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WEAR CHARACTERIZATION OF HNT FILLED GLASS-EPOXY COMPOSITES USING TAGUCHI'S DESIGN OF EXPERIMENTS AND STUDY OF WEAR MORPHOLOGY

Glass-epoxy composites are increasingly being used in several industrial applications, viz. automobile, marine, aerospace, electrical and electronics components, especially in tribological components, viz. bearings, impellers, cams, driving wheels, bolts, nuts, seals, bushes and gears, which are used extensively in machinery because their lower weight, exceptional strength, resistance to corrosion capabilities, and cost effectiveness. The work focuses on optimization of the process parameters of the dry sliding wear test, viz. the applied load, disc rotation speed, weight percentage (wt.%) of the Halloysite nanotube (HNT) filler, time as well as the track diameter to minimize the wear rate of the glass fabric reinforced epoxy composite against EN-32 steel. In this research, the specimens are fabricated in accordance with the ASTM G-99 standard and the experiment is carried out with various combinations of parameters using a pin-on-disc tribometer, while keeping the time and track diameter constant. To proceed further, trial runs are conducted using MINITAB 19 software to optimize the process parameters for minimum wear by developing Taguchi's design of experiments (DOE) based on the L45 orthogonal array (OA), and subsequent analysis of the signal-to-noise (S/N) ratio. The results of the optimization clearly indicate that the wt.% of HNT is the most significant parameter that has a significant effect on minimizing the applied load, speed and sliding wear rate. In overview, the experiment results showed that the combined parameters influenced the wear. In addition, scanning electron microscopy (SEM) is performed to study the surface morphologies of the worn specimens and determine the wear mechanism in accordance with the test results. The wear mechanism clearly indicates that there is a larger amount of matrix debris, fiber breakage and fiber-matrix debonding in the neat composites as compared to the HNT filled glass-epoxy composites since a distinct pattern of micro coring and segregation of the filler along the peripheries of the glass fiber-epoxy interstitial sites, leading to strong bonding between the fibers and matrix are observed in the HNT filled composites. The strong bonding thus resists the wear to a certain extent, and the wear debris is relatively less in the HNT filled composites as compared to the neat composites.

Keywords: **glass-epoxy, Halloysite nanotube, sliding wear, Taguchi analysis, micrographs**

INTRODUCTION

In recent years, industries around the globe have been focused on manufacturing quality products for various applications; hence researchers are concentrating their efforts on developing advanced materials, viz. composites, high strength steels, shape memory alloys and excellent ceramics. Among these materials, due to their many advantages and unique characteristics, polymer composites are preferred [1]. In the category of composites, glass fabric reinforced epoxy composites are extensively used for several applications because of their exceptional properties [2]. Currently, several experimental trials are being conducted world-wide to enhance the characteristics of polymer composites with the inclusion of various fibers, fillers and their combinations [3, 4]. The addition of organo-modified montmorillonite (OMMT) nanoclay, significantly increases the mechanical and tribological properties of polymer composites [5]. Naveed Anjum et al. [6] investigated the wear of glass-epoxy composites with the addition of different wt.% of SiO_2 filler. They constructed a Taguchi based L27 orthogonal array considering the parameters, viz. sliding distance, normal load, and sliding velocity to evaluate the wear performance. The results of the study revealed that the wear volume moderately increases with the sliding distance, applied load, and sliding velocity.

Annappa and Basavarajappa [7] conducted research on the wear of functionally graded graphite particles on the dry sliding wear properties of glass-epoxy composites. The following parameters, viz. applied load, sliding distance and sliding velocity, were considered as the control variables for the wear conditions. Their study revealed that the inclusion of graphite particles decreases the specific wear rate.

Senthil Kumar et al. [8] investigated the sliding wear properties for two conditions, viz. with and without an addition of E-glass reinforcement in the epoxy matrix filled by Cloisite 25A nanoclay in different wt.%. In their studies, the Taguchi method, ANOVA and the signal-to-noise ratio (S/N ratio) were thoroughly discussed and they developed an L25 orthogonal array to determine the wear rate by optimizing the variables such as normal load, sliding distance, filler content, and sliding velocity. In the results, they concluded that the combination of variables had a great influence on minimizing the wear volume.

Agrawal et al. [9] conducted sliding wear tests of glass fiber reinforced epoxy composites against EN31 steel under three different environment conditions, viz. inert gas, dry, and oil-lubricated. The parameters, viz. sliding speed, sliding time and applied load were considered as the tribological conditions. The minimum wear rates were achieved in the oil-lubricated conditions followed by the dry friction conditions and the inert gas (argon) atmosphere.

The Halloysite nanotube (HNT) is a new class of filler which exists naturally in a tubular morphology similar to the single-wall carbon nanotube (SWCNT). It has an outer diameter of 50÷200 nm, internal diameter of 5÷30 nm and a length of 0.5÷2 mm. Chemically, HNTs belong to the kaolinite family containing rolled aluminosilicate layers, i.e. a tetrahedral SiO_4 sheet adjacent to an octahedral AlO_2 $(OH)_4$ sheet [10]. Besides that, HNT has unique characteristics, viz. better mechanical characteristics, biocompatibility, a tubular microstructure, flame retardancy, biocompatibility, and a versatile surface chemistry which has inspired several researchers to fabricate and characterize HNT-polymer nanocomposites [11].

From the literature review, it is evident that even though significant research work is carried out to evaluate the wear properties of polymer nanocomposites fabricated with the addition of different nanoparticles, a wide scope to identify and introduce a new class of nanofiller which enhances the tribological performance for real-time engineering applications still exists [12]. Hence the current work was undertaken to evaluate the effect of HNT on the wear resistance properties of glass-epoxy composites. In conjunction with the above scope, a detailed analysis is conducted to optimize the wear control factors (load, sliding speed and wt.% of HNT) under dry sliding conditions using the Taguchi experimental design approach to achieve the minimum wear rate [13, 14]. Additionally, the wear morphologies are studied from the microstructure of the worn surfaces.

EXPERIMENTAL PART

Materials and methods

In this study, an E-glass woven fabric mat (supplied by SunTech Fabrics Pvt. Ltd, Bengaluru, India) is used as the reinforcement, an epoxy matrix (Lapox L-12, supplied by ATUL India Ltd, Gujrat, India) and a nanofiller, viz. Halloysite nanotubes (HNT) purchased from the Sigma-Aldrich Company, Bengaluru, India are used to fabricate composite laminates by the vacuum bag molding technique [15, 16]. In conjunction, a hardener (K6) is mixed in the ratio of 1:10 by weight with the epoxy resin. The details of prepared composites are presented in Table 1. In addition, specimens (12 mm×12 mm×3 mm) are prepared by cutting composites as per the ASTM G99 standard test specifications to conduct the experimental trials.

TABLE 1. **Designation and composition of prepared composites**

Sl. No.	Designation	Composition [wt.%]			Fabrication method
		Epoxy	HNT	Glass fiber	
1	E/GF	40	-	60	Vacuum bag molding
2	E/GF/1-HNT	39	1	60	
3	E/GF/2-HNT	38	2	60	
4	E/GF/3-HNT	37	3	60	
5	E/GF/4-HNT	36	4	60	

Testing details

A pin-on-disc tribometer (TR-20LE-PHM-600, supplied by DUCOM Industries, India) is used to perform wear tests under dry sliding condition as shown in Figure 1. The specimens are mounted against the counter disk made of EN32 steel of 72 HRC hardness. Three trials are conducted for each specimen separately on the test rig and their average values are recorded. The disc is cleaned with acetone before conducting each trial as per the wear conditions. Table 2 represents the levels of wear conditions employed to conduct the Taguchi analysis. The weight loss of the test specimens is measured using a precision electronic weighing balance with an accuracy of ±0.0001 g. Finally, Equation (1) is used to find the value of the specific wear rate based on the wear volume

$$K_S = \frac{\Delta w}{\rho \times t \times V_S \times F_n \times 10^3} \tag{1}$$

where: Ks – specific wear rate [mm^3/Nm], $\Delta W = W_1 - W_2$ weight loss [g], W_1 – initial weight of the specimen [g], W_2 – final weight of the specimen after wear out [g], ρ – density of the composite [g/cm^3], t – time [s], $V_S = \frac{\pi D N}{60000}$ – sliding velocity [m/s], D – diameter of track [mm], N – speed [RPM], F_n – average normal load [N].

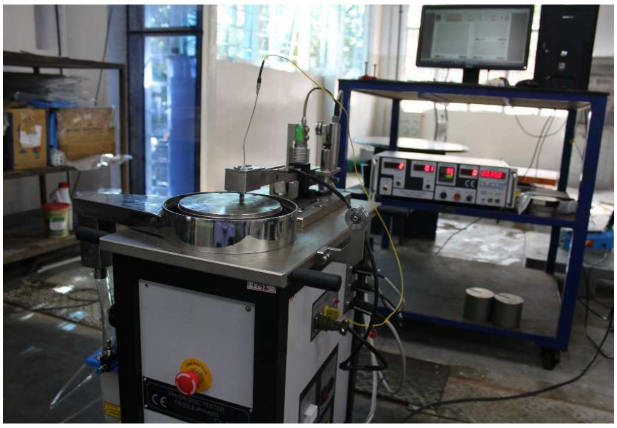

Fig. 1. Pin-on-disc tribometer

TABLE 2. **Levels of wear conditions**

Level	**Process parameters**		
	Load F_n [N]	**Speed N [RPM]**	**HNT filler loading [wt.%]**
1	40	400	0
2	80	800	1
3	120	1200	2
4	-	-	3
5	-	-	4

Three levels of load in N, speed in RPM are considered, for 5 different levels of HNT filler loading in wt.%, viz. 0, 1, 2, 3 and 4 to find out the threshold wt.% of HNT filler loading for an optimal set of process parameters during evaluation of the wear conditions of the HNT filled composite specimens.

Taguchi experimental design

The Taguchi method is an important statistical tool employed in the study to optimize the process to yield the minimum wear factors in the wear tests carried out using a typical pin-on-disc tribometer. In general, the technique involves analysing the signal-to-noise (S/N) ratio and identifying the significant factors which influence the output parameters. In the present work, "smaller the better" is considered the S/N ratio characteristic to evaluate the wear, since the wear and wear rate have to be minimized for the HNT filled glass fibre reinforced epoxy laminate composites**.** Currently, tribological characterization is carried out with three process parameters, viz. HNT filler loading (wt.%), load [N] and speed [RPM] while keeping the time (3600 s) and track diameter (100 mm) as constant variables to minimize the output parameter such as the specific wear rate [mm^3/Nm].

Table 3 presents the experimental design matrix along with the quantified values of specific wear rate based on the L45 orthogonal array. These measured values are correlated to obtain a signal-to-noise ratio. The influence of these process parameters on the tribological characteristics is evaluated using SN response tables. Furthermore, all the test results are subjected to ANOVA analysis with a significance level of 5% using MINITAB 19 software specifically used in (DOE) applications.

Microstructural analysis

Micrographs of the worn out surface are taken by means of a scanning electron microscope (TESCAN - VEGA 3) operating at an accelerating voltage of 25 kV. To render a conducting layer on the worn surfaces, the specimens are coated with few nanometer thick layer of gold, before conducting the SEM observation [17].

RESULTS AND DISCUSSIONS

Taguchi's optimization

The measured values of the specific wear rate after conducting the experiments with respect to various combinations of process parameters in accordance with the L45 orthogonal array are displayed in Table 3.

TABLE 3. **Standard L45 orthogonal array with experimental results**

Experiment No.	**Load F_n**	**Speed N**	**HNT filler loading**	**Specific wear rate K_s**
	[N]	**[RPM]**	**[wt.%]**	**$10^{-5}\times$ [mm^3/Nm]**
1	40	400	0	60.12
2	40	400	1	13.91
3	40	400	2	14.73
4	40	400	3	19.40
5	40	400	4	20.79
6	80	400	0	51.59
7	80	400	1	12.16
8	80	400	2	11.32
9	80	400	3	12.93
10	80	400	4	13.52
11	120	400	0	67.16
12	120	400	1	13.51
13	120	400	2	12.45
14	120	400	3	21.58
15	120	400	4	22.88
16	40	800	0	34.81
17	40	800	1	10.40
18	40	800	2	11.31
19	40	800	3	11.85
20	40	800	4	12.99
21	80	800	0	35.99
22	80	800	1	7.24
23	80	800	2	7.36
24	80	800	3	10.24
25	80	800	4	10.66
26	120	800	0	35.98
27	120	800	1	8.11
28	120	800	2	7.92
29	120	800	3	11.68
30	120	800	4	13.35
31	40	1200	0	59.20
32	40	1200	1	8.89
33	40	1200	2	15.84
34	40	1200	3	22.29
35	40	1200	4	21.83
36	80	1200	0	55.59
37	80	1200	1	14.28
38	80	1200	2	11.89
39	80	1200	3	13.66
40	80	1200	4	14.73
41	120	1200	0	71.43
42	120	1200	1	15.07
43	120	1200	2	9.21
44	120	1200	3	22.29
45	120	1200	4	25.88

The rank of parameters determined using the obtained values of S/N ratios and mean of means of different process parameters affecting the wear rate are shown in Table 4 and 5, respectively. It can be seen that the wt.% of HNT exhibits an influence on the wear rate, ranked 1, followed by the speed and load, respectively. Overall, the conclusion drawn is that level 3 of the wt.% of HNT filler loading (2 wt.%) followed by level 2 of the speed (800 RPM) and level 1 of the load (40 N) are considered as the optimal values for the minimum wear rate due to the smaller response values. The main effects plot for the S/N ratios and the main effects plots for the means are presented in Figure 2 and Figure 3, respectively; it is also evident that the same levels of the parameters contribute to the process to minimize the wear values.

TABLE 4. **Response table for signal-to-noise ratios (smaller the better)**

Level	Load [N]	Speed [RPM]	HNT filler loading [wt.%]
1	25.76	27.68	34.50
2	26.69	24.49	24.05
3	28.20	28.48	23.20
4			25.98
5			26.69
Delta	2.44	3.98	11.30
Rank	3	2	1

TABLE 5. **Response table for means (smaller the better)**

Level	Load [N]	Speed [RPM]	HNT filler loading [wt.%]
1	23.02	28.35	54.65
2	24.88	18.78	16.22
3	29.28	30.05	14.79
4			20.56
5			22.40
Delta	6.25	11.27	39.86
Rank	3	2	1

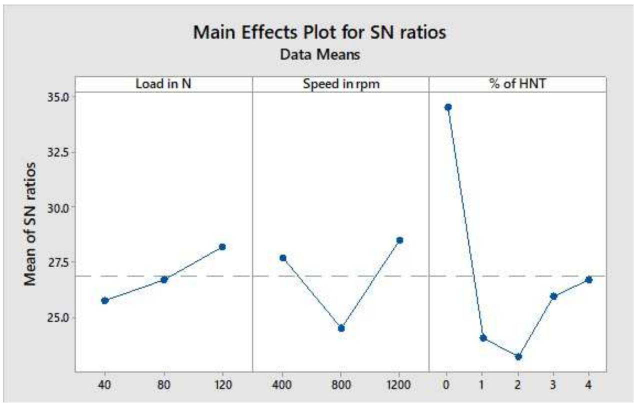

Fig. 2. Main effects plot for S/N ratios – wear rate

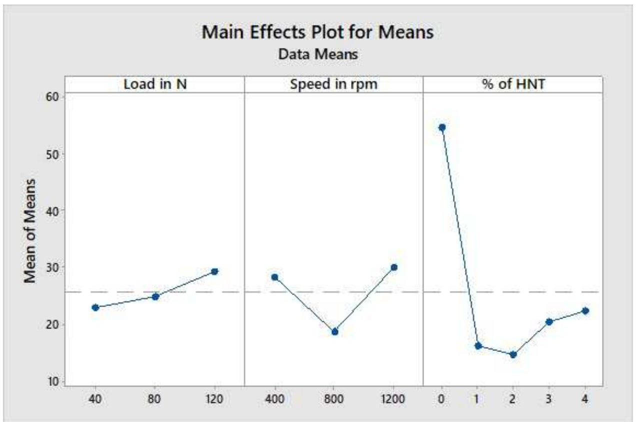

Fig. 3. Main effects plot for means – wear rate

The main effects plot for the S/N ratios clearly shows that the delta values (the difference between the maximum and minimum) is highest for the HNT filler loading, followed by the speed and load. The significant reason for the reduction in the wear rate for filler loading of up to 2 wt.% and a further increase beyond 2 wt.% is the de-bonding phenomena that occurs due to micro coring and agglomeration of HNT in the matrix phase with the increased percentage of filler addition. This is also depicted in the SEM micrographs in Figure 4d-e, i.e. the increased percentage of filler addition leads to fiber pullout - pushing the fibers out of the laminate bond due to the agglomeration of HNT filler along the peripheries of the matrix-fiber bonds.

The main effects plot for the means clearly shows that the mean of means is smaller for the load of 40 N, speed of 800 RPM and 2 wt.% HNT, thus extending the scope for optimization of the wear and the subsequent wear rate in the HNT filled glass reinforced epoxy composites.

ANOVA for wear properties

The analysis of variance is carried out to ascertain the percentage contribution of each parameter. This analysis is conducted with a significance level of 5%, i.e. for a confidence level of 95%. Finally, the source with *p*-values are considered to confirm whether the effects are statistically significant or not (if the *p*-values are less than 5%, it reveals that the effects are significant) [18, 19].

Table 6 shows the analysis of variance for the specific wear rate. It is observed that the wt.% of HNT has a contribution percentage of 82.33, hence it is considered as a major contributor for variance in the specific wear rate for the set of experimental trials over the other parameters, viz. speed and load with a 9.34% and 5.71% contribution, respectively.

In this study, the obtained source *p*-values are less than 0.05; it implies that the effects are important, which means that the process parameters significantly affect the wear rate. The R^2 value, adjusted R^2 value are equal to 0.973 and 0.964, respectively.

TABLE 6. **Analysis of variance (ANOVA) for specific wear rate**

Source	DF	Adj. SS	Adj. MS	*F*-value	*p*-value	% Contribution
Load in N	2	677.0	338.49	39.40	0.001	5.71
Speed in rpm	2	1107.4	553.69	64.46	0.000	9.34
% of HNT	4	9758.6	2439.66	284.01	0.000	82.33
Error	36	309.4	8.59			2.62
Total	8	11852.4				
R-square		0.973	Adj. *R*-square		0.964	

This indicates that the adjusted R^2 value is very close to the normal R^2 value, which demonstrates that the process parameters have a strong effect on the wear characteristics, i.e. the specific wear rate and further, the statistical correlation with the experimental trials holds good with close agreement for the validated set of values.

Study of wear morphology

Figure 4a-e shows the SEM micrographs of the worn out surfaces of 0 wt.%, 1, 2, 3 and 4 wt.% of HNT additions in the glass-epoxy composites. Figure 4a indicates that the neat composite exhibits fiber breakage, fiber matrix debonding and a greater amount of matrix debris due to the brittle nature which fails to resist the action of stress by a significant increase in the normal load. Thus, the wear rate is the maximum for the neat composites, since the laminates do not resist the loading and easily undergo wear under the action of the applied load conditions.

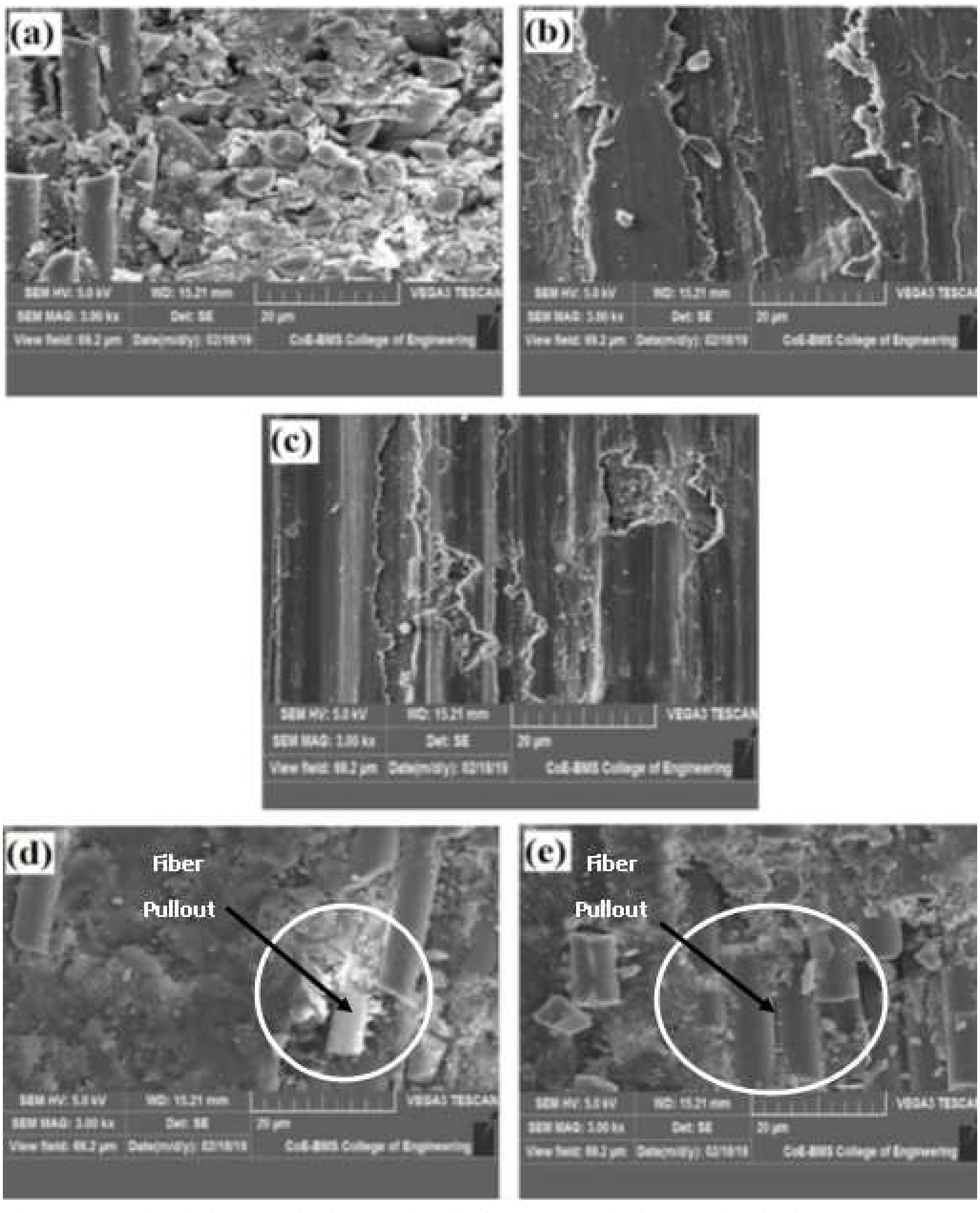

Fig. 4. Worn surface micrographs of E/GF (a), E/GF/1-HNT (b), E/GF/2-HNT (c), E/GF/3-HNT (d), E/GF/4-HNT (e) nanocomposites at higher loading conditions (120 N and 1200 RPM)

Furthermore, it is critically observed that the 1 wt.% loading and 2 wt.% loading result in stronger bonding between the HNT filler and the matrix fiber complex. Thus, it exhibits better wear resistance characteristics in terms of a significantly smaller quantity of debris, and limited exposure of fibers as shown in Figure 4b and 4c, respectively. However, the surface morphologies for the worn out surfaces of 3 wt.% loading and 4 wt.% loading of HNT as shown in Figure 4d and 4e, respectively exhibit severe damages by micro ploughing and also cause delamination at the fiber-matrix interfaces due to agglomeration. The debonding exhibited with the increased percentage of filler is clearly represented by the fiber pullout in Figure 4d and 4e, wherein the filler agglomeration pushes out the fibers. Hence, it results in a poor wear resistance property beyond the loading of 2 wt.% of HNT filler [20, 21].

CONCLUSIONS

The development and characterization of HNT filled glass-epoxy composites for wear resistance applications yielded sufficient findings. After thorough evaluation of these findings, the following conclusions are drawn:

- The addition of HNT to glass-epoxy composites reduces the sliding wear rate compared to neat composites with an increase in the load and speed values.
- Furthermore, from the statistical validations it was found that the percentage of HNT filler content is a major contributor to optimization of the wear rate, (i.e. the percentage of HNT filler content is found to exhibit an influence on the speed, and load in the wear process).
- The minimal wear rate is observed in the 2 wt.% HNT filled composites, at the speed of 800 RPM and 40 N applied load, hence these are considered as the optimal parameters.
- The worn out surface analysis using SEM micrographs reveals that some of the important morphologies such as a higher amount of matrix debris, severe brittle fracture, voids, micro cracking, micro ploughing and matrix debris, inclined fiber breakage etc. result in a decline in the wear performance of glass-epoxy composites beyond the threshold limit of 2 wt.% HNT nanofiller.

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