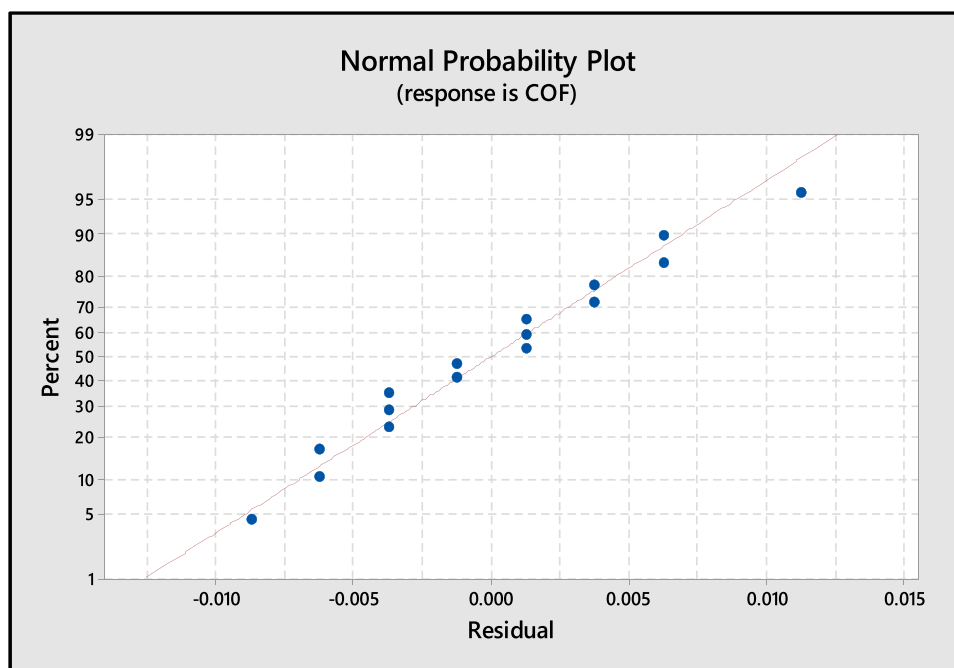


Figure 7. Probability plot for COF.

on both output results shown in the given tables. Time has a negligible effect on the output results. From the tables, it is observed that the greater the F value, the more influence of input parameters on M and COF. The value of P of all input factors is less than 0.05, which has more effect on the results. The value of the adjusted coefficient of determination ( $R\text{-Sq}(\text{adj})$ ) is 97.39% and 97.49%, respectively for both results. This information indicates that deviation in the obtained value of M and COF is the function of the input factors within the range in this research. So, ANOVA analysis describes the percentage contribution of all the input factors on the mass loss and COF.

### 3.2. Main effect and residual plots

Figures 4 and 5 illustrate the main effect plots, which show the effect of different input parameters on M and COF in wear tests. Parameters that have a greater slope present a greater influence on wear performance. Steeper the slope higher the effect on mass loss of nanocomposites. The load has a relatively steep slope, which causes mass loss to be larger. The higher the GNP concentration, the smaller the mass loss, according to the slope of the nanoparticle. There is a higher loss in mass due to the heat generation between Pure Epoxy had the most mass loss when tested at 300 RPM, 2 kg load, and 10 min. The reinforcement of pure Epoxy can explain the improvement of wear resistance by high strength and hardness GNP. This improved tribological behavior of nanocomposites can also be attributed to the transfer film's improved characteristics. During the wear process, GNP sustains a lot of loads and reduces the stress between the Epoxy and GNP, thereby securing the matrix. GNP reinforced nano epoxy composites have improved wear resistance.

The normal probability plot shows the distribution of residuals near the adjacent line. Figures 6 and 7 give the information on this distribution. The distribution of all residuals is very close to the normal distribution line. An optimal linear relationship found between response and input variables is called controlling parameters. It is confirmed via the normal dispersion of all the residuals. The residual is plotted on the y-axis, while the estimated response is plotted on the x-axis. This graph displays nonlinearity, unequal error variances, and outliers [29, 30]. Figures 4 and 5 show the residual order versus the experimental order. There is no more fluctuation of all the points in the plot near the main line. So that we can say due to symmetrical axes, the given model directly fits the obtained results precisely.

Equations (1) and (2) is regression equation of mass loss and COF that linked with input parameters  
Regression equation

$$-M^{-1} = -0.016347 - 0.009689C + 0.002091P + 0.000178T \quad (1)$$

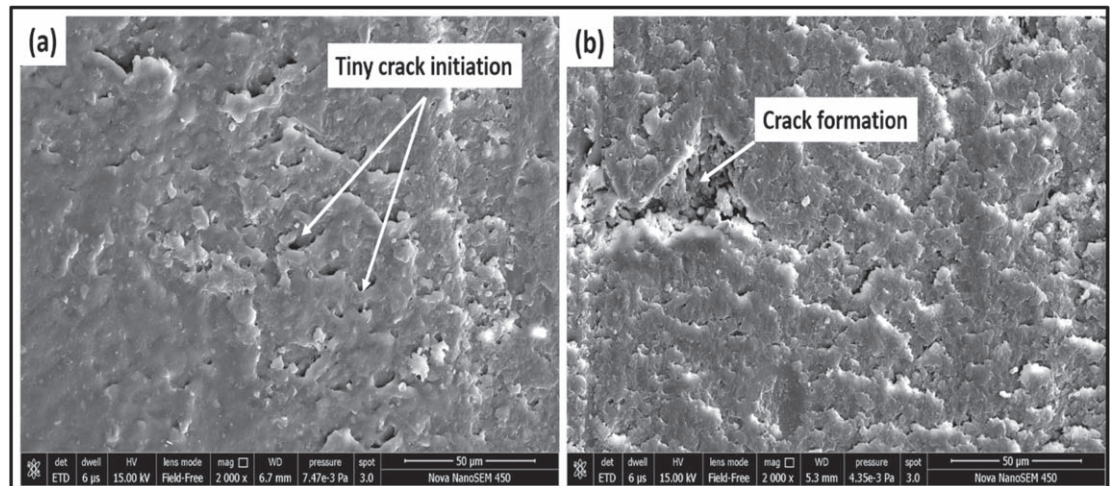


Figure 8. Worn surfaces SEM image of pure Epoxy at different conditions.

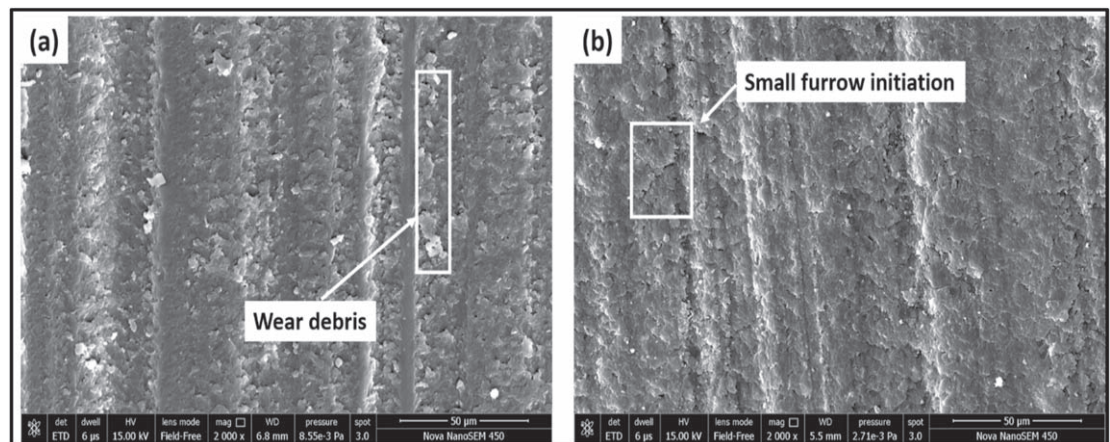


Figure 9. Worn surfaces SEM image of nano epoxy composites (0.1 wt% GNP) at different conditions.

Table 6. Optimum result analysis.

| Responses | Optimum parameters | Optimum value (predicted) | Optimum value (experimental) |
|-----------|--------------------|---------------------------|------------------------------|
| M (mg)    | C4P1T1             | 51.45                     | 53.10                        |
| COF       | C4P1T1             | 0.48                      | 0.49                         |

Regression equation

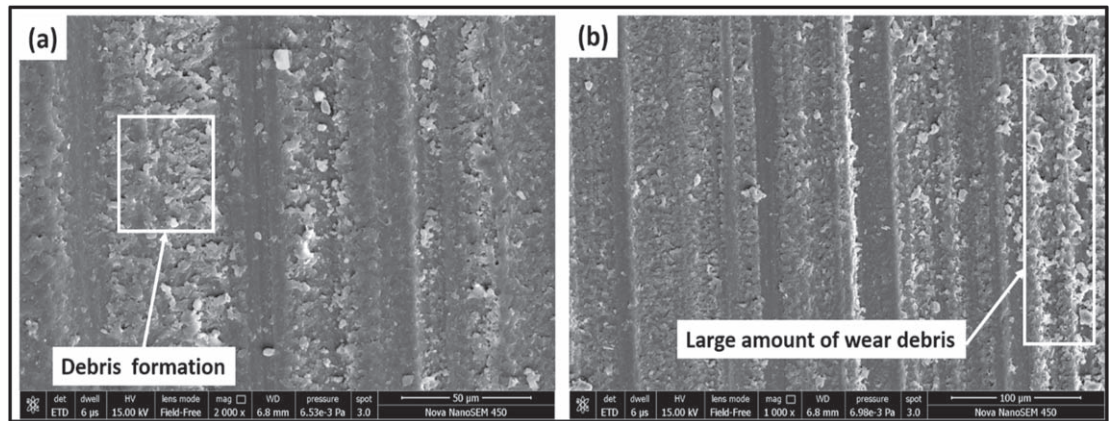
$$\frac{(\text{COF})^{\lambda-1}}{\lambda \times g^{\lambda-1}} = -0.0911 - 0.2675C + 0.03056P - 0.00196T$$

$$(\lambda = -4, g = 0.471542 \text{ is the geometric mean of COF}) \quad (2)$$

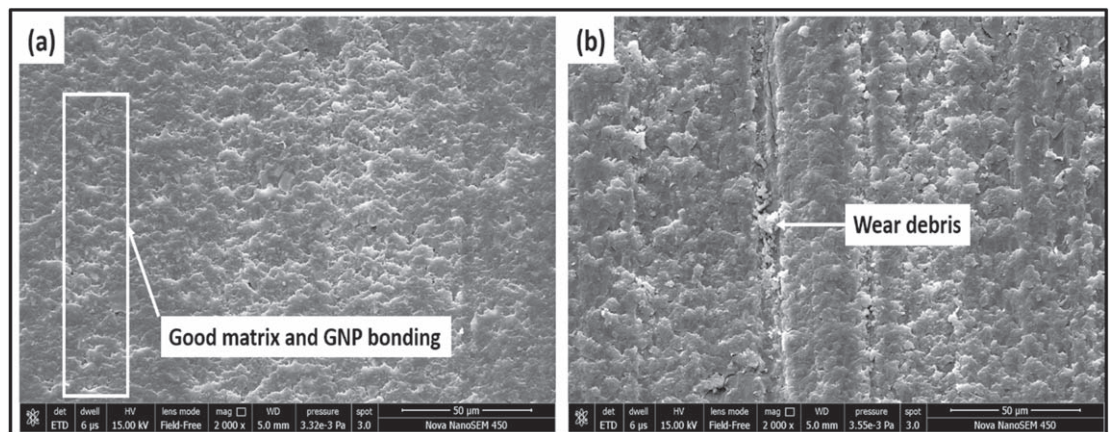
Where M is the mass loss, C is the nanoparticle wt%, P is the load, and T is the time. Equations (1) and (2) can estimate the anticipated mass loss and COF values. This equation gives the optimum value of both M and COF. Then the experimental value is compared with this optimum value. The difference between observed and expected values is known as the residual error.

### 3.3. Prediction of optimum condition

Table 6 shows the optimum parameters for the wear test, which show the best results. Using these parameters confirmation test was performed. C4P1T1 parameters give the lowest mass loss and friction coefficient for epoxy



**Figure 10.** Worn surfaces SEM image of nano epoxy composites (0.25 wt% GNP) at different conditions.



**Figure 11.** Worn surfaces SEM image of nano epoxy composites (0.5 wt% GNP) at different conditions.

nanocomposites. The test was repeated three times to predict more accurate results. Then  $M$  and  $COF$  were determined. The experimental value of  $M$  and  $COF$  is closer to the predicted value. Confirmation experiments carried out under the same conditions are used to validate it.

### 3.4. SEM analysis of worn surfaces

Figure 8 displays the worn surfaces of pure Epoxy. The worn surface is relatively rough at low load and less time conditions (0.5 kg load and 4 min) shown in figure 8 (a). The high amount of matrix removal from the surface is present in higher load conditions (2 kg load and 10 min), as shown in figure 8 (b). The contact temperature and mass loss significantly increase, resulting in an accelerated matrix split. Surface damage is increased due to this phenomenon. Cracks are generated due to the removal of base material called matrix [26]. Figure 9 shows the worn surface image of 0.1 wt% GNP-filled epoxy composite at different conditions. In the first figure, wear debris is observed in low load conditions, while in the second figure, small furrow initiation takes place in higher load. These small furrow leads to crack formation. Figure 10 shows the worn surface image of 0.25 wt% GNP-filled epoxy composite at different conditions. A large amount of wear debris is visible in the second image. That is the indication of the losing matrix. The images of the worn surfaces of 0.5 wt% GNP-filled epoxy composite is as shown in figure 11. The worn surfaces are noticeably smoother under similar input levels, and matrix detachment is significantly reduced with the addition of GNP. The addition of GNP prevents the crack and fracture propagation in the Epoxy [27]. It is important to note that wear is often caused by a combination of causes rather than a single mechanism. [14].

This suggests that including GNPs into an Epoxy composite decreases wear and enhances wear resistance. The failure of the specimens was predominantly due to matrix deformation. When the stresses at the matrix interface are more than interfacial strength, cracking [20, 22] is generated, shown in figure 8. The wear track on

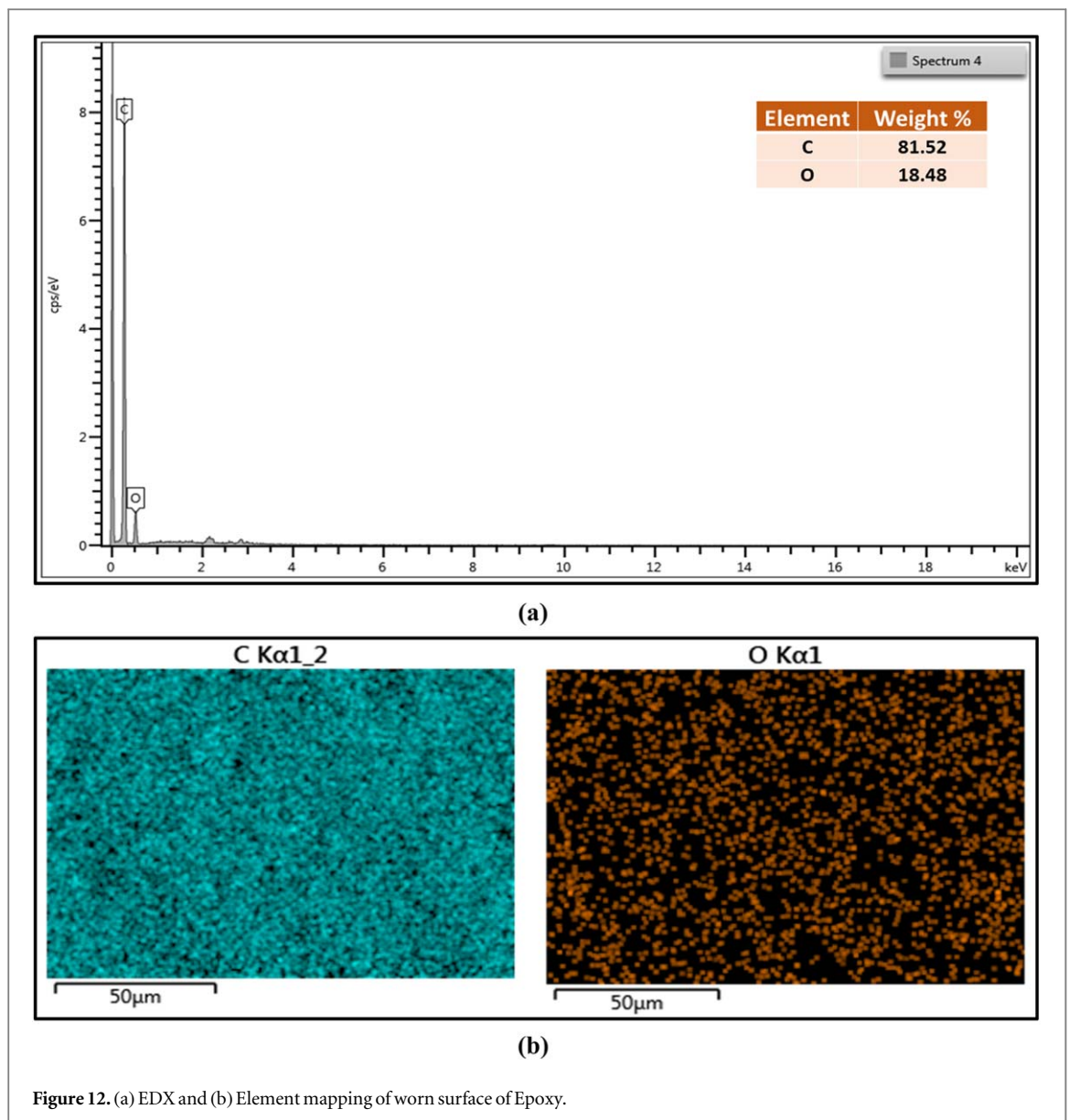


Figure 12. (a) EDX and (b) Element mapping of worn surface of Epoxy.

the pure epoxy resin is broader and more profound, indicating more material loss and abrasive wear. Wear loss results are linked with the microstructure of specimens for their support. It is indicated that as the load increases, more surface damage occurs. The  $M$  of composites is higher under increasing loads, as shown in the graph. Shear forces dominate over the other forces between the GNP in the epoxy matrix at lower stresses due to sliding action [15]. GNP dispersed most of the sliding surfaces which reduces the surface contact area for composites. GNP acts as a shield for the surface, protecting the specimen from additional damage. The composites are subjected to more wear due to the hard debris. Debris removed from the specimen surface produces a layer called thin film. It is the leading cause of wear reduction at higher loads. Figure 9 (a) shows a smoother surface that indicates suitable matrix and nanoparticle bonding. Typical wear scar generated under various loading circumstances, offering the highest wear among all applied loads. SEM images depict matrix degradation and removal due to the creation and spread of micro and macrocrack at the surfaces.

For a better understanding of worn surfaces of neat Epoxy and nano epoxy composites, an EDX test was performed. It gives information on different elements present in Epoxy and nanocomposites and their distribution on the worn sample surface. Figures 12 and 13 show the EDX and element mapping of worn surfaces. EDX graph of neat Epoxy shows carbon and oxygen elements present in different weight percentages. It contains 81.48% C and 18.48% O. Dot mapping confirms the presence of C and O. EDX graph of nano epoxy composites containing C, O, Al, Si, and Ca elements in different weight percentages. It is confirmed by element dot mapping.

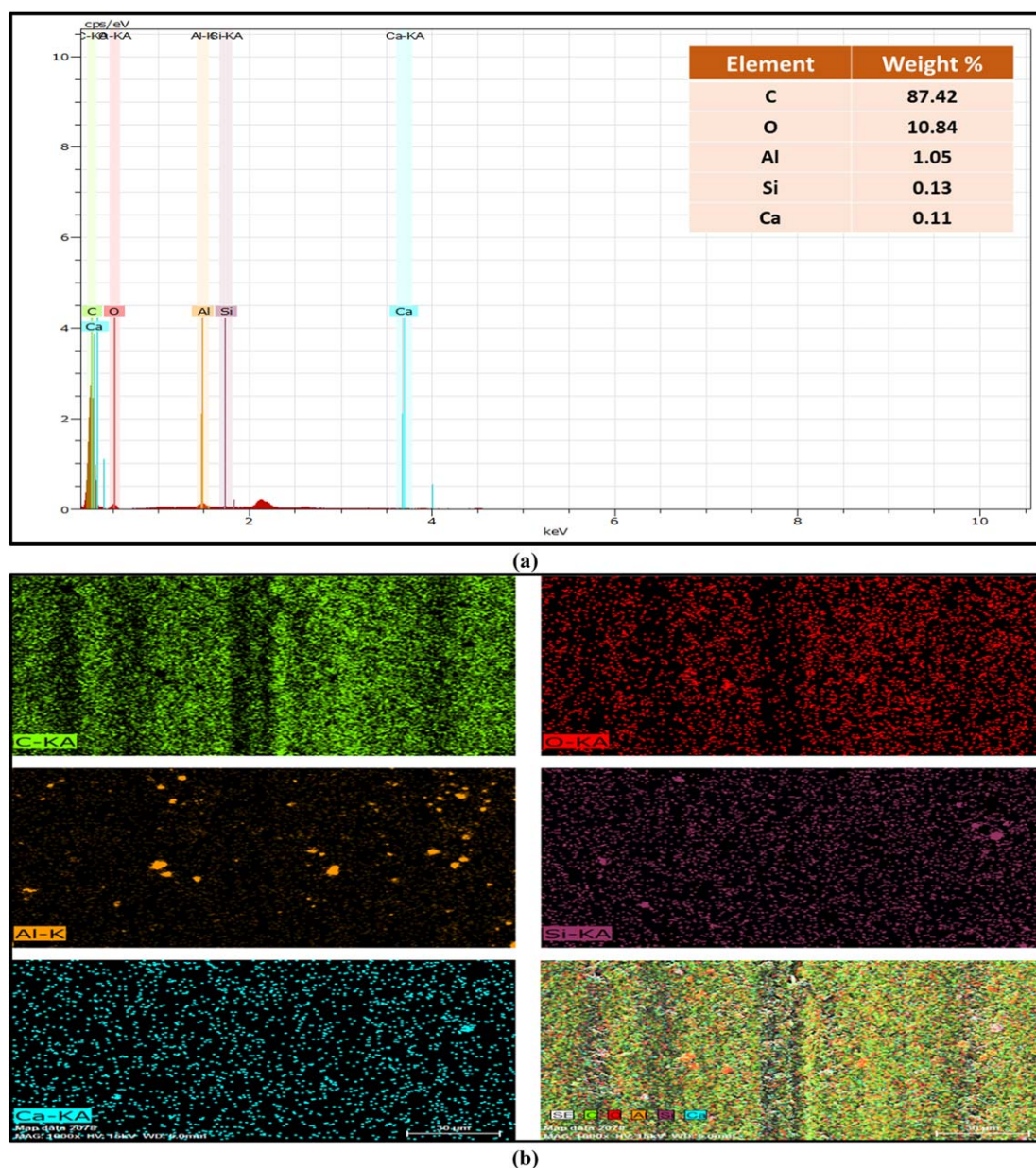
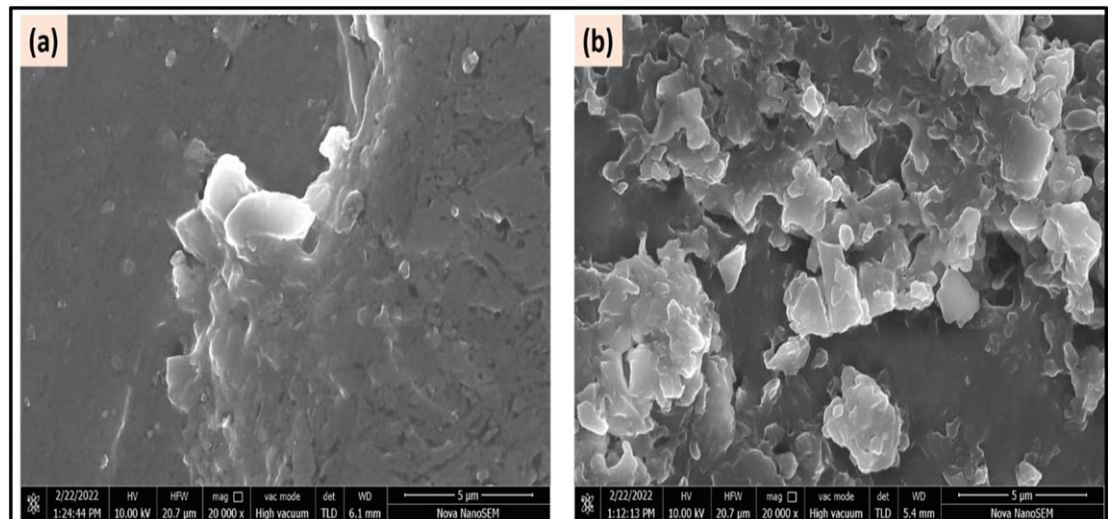


Figure 13. (a) EDX and (b) Element mapping of worn surface of nano epoxy composite (0.5 wt% GNP).

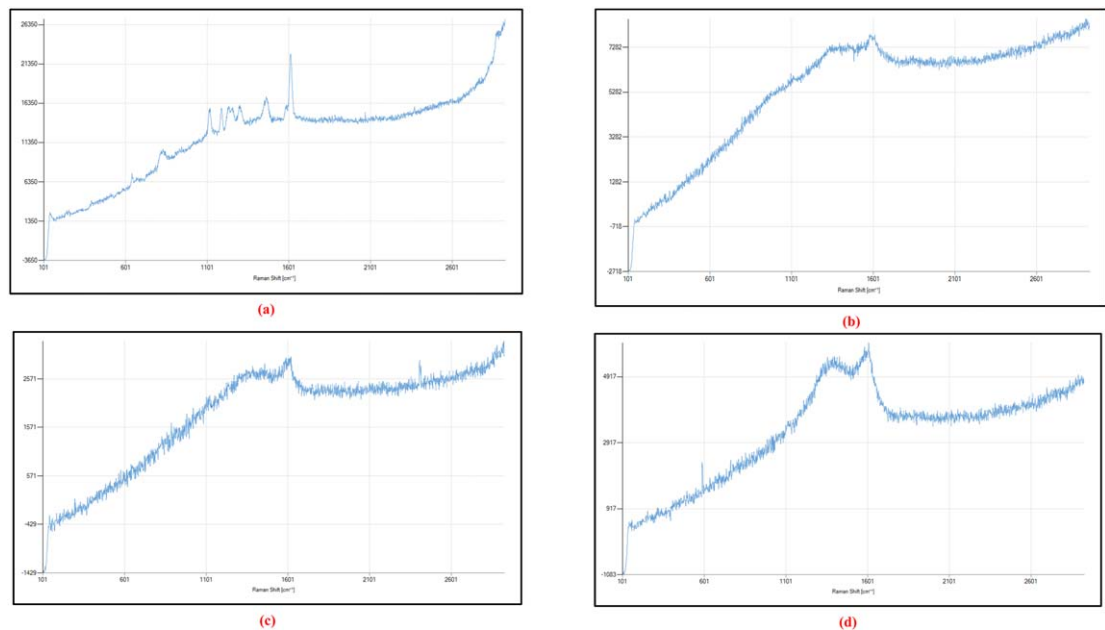
The dot maps of Carbon confirm GNP present in nano epoxy composites. These data also support the homogeneous dispersion of GNP in the composite. GNP is difficult to perceive on a worn surface. C-dot mapping, on the other hand, can validate their existence. In figure 9, the EDX graph shows 81.52% carbon element, while in figure 10, it is 87.42%.

Figure 14 shows the image of Epoxy and GNP-filled epoxy composites for comparison purposes. In the second image proper dispersion of GNP occurs in the nanocomposites.

Figure 15 shows the Raman spectra of worn surfaces of neat Epoxy and different weight percent GNP filled epoxy composites. Epoxide vibration of neat Epoxy is between of  $1230\text{ cm}^{-1}$  and  $1280\text{ cm}^{-1}$  [33]. In figure 15 (a) epoxide band lie at  $1260\text{ cm}^{-1}$ . The strength of this peak is proportional to the amount of epoxide groups present in the resin mixture. It is observed that epoxide ring deformation is weak at peaks at  $910\text{ cm}^{-1}$ ,  $735\text{ cm}^{-1}$ , and  $640\text{ cm}^{-1}$  [34]. There is no change in peak intensity at  $1601\text{ cm}^{-1}$  during wear in all nanocomposites, including Epoxy. Data can be normalized using this peak. According to Marcos Ghislandi *et al* [35], graphene shows D, G, and 2D bands at  $1350$ ,  $1570$ , and  $2680\text{ cm}^{-1}$ . It is determined from figure 15 (b)–(d) that GNP-filled epoxy composites show a similar band trend of D and G at almost the same peak intensity. It is a clear indication of GNP present in epoxy composites.



**Figure 14.** SEM image of (a) Epoxy (b) GNP dispersion in epoxy.



**Figure 15.** RAMAN spectra of (a) Epoxy & (b) 0.1 wt. GNP (c) 0.25 wt% GNP (d) 0.50 wt% GNP filled epoxy composites.

## 4. Conclusions

The following conclusions are drawn from the current research:

1. The lowest wear is obtained at 0.5 wt% of GNP. It is confirmed that the addition of GNP to the Epoxy improves the wear characteristics. M and COF are mostly affected by GNP weight percent compared to other factors taken in the experiment.
2. C4P1T1 is used as an optimal variable for reducing the mass loss and COF. The experimental results are found to be closer to the optimum results.
3. Analysis of variance shows the nanofiller quantity and load have more impact on wear for epoxy nanocomposites compared to time.

4. The many types of mechanisms responsible for specimen wear were revealed by SEM images of worn surfaces of specimens. EDX mapping shows the increment in Carbon percentage which is an indication of GNP present in different composites.

## Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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