



# Adhesive wear characteristics of mono and hybrid CF/Ep composite with nano-HAP filler

Divya GURKAR SOMASHEKAR<sup>1</sup>, Naveena BETTAHALLI ESWAREGOWDA<sup>1,\*</sup>, and Suresha BHEEMAPPA<sup>2</sup>

<sup>1</sup> Department of Automobile Engineering, Dayananda Sagar College of Engineering, Bangalore-560078, Karnataka, India

<sup>2</sup> Department of Mechanical Engineering, The National Institute of Engineering, Mysuru-570008, Karnataka, India

\*Corresponding author e-mail: naveena-au@dayanandasagar.edu

**Received date:**

25 April 2024

**Revised date:**

17 May 2024

**Accepted date:**

29 May 2024

**Keywords:**

Hybrid composites;  
Hydroxyapatite;  
Carbon fiber;  
Epoxy;  
Two-body adhesive wear

**Abstract**

Composites materials with more than two reinforcing materials are called hybrid composites. Tailoring the composites by hybridizing fillers, fibers and matrix will yield better properties compared to mono-composites. Hence, an effort has been made in the current research work to develop carbon fiber epoxy hybrid nanocomposites, comprising different weight percentage of Hydroxyapatite (HAP) to evaluate the potential effects on tribological properties using two body sliding wear method. Taguchi technique ( $L_{27}$  array) has been adopted to investigate the impact of parameters such as filler inclusion (0%, 1.5%, and 3%), load (30, 45, and 60 N), sliding velocity (1, 2, and 3  $m \cdot s^{-1}$ ) and distance (1000, 2000, and 3000  $m \cdot s^{-1}$ ) on wear loss of developed composite. It was observed that the combination of 1.5 wt% HAP composite showed the lowest  $K_s$  and the COF. The combination of 1.5 wt% HAP filler, 1  $m \cdot s^{-1}$  sliding velocity, 45 N load and 3000 m sliding distance exhibited the lower  $K_s$  and COF of  $0.44652 \times 10^{-14}$  ( $m^3 \cdot Nm^{-1}$ ) and 0.136 respectively. The significance of the parameters was assessed using analysis of variance, revealing that the filler's contribution significantly impacted wear resistance. Developed mathematical model using Regression analysis and the predicted values from K-Nearest Neighbors (KNN) have showed good agreement with experimental values. Micrograph images were captured to analyze the wear mechanisms evident on worn surfaces, revealing failure mechanisms such as extensive matrix damage, fiber exposure resulting from matrix removal, and fiber breakage.

## 1. Introduction

Material engineers strive to develop so called advance materials having unique characteristics such as, light weight, safe, efficient, durable, economic, adopting to different working environment. It is well known that finding a single material with all of these properties is difficult. Therefore, researchers invented a newer material by heterogeneously combining and binding two or more materials based on desired properties for a particular application and such materials are termed as composite materials [1].

Composites have benefits of showing the unique properties such as higher ratio of strength to weight, good durability, ease of design and production than traditional materials. The utilization of diverse materials with varying properties has expedited the progress in creating and evaluating composites for a range of engineering purposes, including but not limited to structural, aeronautical, automotive, tribological, thermally conductive, electrical insulating, and packaging applications [2,3].

Composites are classified into metal matrix, ceramic matrix, and polymer matrix categories, depending on the matrix material used. Polymer matrix composites (PMCs) are increasingly being utilized in automotive, aerospace, defense and medical industries due to their advantageous characteristics. Including high strength-to-weight ratio, superior stiffness, resistance to wear and corrosion, optimal damping behaviour, dimensional stability, enhanced adhesion quality, and

outstanding thermal stability and chemical resistance. Particularly attributed to the properties of thermoset polymers, making them highly beneficial in the production of polymeric composites [4,5].

Broadly used matrix materials in PMCs are epoxy, polyester, vinyl ester, polypropylene, polystyrene, polyethylene. Reinforcement in PMCs are carbon, glass, basalt, graphite, ZnO, Al<sub>2</sub>O<sub>3</sub>, carbon nanotubes. Epoxy resins are the common matrix material used for fiber reinforcement as they hold many merits like high strength, stiffness, design flexibility, chemical inertness and small shrinkage during curing [6]. Epoxy resin, a thermoset material, is widely employed in various engineering applications [1,2]. Polymer-based tribomaterials are increasingly substituting the bulky metal components in structures, housings, bed ways, sliding elements, bearings, and bushings.

Carbon fibre is the predominantly employed synthetic fibres in PMCs due to low density, high specific strength, and modulus at various temperatures. Carbon filaments are produced from polyacrylonitrile, rayon, or petroleum pitch precursors. Material's scientists prefer polyacrylonitrile based carbon fibers because of the lower density with 1.75  $g \cdot cm^{-3}$  to 1.8  $g \cdot cm^{-3}$  and good strength compared to other types [7,8]. Carbon fiber fabric is of unidirectional or bidirectional. In unidirectional carbon fabric, carbon fibers run in the same direction rather than being woven together. In unidirectional fabric, carbon filament can take the load effectively along only on its axis. This becomes a critical aspect for the designer to define the load path [9].

Dhieb *et al.* [10] investigated the degradation of unidirectional carbon fibre reinforced epoxy composites when they were exposed to dry and wet reciprocating sliding along parallel, anti-parallel, and perpendicular fibre orientations. More degradation in wear resistance and surface degradation such as cracking and fiber pull outs were found under sliding in demineralized water along antiparallel direction. Suresha *et al.* [11] investigated the two-body abrasive wear properties of glass fibre and carbon fibre reinforced vinyl ester composites and identified that glass fabric reinforced composites had the highest specific wear rate (Ks) in comparison with CF reinforced composites.

Composites materials with more than two reinforcing materials are called hybrid composites. Tailoring the composites by hybridizing fillers, fibers and matrix will yield better properties compared to mono-composites. Because of the synergistic effect of different constituents of hybrid composites, component performance could be improved. Combination of two or more fibers are used in the components like space shuttles, landing gear doors, rudders, elevators, bicycle frames, leaf spring, boat hulls etc., where good mechanical properties are essential, but could not find the space in the applications where hardness and wear resistance is the major challenge. To overcome this, along with the fiber reinforcement, incorporation of organic and inorganic functional fillers is used. Ceramic particles are most used filler materials in PMCs. Silicon carbide, aluminum oxide, zinc oxide, silicon dioxide, silicon nitride, boron nitride and hydroxyapatite are the best examples for ceramic fillers [12-15].

Bazgari *et al.* [16] produced epoxy nanocomposites by adjusting the concentration of 30 nm nano  $\text{Al}_2\text{O}_3$  particles, ranging from 1 wt% to 3 wt%. The wear test outcomes indicated that the composite with a 1 wt% filler exhibited enhanced wear resistance. It was noted by Teng *et al.* [17] that with the incorporation of carbon nanotube biological lubricant the coefficient of friction (COF) dropped down by 53.84% in comparison with dry conditions. Srinivas *et al.* [18] used combination of silicon carbide ( $<60 \mu\text{m}$ ) and Graphite ( $20 \mu\text{m}$ ) particles and separately reinforcing with epoxy by varying wt% of constituents. They conducted dry sliding wear, results revealed that graphite particulate filled with more wt% shows lower COF. And also concluded that hybrid nano composites showed good wear resistance compared to mono composites.

Nanoparticles have become the preferred filler materials in fiber-reinforced composites over microparticles, offering essential property advantages for material engineers in designing and developing products. The incorporation of nanomaterials decreases porosity, enhances resistance to moisture absorption, and increases thermal stability. Nanofillers also generate a synergistic effect when combined with other components in hybrid composites, effectively hindering crack propagation in comparison to microparticles [19]. Choosing nano-sized materials over micro and macro-sized materials in composites fabrication offers several advantages due to their unique properties at the nanoscale. Nano-sized reinforcements, such as nanoparticles, nanofibers, and nanotubes, exhibit superior mechanical properties, including high strength, stiffness, and toughness, compared to larger counterparts [20,21]. Their higher surface area-to-volume ratio enables improved interaction at the interface with the matrix material, enhancing adhesion and load transfer for reinforced composites with superior mechanical performance [22,23]. Additionally, nano-sized

materials offer tailored electrical and thermal properties, making them ideal for applications in electronics and thermal management. Their lightweight nature contributes to weight reduction without compromising durability, making them advantageous for industries such as automotive and aerospace. Nano-sized materials hold great potential for creating lightweight, durable, and high-performance composites across various applications [24-27].

HAP shows the similar properties to those of lubricating fillers such as graphite, molybdenum disulphide, boron nitride, sulphides, selenides and tellurides. These solid lubricants exhibit features such as lower shear strength along the sliding direction, elevated compression strength in the load direction, and effective adhesion of the solid lubricant to the substrate surface. These fillers will be chemically stable and have a high hardness, and they will be able to work under high load, thermal, and wear conditions [28]. Due to its ability to effectively resist the wear and tear process, it can be used in tribological applications.

From the extensive literature survey, it is noted that carbon fiber exhibits improved mechanical, tribological and viscoelastic characteristics in comparison with other synthetic fibers like glass and Kevlar fibers. Although many works have been reported on carbon fiber, various micro filler (ceramic and lubricating particles) individually, no work has been reported on the reinforcement combination of bidirectional fabric, hydroxyapatite nanofillers into epoxy, adopted in this research works. Hence, an effort has been made in the current research work to develop carbon fiber epoxy hybrid nanocomposites, comprising different weight percentage of hydroxyapatite to evaluate the potential effects on tribological properties using two body sliding wear method. Experiments are carried out on composites using the Taguchi method with the aim of reducing the time required to determine the best wear conditions [29]. Taguchi technique ( $L_{27}$  array) has been adopted to investigate the effect of parameters such as filler inclusion, load, sliding velocity and distance on wear loss of developed composite. Using design of experiments the experiment is conducted for investigating optimal combination of parameters and levels for obtaining minimum Ks and COF.

## 2. Experimental details

### 2.1 Materials

In this research work, matrix material and hardener used were Araldite LY 1564 and Aradur 22962 respectively. Both matrix material and hardener were purchased from Huntsman Advanced Materials India Private Limited, Mumbai. The epoxy had a density ranging from  $1.1 \text{ g}\cdot\text{cm}^{-3}$  to  $1.2 \text{ g}\cdot\text{cm}^{-3}$ , while the hardener's density ranged from  $0.8 \text{ g}\cdot\text{cm}^{-3}$  to  $0.9 \text{ g}\cdot\text{cm}^{-3}$ . The mixing ratio of matrix to hardener was set at a fixed weight ratio of 10:2.5. Carbon fabric (T300-3k) sourced from CF Composites in New Delhi, India, was utilized as the primary reinforcement in the hybrid composites, exhibiting a low density of  $1.76 \text{ g}\cdot\text{cm}^{-3}$ , high tensile strength of 3.5 GPa, and a Young's modulus of 230 GPa. Hydroxyapatite filler, with particle sizes ranging from 15 nm to 40 nm and a density of  $3.14 \text{ g}\cdot\text{cm}^{-3}$ , was considered into the research and obtained from Sigma Aldrich Ltd, India.

## 2.2 Fabrication of hybrid composites

Nano particles HAP was used as filler materials in plain weave carbon fabric reinforced Araldite LY 1564 epoxy system. For improving the properties, epoxy and the fillers were modified with external agent. Using ultrasonication process, even dispersion of the fillers in the system is achieved. Amine containing liquid rubber hydroxyl terminated polybutadiene (HTPB) is used to modify the resin. It is carried out by mixing HTPB with the hardener at 22°C temperature around 30 min and then by adding the mixture to epoxy containing nano-filler.

Surface modification of filler particles is carried out by removing the hydroxyl group present on them. This is accomplished by combining silane coupling agent (KH560) and acetic acid solution in a 1:5 weight ratio and then nano-particles are mixed with it using 1:1 weight ratio respectively using ultrasonication process for 30 min. Afterwards, nano-particles are sieved and dried at 110°C for 3 h in an oven.

Even distribution of the fillers in resin is obtained using mechanical stirrer followed by ultrasonication process for 30 min using ultrasonicator. Ultrasonication process promotes the even-dispersion of the particles in the matrix by avoiding agglomerations. Then, calculated amount of hardener is mixed thoroughly.

Fabrication of the ceramic particles incorporated composites were done through vacuum bagging technique. Initially, the hand lay-up technique was adopted to fabricate laminates. Required number of carbon fabric layers (60 wt%) and resin (resin and hardener) was carried out as a first part of vacuum bagging process. Later, the prepared material stack was placed in vacuum bagging set-up. Here, release films were used to avoid sticking of resin to the peel ply. To absorb excess resin and to ensure adequate vacuum pressure, breather material

was placed over. Then, a plastic sheet was put over it and sealed to set apart from the atmosphere. Vacuum is created in the enclosed carbon-epoxy stack using vacuum hose connected at the sealant to compact the laminate. The fabrication process was carried out as illustrated in Figure 1. The laminates are post-cured after the vacuum bagging process to improve the quality. Table 1 indicates the composition of various constituents utilized in the fabrication of hybrid composites.

## 2.3 Two-body adhesive wear test

Adhesive wear is the phenomena of wear occurring due to sliding of two solid surfaces. When a body slides relative to the each other, material loss can be seen. When the pressure and force at the adhesive junction exceeds the inherent properties of the materials involved, wear loss may occur. The adhesive wear loss is quantified through experimental measurement utilizing a pin-on-disc wear test apparatus. The setup involves a stationary pin subjected to perpendicular loading against a rotating steel disc. Pin used here was a cylindrical part with 8 mm diameter and 30 mm length. To the tip of it, the wear sample with ASTM G-99 [30] standard dimension was glued as shown in Figure 2.

Wear test was accomplished with Taguchi Technique to determine the optimum parameters and their level combination exhibiting lower specific wear rate. Enough care was taken in mounting the sample in the pin to make perfect surface contact with the disc. Figure 3 depicts a schematic illustration of the wear test setup. Prior to and following the wear test, specimens underwent cleaning with acetone in an ultrasonic cleaner, followed by drying to determine weight. Equation (1-2) were employed to calculate wear volume and specific wear rate, respectively.

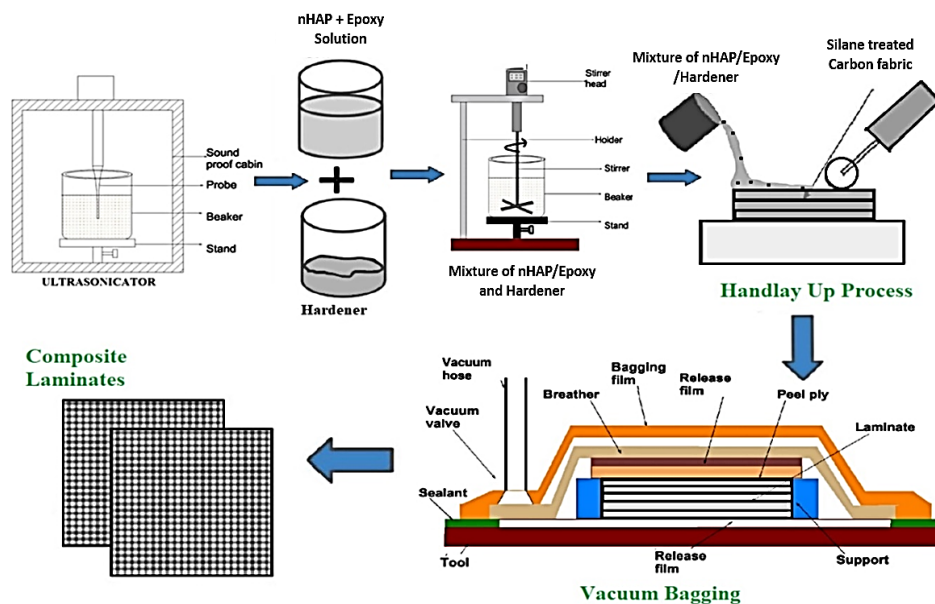
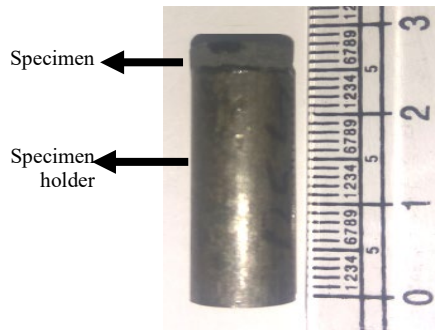


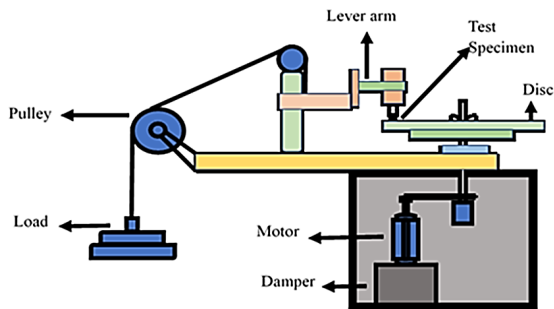
Figure 1. Schematic diagram of composite laminate preparation.

Table 1. Hybrid composite compositions.

Composite code	Reinforcement (wt%)	Matrix (wt%)	HAP Filler (wt%)
HCC1	60	40	0
HCC2	60	38.5	1.5
HCC3	60	37	3



**Figure 2.** Coupon of diameter 8mm with 3mm thick embedded to cylindrical pin of diameter 8mm and height 30 mm.



**Figure 3.** Schematic drawing of Pin on Disc apparatus.

Wear volume is given by

$$V = \frac{m}{\rho} \text{ [mm}^3\text{]} \quad (1)$$

where,  $m$  = weight loss in g,  $\rho$  = actual density in  $\text{g}\cdot\text{mm}^{-3}$

Specific wear rate is given by

$$K_s = \frac{V}{L \times D} \text{ [m}^3\cdot\text{Nm}^{-1}\text{]} \quad (2)$$

where,  $V$  is wearing volume ( $\text{m}^3$ ),  $L$  is load (N) and  $D$  is abrading distance (m)

### 3. Results and Discussion

#### 3.1 SEM and EDAX Analysis

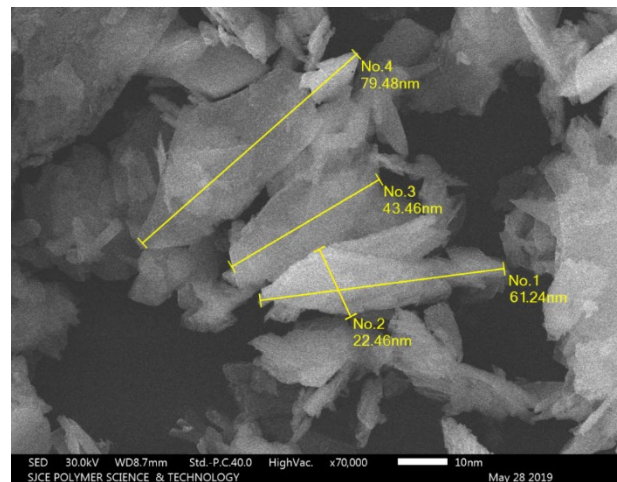
Scanning electron microscopy was utilized to analyze the morphology of the filler powders. The examination revealed that the HAP particles exhibit a needle-shaped structure, with an average particle size ranging from 15 nm to 40 nm. Figure 4 illustrates the morphology of nano HAP particles.

EDAX characterization of HAP nanoparticles is shown in Figure 5. The chemical formula of HAP is  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , However, presence of Hydrogen could not be seen in the EDAX spectra as the H atom does not have K Shell, which is important to identify the element. The percentage share among all the elements is shown in Table 2. Phosphorus and calcium are the major building blocks for ceramic materials along with silicon. The presence of calcium and phosphorus affirms the aforementioned chemical formula.

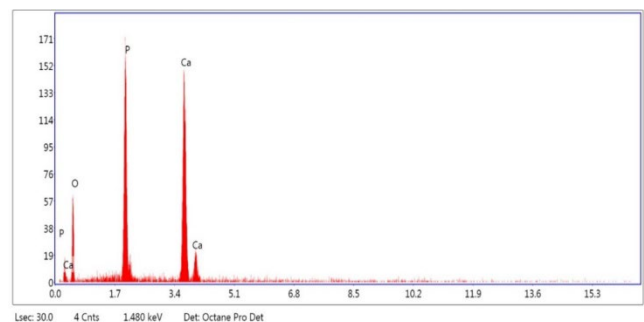
#### 3.2 Two-body adhesive wear test

To investigate its potential towards the wear resistance, dry sliding wear test was conducted based on DOE. DOE technique provides required information from the minimum time, money and the materials [31]. Taguchi technique, a part of DOE relates the parameters and the different levels with the responses. It gives the optimum combination of parameter and the levels showing the better result along with the significance of each parameter [32-34]. In the present work, Taguchi technique was well implemented with  $L_{27}$  orthogonal array consisting of four control factors such as filler content (A), sliding velocity (B), load (C) and sliding distance (D) of three levels as shown in the Table. 3.

Noted COF and calculated  $K_s$  were converted to SNR using Minitab software. Table 4 shows the experimental results and the SNR developed by software. Here the use of Taguchi technique reduced the number of experiments from 81 to 27 to give the optimum combination of 4 parameters with each 3 levels yielding lower  $K_s$  and COF. Synthetic fiber such as glass or carbon reinforcement in the polymer will restrict wear for some extent, but to enhance this property addition of micro or nano filler is required. This is beneficial in terms of reducing COF as well as  $K_s$  [35,36]. From the experiment, it was observed that HCC2 composite showed the lowest  $K_s$  and the COF of  $(0.55834) \cdot 10^{-14}$  ( $\text{m}^3\cdot\text{Nm}^{-1}$ ) and 0.156 respectively under the lowest sliding velocity of  $1 \text{ m}\cdot\text{s}^{-1}$  (B1), higher applied load of 60 N (C3) and 3000 m sliding distance (D3). From the experiments, it was also noted that the addition of the HAP is yielding lower  $K_s$  and COF.



**Figure 4.** Scanning electron micrograph of nano HAP particles.



**Figure 5.** EDAX Characterization of nano HAP particles.

**Table 2.** Elemental composition of HAP.

Element	Weight %	Atomic %
O	49.88	68.98
P	20.62	14.73
Ca	29.50	16.29

**Table 3.** Wear test conditions for CF/Ep and HAP filled hybrid CF/Ep composites.

Parameters	Units	Code	Levels		
			1	2	3
Filler	wt.%	A	0	1.5	3
Sliding velocity	m/s	B	1	2	3
Load	N	C	30	45	60
Sliding distance	m	D	1000	2000	3000

**Table 4.** SNR for the noted responses in CF/Ep and HAP filled hybrid CF/Ep composites.

Expt. Run\ Code	A	B	C	D	Ks $\times 10^{-14}$	Ks (SNR)	COF	COF (SNR)
1	0	1	30	1000	0.68425	3.297291	0.225	12.9563
2	1.5	1	30	2000	0.59835	4.463434	0.187	14.5632
3	3	1	30	3000	0.61621	4.206905	0.179	14.9429
4	1.5	2	30	2000	0.61313	4.249869	0.193	14.2889
5	3	2	30	3000	0.64522	3.807324	0.194	14.2440
6	0	2	30	1000	0.70342	3.058299	0.239	12.4320
7	3	3	30	3000	0.66614	3.529602	0.193	14.2889
8	0	3	30	1000	0.72514	2.792401	0.251	12.0065
9	1.5	3	30	2000	0.62125	4.13642	0.204	13.8074
10	1.5	1	45	1000	0.52132	5.660578	0.169	15.4423
11	3	1	45	2000	0.56245	5.001796	0.174	15.1890
12	0	1	45	3000	0.58214	4.700496	0.179	14.9429
13	3	2	45	2000	0.59427	4.522297	0.172	15.2894
14	0	2	45	3000	0.61518	4.221227	0.192	14.3340
15	1.5	2	45	1000	0.55922	5.050055	0.168	15.4938
16	0	3	45	3000	0.64331	3.833687	0.207	13.6806
17	1.5	3	45	1000	0.58142	4.713338	0.201	13.9361
18	3	3	45	2000	0.61812	4.179387	0.185	14.6566
19	3	1	60	1000	0.60826	4.320071	0.189	14.4708
20	0	1	60	2000	0.65145	3.725378	0.198	14.0667
21	1.5	1	60	3000	0.55834	5.06467	0.156	16.1375
22	0	2	60	2000	0.67146	3.462572	0.207	13.6806
23	1.5	2	60	3000	0.58214	4.700496	0.169	15.4423
24	3	2	60	1000	0.63334	3.969593	0.191	14.3793
25	1.5	3	60	3000	0.59914	4.450449	0.181	14.8464
26	3	3	60	1000	0.65523	3.673649	0.201	13.9361
27	0	3	60	2000	0.68247	3.32132	0.226	12.9178

From the Taguchi analysis the impact of the control factors on the Ks and COF was analyzed and tabulated in Table 5. In both the cases, Contribution of filler was more followed by applied load, sliding velocity and the sliding distance.

The statistical method is to know individual effects of control parameters on the test responses. From the ANOVA General linear model, contribution of the control factors on Ks and COF are evaluated in recorded in Table 6.

The analysis was carried out by taking significance level at 5% and confidence level 95%. Prominent role was played by filler content in reducing the Ks. Contribution of filler for this was shown as 47.81% in the ANOVA response. Applied load also showed 32.85% influence in decreasing the Ks, followed by 15.38% of sliding velocity impact. Very less contribution of 2.51% was noted because of sliding distance towards the Ks. Similar trend was observed in case of COF. Highest of 43.42% contribution towards COF was exhibited by the presence of filler, followed by 21.38% of load, 16.54% of sliding velocity, and 14.64% of sliding distance. Impact of sliding distance was more on COF compared to Ks. Adjusted R-Squared (R-sq(adj)) and Predicted R-Squared (R-sq(pred)) are shown in the Table. R-sq(adj) compares the goodness-of-fit for the regression model and R-sq (pred) shows how well predictions can be made using regression model.

### 3.3 The main effects plot and regression analysis

Main effect plots of SNR indicating the relation between the parameter with respect to their levels on Ks and COF is shown in

Figure 6(a-b). By considering highest value of S/N ratio the optimized parameters for attaining lower Ks and COF are filler: 1.5 wt%, Sliding velocity:  $1 \text{ m}\cdot\text{s}^{-1}$ , Load: 45 N and Sliding distance: 3000 m.

With the increase in sliding velocity, continuous increase in Ks and COF was noted due to increased rubbing between the sample and the steel counter face surface. In case of load level, Ks and COF decreased and increased. Increase in the load, increased the thermal softening of the composite in turn leading loosening network arrangement in the sample. This deteriorates material bonding resulting in higher wear loss and COF. With the increase in sliding distance, reduced Ks and COF were observed due to the developed transfer film on the steel disc [14]. Transfer film formed by wear debris consists of epoxy and the lubricating ceramic nano filler in the initial stage of the wear prevents fragmentation of epoxy surface further. A transfer film protects the fiber damage and reduces the material removal from sample surface and also reduces COF [37]. Same observation was made by Basavarajappa *et al.* [38]. The combination of 1.5 wt% HAP,  $1 \text{ m}\cdot\text{s}^{-1}$  sliding velocity, 45 N normal applied load the sliding distance of 3000 m showed the optimum result. From this the optimum combination of parameter and level was noted for conducting the confirmation test.

Figure 7 and Figure 8 show the interaction between the various parameter and the level. Parallel lines show there is no interaction between the parameters and the intersecting lines symbolize strong interaction [39].

Regression study was carried out to understand the relation between the different parameters and the response. Obtained mathematical expression for Ks and COF is shown in Equations (3-4).

**Table 5.** Responses towards Ks and COF for CF/Ep and HAP filled hybrid CF/Ep composites.

Level	Ks				COF			
	A	B	C	D	A	B	C	D
1	3.601	4.493	3.727	4.059	13.45	14.75	13.73	13.89
2	4.721	4.116	4.654	4.118	14.88	14.40	14.77	14.27
3	4.135	3.848	4.076	4.279	14.60	13.79	14.43	14.76
Delta	1.120	0.646	0.927	0.220	1.44	0.96	1.05	0.87
Rank	1	3	2	4	1	3	2	4

**Table 6.** ANOVA results for CF/Ep and HAP filled hybrid CF/Ep composites.

Ks						
Source	DF	Adj SS	Adj MS	F-Value	P-Value	P (%)
A	2	0.029162	0.014581	295.65	0.000	47.81
B	2	0.009380	0.004690	95.09	0.000	15.38
C	2	0.020041	0.010021	203.18	0.000	32.85
D	2	0.001530	0.000765	15.51	0.000	2.51
Error	18	0.000888	0.000049			1.45
Total	26	0.061002				100
S = 0.0070228, R-sq = 98.54%, R-sq(adj) = 97.90%, R-sq(pred) = 96.73%						
COF						
Source	DF	Adj SS	Adj MS	F-Value	P-Value	P (%)
A	2	0.005579	0.002789	96.93	0.000	43.42
B	2	0.002125	0.001063	36.93	0.000	16.54
C	2	0.002747	0.001374	47.73	0.000	21.38
D	2	0.001882	0.000941	32.70	0.000	14.64
Error	18	0.000518	0.000029			4.03
Total	26	0.012852				100
S = 0.0053645, R-sq = 95.97%, R-sq(adj) = 94.18%, R-sq(pred) = 90.93%						

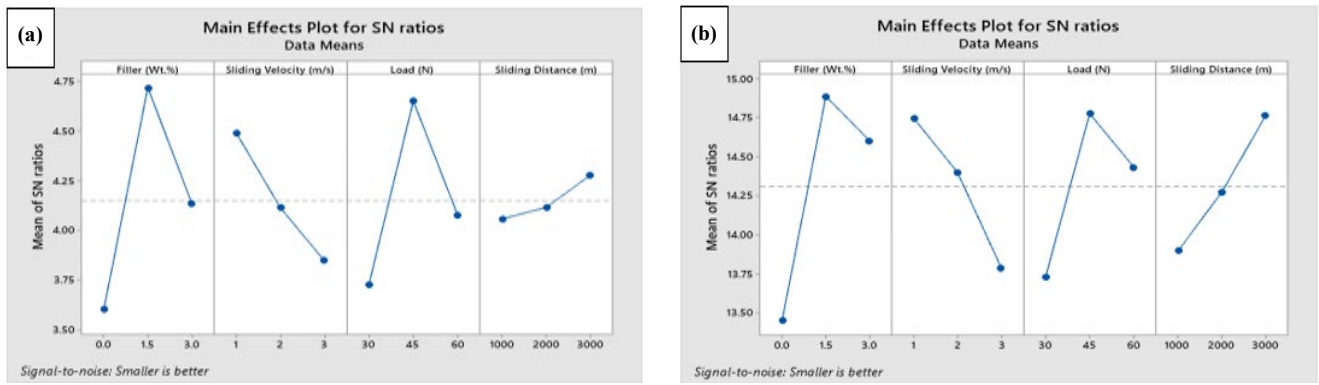


Figure 6. Main effects plot of SNR for mono and HAP filled hybrid CF/Ep composites on (a) Ks (b) COF.



Figure 7. Interaction plots of SNR for Ks for mono and HAP filled hybrid CF/Ep composites.

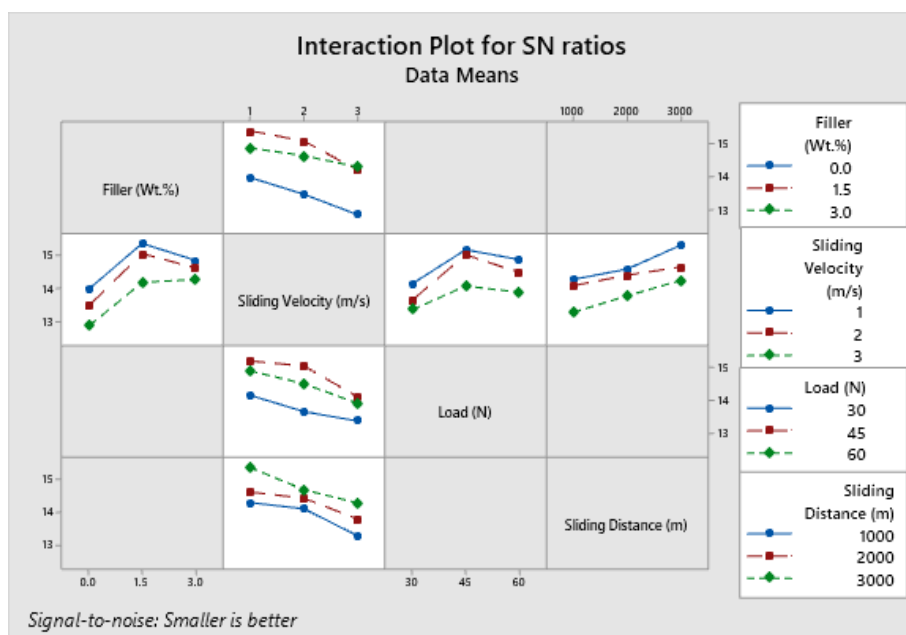


Figure 8. Interaction plots of SNR for COF of mono and HAP filled hybrid CF/Ep composites.

$$K_s = 0.6532 - 0.01332A + 0.0227B - 0.000857C - 0.000009D \quad (3)$$

$$\text{COF} = 0.2309 - 0.00911A + 0.01072B - 0.000544C - 0.000010D \quad (4)$$

Forecasting of experimental outcomes is also conducted using the K-Nearest Neighbors (KNN) Algorithm implemented in Python. K-Nearest Neighbors (KNN) is a widely used supervised machine learning algorithm for classification and regression tasks. It predicts new data points based on the majority class or average value of their 'k' nearest neighbors in the feature space, with 'k' representing the number of neighbors considered. KNN is non-parametric, making no assumptions about data distribution. While simple and easy to implement, it may be computationally expensive for large datasets and less effective with high-dimensional or imbalanced data. Additionally, KNN is utilized in forecasting experimental outcomes, where a trained dataset establishes relationships between inputs and outputs to predict values for new experiments. The 'k' factor selection is crucial, balancing between overfitting and error maximization during training and validation, often guided by the 'elbow curve' [40-42].

Obtained experimental results were compared with the regression expressions obtained from the software Minitab and from the KNN Model and expressed in Figure 9 and Figure 10. From this, it was noted that the predicted values using KNN model are near to the experimental results. With the higher experimental runs, KNN displayed very close results to experimental one.

By taking SNR plots as reference, confirmation test was conducted and the improvement in terms of percentage is tabulated in Table 7. Both from the experiments conducted and from the SNR plot, good performance was observed in 1.5 wt% HAP filled composite. By reducing applied load from 60 N to 45 N,  $K_s$  and COF were optimized.  $K_s$  and COF were reduced by 20.02% and 12.62%, respectively.

### 3.4 Analysis of worn surfaces after wear test

Figure 11 compares the worn surfaces of HAP filled CF/Ep hybrid composites subjected to different loading conditions.

Figure 11(a) shows worn surface of 1.5 wt% HAP filled CF/Ep hybrid composite under tribo-test conditions  $1 \text{ m} \cdot \text{s}^{-1}$  sliding velocity, 60 N applied normal load and 3000 m sliding distance (A2B1C3D3). The HAP filled hybrid composite slid under higher load, causing more damage to the epoxy matrix, as well as gradual fiber removal, which usually occurs in the order of fiber thinning, fiber fracture, and finally broken fiber pieces. Worn surface features such as severe matrix damage, fiber exposed due to removal matrix and fiber breakage are marked in the Figure 11(a). Figure 11(b) depicts the worn surface of 1.5 wt% HAP filled CF/Ep hybrid composites under  $1 \text{ m} \cdot \text{s}^{-1}$  sliding velocity, 45 N applied normal load and 3000 m sliding distance (A2B1C2D3). The worn surface of the same nanocomposites sample at a relatively lower load (45 N) appears much smoother. The fibers were always removed gradually and completely, which contributed to the nanocomposites' wear resistance. Fiber removal in the nanocomposite was very much less because of the sample slid relatively at a lower load of 45 N. As a result, the nanocomposite's specific wear rate is much lower than the composites at higher loading (60 N).

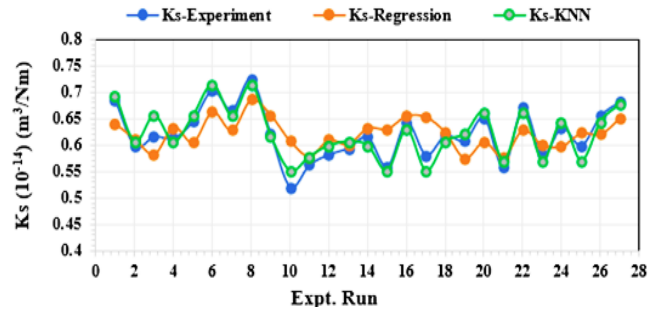


Figure 9. Validation of experimental results of  $K_s$  with obtained regression equation and KNN Model for mono and HAP filled hybrid CF/Ep composites.

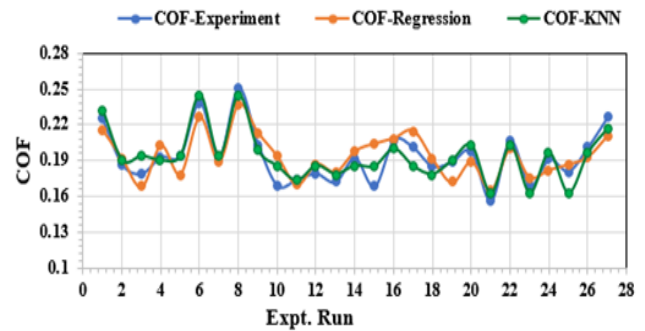


Figure 10. Validation of experimental results of COF with obtained regression equation and KNN Model for mono and HAP filled hybrid CF/Ep composites.

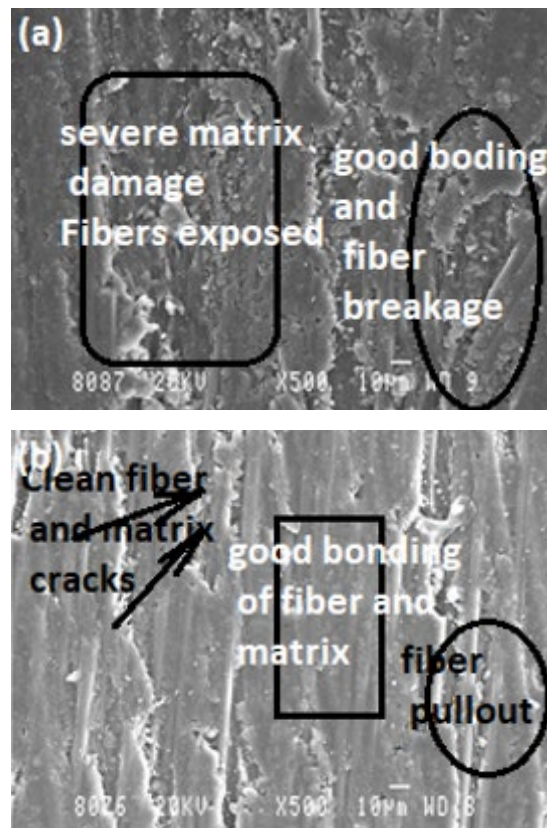


Figure 11. Worn surface of tested samples under the dry sliding condition: (a) A2B1C3D3, and (b) A2B1C2D3.



**Table 7.** Confirmation test for dry sliding wear of HAP filled hybrid CF/Ep composites.

	Initial test based on L <sub>16</sub> array	Confirmation test	Result
Level	A2B1C3D3	A2B1C2D3	(%)
K <sub>s</sub> × 10 <sup>-14</sup> (m <sup>3</sup> ·Nm <sup>-1</sup> )	0.55834	0.44652	20.02
COF	0.156	0.136	12.62

#### 4. Conclusion

In this study, ceramic particle-infused composites were fabricated using the vacuum bagging technique, examining the impact of various weight percentages of nHAP-filled CF/Ep hybrid composites on dry sliding wear behavior under diverse operating conditions. Taguchi's L27 Design of Experiment (DoE) method was utilized to optimize parameters including filler content, load, sliding velocity, and distance based on response characteristics (K<sub>s</sub> and COF) of the developed composites. The ANOVA analysis revealed that the filler contributed 47.81% to the response, with the applied load demonstrating a 32.85% decrease in K<sub>s</sub>, followed by a 15.38% impact from sliding velocity. Conversely, the sliding distance had a minimal contribution of 2.51% towards K<sub>s</sub>. This trend was also observed in the coefficient of friction (COF). The optimized parameters were found to be 1.5 wt% HAP filler, 1 m·s<sup>-1</sup> sliding velocity, 45 N load, and 3000 m sliding distance, resulting in lower K<sub>s</sub> and COF values of 0.44652 × 10<sup>-14</sup> (m<sup>3</sup>·Nm<sup>-1</sup>) and 0.136, respectively. Analysis of variance revealed the significant influence of filler content on wear resistance. Regression analysis was employed to establish empirical relationships predicting the K<sub>s</sub> and COF of the hybrid nanocomposites based on coded parameters. Experimental results, regression values obtained from Minitab software, and predicted values from K-Nearest Neighbors (KNN) were plotted, showing a strong correlation between input and output variables. Micrograph images were examined to observe wear mechanisms on worn surfaces, identifying failure mechanisms such as severe matrix damage, fiber exposure due to matrix removal, and fiber breakage.

#### References

- [1] G Gubin, Hand book of composites, Van Nostrand, Reinhold Co., New York, 1982. ISBN 0-442-24897-0.
- [2] D. Hull, and T. W. Clyne, An Introduction to Composite Materials, Cambridge University Press, Second Edition, 1996.
- [3] G. S. Suresha, G. Divya, H. M. Hemanth, and Somashekar. "Physico-mechanical properties of nano silica-filled epoxy-based mono and hybrid composites for structural applications", *Silicon*, vol. 13, pp. 2319-2335, 2021.
- [4] I. O. Oladele, T. F. Omotosho, and A. A. Adediran, "Review article - polymer-based composites: An indispensable material for present and future applications", *Hindawi, International Journal of Polymer Science*, vol. 2020, Article ID 8834518, 2020.
- [5] T.-D. Ngo, "Introduction to composite materials", *Composite and Nanocomposite Materials - From Knowledge to Industrial Applications*, Intech Open, Jul. 15, 2020.
- [6] D. Carollan, A. J. Kinloch, Alvankovic, S. Sprenger, and A. C. Taylor, "Mechanical and fracture performance of carbon fiber reinforced composites with nanoparticle modified matrices", *Procedia Structural Integrity*, vol. 2, pp. 96-103, 2016.
- [7] M. E. Islam, T. H. Mahdi, M. V. Hosur, and S. Jeelani, "Characterization of carbon fiber reinforced epoxy composites modified with nanoclay and carbon nanotubes", *Procedia Engineering*, vol. 105, pp. 821-828, 2015.
- [8] F-h Su, Z-Zhu Zhang, and W-min Liu, "Mechanical and tribological properties of carbon fabric composites filled with several nano-particulates", *Wear*, vol. 260, pp. 861-868, 2006.
- [9] A. Sayam, A. N. M. M. Rahman, M. S. Rahman, S. A. Smriti, F. Ahmed, M. F. Rabbi, M. Hossain, and M. O. Faruque, "A review on carbon fiber-reinforced hierarchical composites: Mechanical performance, manufacturing process, structural applications and allied challenges", *Carbon Letter*, vol. 32, no. 5, pp. 1173-205, 2022.
- [10] H. Dhieb, J. G. Buijnsters, F. Eddoumy, L. Vázquez, and J. P. Celis, "Surface and sub-surface degradation of unidirectional carbon fiber reinforced epoxy composites under dry and wet reciprocating sliding", *Composites: Part A*, vol. 55, pp. 53-62, 2013.
- [11] B. Suresha, G. Chandramohan, P. Samapthkumaran, Siddaramaiah, and S. Seetharamu, "Three-body abrasive wear behaviour of carbon and glass fiber reinforced epoxy composites", *Materials Science and Engineering A*, vol. 443, pp. 285-291, 2007.
- [12] P. H. C. Camargo, K. G. Satyanarayana, and F. Wypych, "Nanocomposites: Synthesis, structure, properties and new application opportunities", *Materials Research*, vol. 12, no. 1, pp. 1-39, 2009.
- [13] E. Roussi, A. Tsetsekou, D. Tsiourvas, and A. Karantonis, "Novel hybrid organo-silicate corrosion resistant coatings based on dendritic polymers", *Surface and Coating Technology*, vol. 205, no. 10, pp. 3235-3244, 2011.
- [14] B. Wetzela, F. Haupterta, and M. Q. Zhang, "Epoxy nanocomposites with high mechanical and tribological performance", *Composites Science and Technology*, vol. 63, no.14, pp. 2055-2067, 2003.
- [15] A. L. Crăciun, C. Pinca-Bretotean, C. Birtok-Băneasă, and A. Josan, "Composites materials for friction and braking application", *IOP Conference. Series: Materials Science and Engineering*, vol. 200, pp. 1-10, 2017.
- [16] D. Bazrgari, F. Moztarzadeh, A. A. Sabbagh-Alvani, M. Rasoulianboroujeni, M. Tahriri, and L. Tayebi, "Mechanical properties and tribological performance of Epoxy/Al<sub>2</sub>O<sub>3</sub> nanocomposite", *Ceramics International*, vol. 44, no.1, pp. 1220-1224, 2018.
- [17] G. Teng, Y. Zhang, C. Li, Y. Wang, Q. An, B. Liu, Z. Said, and S. Sharma. "Grindability of carbon fiber reinforced polymer using CNT biological lubricant", *Scientific reports*, 11, no. 1, pp. 1-14, 2021.

- [18] K. Srinivas, and M. S. Bhagyashekar, "Wear behaviour of epoxy hybrid particulate composites", *Procedia Engineering*, vol. 97, pp. 488- 494, 2014.
- [19] G. S. Divya, R. Keshavamurthy, C. Siddaraju, and K. V. S. Murthy, "Investigation on sliding wear properties of nano metallic particle reinforced hybrid composites through design of experiments and ANN", *Journal of the Institution of Engineers (India): Series D*, 2023.
- [20] A. B. Rashid, M. Haque, S. M. M. Islam, and K. M. R. U. Labib, "Nanotechnology-enhanced fiber-reinforced polymer composites: Recent advancements on processing techniques and applications", *Heliyon*, vol. 10, no. 2, p. e24692, 2024.
- [21] G. S. Divya, and B. Suresha, "Impact of nano-silicon dioxide on mechanical properties of carbon fabric reinforced epoxy composites," *Materials Today*, vol. 46, pp. 8999-9003, 2021.
- [22] C. I. Idumah, and C. M. Obele, "Understanding interfacial influence on properties of polymer nanocomposites", *Surfaces and Interfaces*, vol. 22, p. 100879, 2021.
- [23] S.-Y. Fu, X.-Q. Feng, B. Lauke, and Y.-W. Mai, "Effects of particle size, particle/matrix interface adhesion and particle loading on mechanical properties of particulate-polymer composites", *Composites Part B: Engineering*, vol. 39, no. 6, pp. 933-961, 2008.
- [24] K. Gajanan, and S. N. Tijare, "Applications of nanomaterials", *Materials Today: Proceedings*, vol. 5, no. 1, pp. 1093-1096, 2018.
- [25] M. M. El-Kady, I. Ansari, C. Arora, N. Rai, S. Soni, D. K. Verma, P. Singh, and A. E. D. Mahmoud, "Nanomaterials: A comprehensive review of applications, toxicity, impact, and fate to environment", *Journal of Molecular Liquids*, vol. 370, p. 121046, 2023.
- [26] G. S. Divya, and B. Suresha, "Role of metallic nanofillers on mechanical and tribological behaviour of carbon fabric reinforced epoxy composites," *Materials Science Applied*, vol. 09, no. 09, pp. 740-750, 2018.
- [27] G. S. Divya, and B. Suresha, "Metallic nanofillers effect in assessing the abrasive wear performance of carbon fabric reinforced epoxy composites," in AIP Conference Proceedings, 2019,
- [28] S. J. Shiao, W. S. Fu, C. L. Tuo, and U. I. Cheng, "Polymer-based self-lubricating material", *Journal of Applied Polymer Science*, vol. 80, no. 9, pp. 1514-1519, 2001.
- [29] G. S. Divya, B. Suresha, H. M. Somashekar, and I. M. Jamadar, "Dynamic mechanical analysis and optimization of hybrid carbon-epoxy composites wear using Taguchi method," *Tribology in Industry*, vol. 43, no. 2, pp. 298-309, 2021.
- [30] ASTM G99-17, Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus, ASTM International, West Conshohocken, PA, 2017.
- [31] B. E. Naveena, R. Keshavamurthy, and N. Sekhar, "Slurry erosive wear behaviour of plasma sprayed fly ash-Al<sub>2</sub>O<sub>3</sub> coatings." *Surface Engineering*, Taylor & Francis, Edition, ISSN: 0267-0844(Print) 1743-2944 (Online), 2017.
- [32] B. E. Naveena, R. Keshavamurthy, and N. Sekhar, "Comparative study on effects of slurry erosive parameters on plasma sprayed flyash-Al<sub>2</sub>O<sub>3</sub> and flyash SiC composite coatings on Al6061 alloy," *International Journal Computational Materials Science and Surface Engineering*, vol. 8, no. 1, pp.57-75, 2019.
- [33] B. E. Naveena, R. Keshavamurthy, and B. H. Channabasappa, Studies on parametric optimization for plasma sprayed fly ash Al<sub>2</sub>O<sub>3</sub> composite coatings," *Applied Mechanics and Materials*, Trans Tech Publications, Switzerland, vol. 813-814, pp. 511-515, 2015.
- [34] B. E. Naveena, and R. Keshavamurthy, "Optimization of process parameters for thermally sprayed composite coatings using Taguchi's technique", *International Journal of Applied Engineering Research, Research India Publications*, vol. 10, no. 55, pp. 3897-3900, 2015.
- [35] S. Deshpande, and T. Rangaswamy, "Sliding wear characteristics of bone powder filled hybrid fiber reinforced epoxy composites", *IOSR Journal of Mechanical and Civil Engineering (IOSRJMCE)*, vol. 13, no.1, pp. 68-78, 2016.
- [36] V. N. Aderikha, and A. P. Krasnov, "Solid lubricants, polymer-based self-lubricating materials", *Encyclopedia of Tribology*, Springer, Boston, MA, 2016, pp. 3186-3193.
- [37] N. Mohan, C. R. Mahesha, Shivarudraiah, N. Rajesh Mathivanan, and B. Shivamurthy, "Dry sliding wear behaviour of Ta/NbC filled glass-epoxy composites at elevated temperatures", *Procedia Engineering*, vol. 64, pp. 1166-1172, 2013.
- [38] S. Basavarajappa, K. Ellangovan, and V. Arun, "Studies on dry sliding wear behaviour of Graphite filled glass-epoxy composites", *Materials & Design*, vol. 30, no. 7, pp. 2670-2675, 2009.
- [39] P. Déprez, P. Hivart, J. F. Coutouly, and E. Debarre, "Friction and wear studies using taguchi method: application to the characterization of carbon-silicon carbide tribological couples of automotive water pump seals", *Advances in Materials Science and Engineering*, vol. 2009, Article ID 830476, pp. 1-10, 2009.
- [40] L. Dong, G. Geng, and M. Zhou, "Design of auto text categorization classifier based on boosting algorithm," *Computer Applications*, vol. 27, no. 2, pp. 384-386, 2007.
- [41] M. S. Hasan, A. Kordijazi, P. K. Rohatgi, and M. Nosonovsky, "Triboinformatics approach for friction and wear prediction of Al-Graphite composites using machine learning methods", *Journal of Tribology*, vol. 144, no. 1, p. 011701, 2024.
- [42] A. Sharma, P. Madhushri, V. Kushvaha, and A. Kumar, "Prediction of the fracture toughness of silicafilled epoxy composites using K-Nearest Neighbor (KNN) method", *2020 International Conference on Computational Performance Evaluation (ComPE)*, pp. 194-198, 2020.