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Multi-response optimization of wear parameters of flax reinforced epoxy composites using Taguchi-GRA-PCA approach

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Taguchi-grey relational analysis (GRA) coupled with principal component analysis (PCA) has been employed to optimize the multi-response wear characteristics of the trimethoxymethylsilane treated flax fibre reinforced epoxy (F-E) composites. Dry sliding wear test has been performed as per ASTM G99 based on the experimental schedule of Taguchi's orthogonal arrays (L_{27}). Wear responses selected for the current investigation are specific wear rate (SWR), coefficient of friction (COF) and shore D hardness. Analysis of variance (ANOVA) of GRG clearly reveals that sliding velocity is most significant factor affecting the wear performance of the F-E composites followed by fibre content and applied load. Field-emission scanning electron microscopy reveals that F-E composite endure thermal softening and cracks around its interfacial bonding region.

Keywords: Flax/epoxy composite, Multi-response optimization, Taguchi-GRA-PCA approach, Wear rate

1 Introduction

In current days, global warming has become the serious threat that is being faced by the environment and the world community as a whole. Therefore, to curb such menace, scientists have shifted their attention to environmentally friendly and recyclable materials instead of working with the synthetic materials. Researchers have started working on developing polymer composites reinforced with natural fibres as an alternative to synthetic fibre-based polymer composites as natural fibres own exceptional properties, like they are biodegradable in nature, consume less energy and low cost for fabricating their composites, environment friendliness, and nontoxicity[1,2]. Natural fibres are bifurcated into plant, animal, and mineral fibres. Amongst them, plant-based natural fibres are sought after as a reinforcement member in the automotive industry owing to their unique light weight and high fibre strength properties[3].

In this current research work, flax fibre is used as a reinforcement material, and like all other natural fibres, flax fibres are hydrophilic in nature due to the presence of pendant hydroxyl and polar groups in the components. Because of this, stress transfer between matrix and fibres gets deteriorated and thus mechanical properties of the composites reduce including their durability characteristics[4,5]. In this regard, several physical and chemical surface modification methods are practised with an intention to enhance the interfacial fibre-matrix bonding of composites. Some of the widely accepted chemical modification techniques are alkali treatment, silane treatment, acid treatment, peroxide treatment, etc. These chemical treatments introduce an additional third layer for enhancing the interfacial bonding of fibre and matrix [6].

It is essential to understand tribological properties of the natural fibre composites before placing in a tribological environment. In this connection, Gang[6] revealed that, chemical modification of the wood fibre by means of coupling agent has formed strong bonding between wood fibre and polyimide. Thus, wear performances of wood fibre reinforced polyimide composites are improved. Liu *et al.*[7] conveyed in their experimental study that mechanical and tribological properties of the corn stack composites has improved tremendously owing to the silane treatment of corn stack fibres, as treatment strengthen fibre-matrix bonding. Rajeshkumar[8] confirmed that the treatment of Phoenix sp. fibre with 15 % NaOH solution has improved the morphological and wear resistance properties of the fibre and its composites. Thus, these

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composites were suggested as friction materials for automobile applications. Behera *et al.*[9] showed that combined effect of alkali-glutamic acid treatment has improved crystallinity index of the sisal fibre. Besides, study also confirmed the improvement of wear, thermal, and micro hardness properties of the sisal fibre and its composites due to the effect of chemical alteration of the fibre. Gupta *et al.*[10] developed the hybrid sisal/jute reinforced epoxy composites and investigated for dry sliding wear characteristics. Experimental results showed that specific wear rate and coefficient of friction of the developed composites increased with increase in applied load and sliding speed. Wear performance of the composites improved as a result of reinforcement of chemically treated fibre into the epoxy matrix. According to authors suggestion, developed hybrid composite was employed for break pad application. Maurya *et al.*[11] reported the tribological properties of the short sisal fibre reinforced epoxy composites. The composites made up of fibre length 10 mm are prepared by hand layup method at the fibre loading of 30 wt.%. Tests were carried out on pin on disk machine using suitable operating parameters to study their influence on the wear characteristics. Result showed that the increase of factors such as load, distance and velocity, increased the SWR but decreased the coefficient of friction (COF) value of the composite. Kumar *et al.*[12] evaluated the dry and friction wear properties of the ramie woven reinforced epoxy composites. Wear result clearly showed that coefficient of friction of the ramie/epoxy composites increases with increase in the fibre loading and found higher than neat epoxy. Wear loss is also significantly affected due to the fibre reinforcement. Lower wear loss is reported by ramie/epoxy composites than neat epoxy. Wear loss and friction values of the composites are decreased with the increase in sliding speed. Fibre fracture, debonding, matrix breakage and fibre cutting were evident from SEM micrographs. Authors suggested that developed composite could be fit for making brake pads exclusively for automotive sectors. Chaudhary *et al.*[13] assessed tribological properties of the hybrid epoxy composites reinforced with jute/hemp/flax fibres (woven form). Three different combinations of the composites were developed and investigated their friction and wear characteristics on a pin on disc machine under pre-defined operating parameters. Result indicated that the incorporation of natural fibres in woven form into epoxy matrix has substantially improved wear performance of hybrid composites. Higher friction force, COF, and SWR were exhibited by jute/epoxy composites. Of the developed hybrid composites, hemp/epoxy composites presented better wear properties. Furthermore, study confirmed that tribological properties of the developed hybrid composites were significantly influenced by sliding speed followed by applied normal load. Rajini *et al.*[14] explored the dry sliding wear performance of the *Cyperus pangorei* fibre reinforced polyester composites under different sliding velocities and contact pressures. Results indicated that specific wear increased when applied load was increased. COF exhibited non-liner decrement for the combination of increase in contact pressure and decrease in sliding velocities. Pradhan *et al.*[15] for the first time explored the tribological property of the novel-short fibre eulaliopsis binata reinforced epoxy (SEB) composites. Different SEB/epoxy composites were developed by varying the weight fraction of the fibre loading (0, 10, 20, 30, and 40 wt.%). Dry sliding wear result confirmed that better value of COF and SWR was exhibited by 20 wt. % reinforced SEB/epoxy composites. That is, the higher fibre content beyond 20 wt.% does not favour the wear performance of the SEB/epoxy composites.

Indeed, it is very important to understand the effect of testing parameters on tribological behavior of the materials before placing in real time applications. Therefore, focus of the present research work is to explore the dry sliding performance of the trimethoxymethylsilane treated flax-epoxy composites using multi-response optimization technique, namely an hybrid approach of Taguchi based - Grey Relational Analysis (TGRA) integrated with Principal Component Analysis (PCA).

2 Materials and Methods

2.1 Materials

The epoxy resin grade VBR 8912 and hardener VBR1209 were procured from Vasavibala Resin Private Limited, Chennai, Tamil Nadu, India and then used as the matrix material. 6 LEA flax yarns were supplied by local vendors and weaved into irregular basket woven fabric (4×2) using table top handloom (Make: ERGO G2). Trimethoxymethylsilane, used for the chemical treatment of flax fibres, was supplied by Sigma Aldrich, Coimbatore, India.

2.1.1 Silane Treatment

The woven flax fabric was dipped in the silane solution, which was prepared by dissolving 0.1 % trimethoxymethylsilane in an acidified distilled water. The flax fabric was immersed in this silane solution at room temperature (28°C) for a duration of 60 min.

After that, silane treated flax fabric was kept inside the oven for drying at 65 ºC for 24 h.

2.2 Composite Preparation

Custom tailored irregular basket woven flax fabric and epoxy (with its hardener) were used as the reinforcement and matrix member respectively. For preparation of silane treated F-E composites, simple hand layup technique followed by curing of laminates in compression moulding machine method was employed. Initially, lower steel mould (dimension 250 mm × 250 mm × 4 mm) was coated with silicon spray over which teflon sheet was covered simply to facilitate for the easy removal of the cured laminates. At an outset, pre-calculated quantity of epoxy and resin were mixed thoroughly in the weight ratio of 1:2 and poured over the four layers of the flax fabric which was stacked one upon the other inside the lower mould. For ensuring evenly distribution of matrix material over the fabric, a steel roller approximately weighing 4 kg was employed. Further to improve the epoxy flow through the flax fabric, lower mould was closed with upper mould and entire mould unit was subjected to compression pressure (250 psi) in a compression moulding machine for 24 h. Once the curing was done, composites were post cured for another 12 h. Finally, prepared composite laminates were cut into appropriate size for various testing characterization. Totally four sets of silane treated F-E composites were fabricated by varying weight fraction of fibres (0, 25, 35, and 45 wt.%), which are denoted as neat epoxy, 25-TFE, 35-TFE, and 45-TFE composites respectively. Fabrication of the composites is illustrated in Fig. 1.

2.3 Testing Methods

2.3.1 Dry Sliding Wear Test

Silane treated F-E composites were subjected to sliding wear test on a pin on disc test rig (DUCOM Instrument Pvt Ltd.) as per ASTM G99. The composite specimens of dimension 6 mm × 6 mm × 3.5 mm were glued to the stainless-steel pin of diameter 6 mm which was mounted by means of specimen holder [Fig. 1(F)]. The specimen was brought in to the contact of the rotating stainless-steel disc (EN31 grade, 62 HRC, Ra 0.25-0.30 µm) running at a specific speed. Wear tests were performed for the selected control factors, as detailed in Table 1. In this test, mass of the F-E specimen before and after the wear was recorded using digital electronic balance (accuracy of 10^{-4} g). Wear loss (volume) in the form of weight loss was converted to the wear rate and specific wear rate by knowing density value of the specimens. Following equations were used to calculate the COF, wear rate and SWR:

Coefficient of friction (µ) = Frictional force (F_f) / Applied normal load (F_n) …(1)

Wear rate (WR), mm^3/m = $\Delta W / \rho L_d$ …(2)

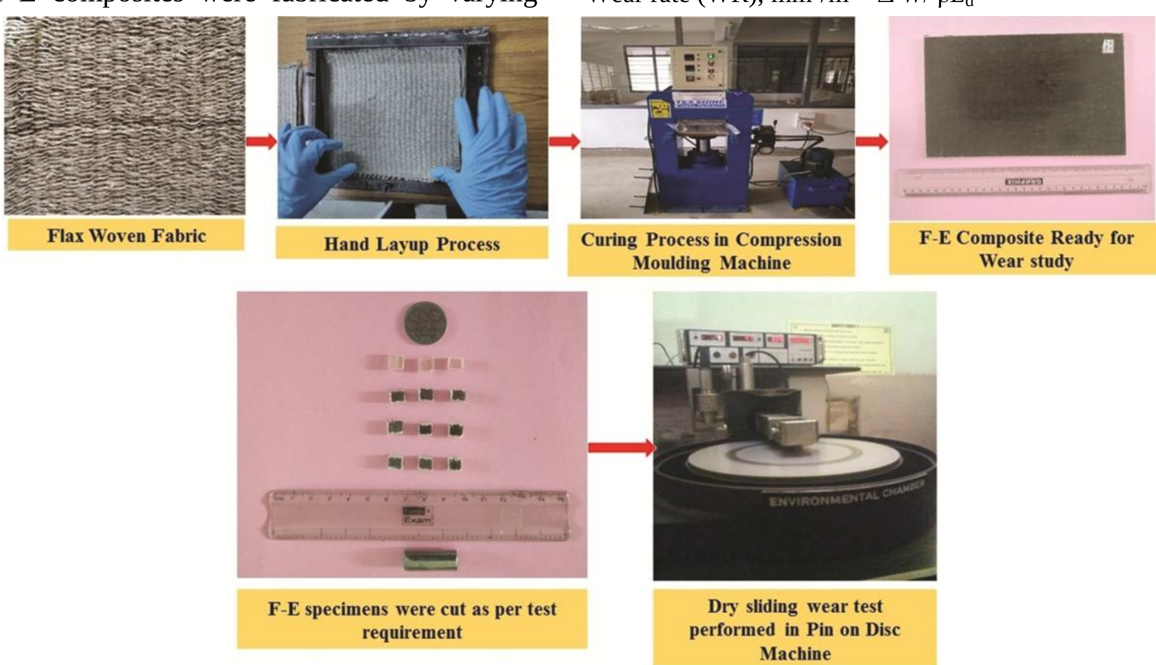

Fig.1 — Fabrication of silane treated F-E composites

Specific wear rate (SWR), mm^3/N-m= $\Delta W/\rho F_n L_d$ ...(3)

where ΔW is the weight loss of the specimen (g); ρ, the density of the composite (g/cc); F_f, the frictional force (N), F_n, the applied normal load (N); and L_d, the sliding distance covered by the specimen during wear test (m).

Table 1 — Test conditions for dry sliding wear

Parameters	Test condition
Composite sample	Neat epoxy, 25-TFE, 35-TFE and 45-TFE
Applied normal load, N	10, 15, 20 and 25
Sliding velocity, m/s	1, 2, and 3
Track diameter, mm	140
Sliding distance, m	1250

Table 2 — Control factors and their levels for dry sliding wear test of F-E composites

Control factors	Levels		
	1	2	3
Applied load (A), N	15	20	25
Sliding velocity (B), m/s	1	2	3
fibre content (C), wf%	25	35	45

2.3.2 Hybrid Approach for Multi-Response Optimization of Dry Sliding wear Property

In this present research work, experimental trails were designed based on Taguchi design of experiment and statistical hybrid approach of Grey Relational Analysis (GRA) is coupled with Principal Component Analysis (PCA) to optimize the multiple output response wear behavior of the silane treated F-E composites.

2.3.2.1 Taguchi Design of Experiment

Genchi Taguchi developed design of experiment (T-DOE) for designing the high quality of experiments through the set of few orthogonal arrays (OAs). The control factors and various level selected for dry adhesion wear under multipass condition is shown in Table 2. According to DOE, L_{27} orthogonal array (OAs) has been selected for the wear studies using MINITAB 16 software. Corresponding experiment plan pertains to wear mode under varying levels of their factors (Table 3).

Several researchers have implemented various statistical tools for the optimization of the wear

Table 3 — Experimental design for dry sliding wear using L_{27} OAs along with its output response characteristics

Experiment No	Load, N	Sliding velocity m/s	Fibre content wt.%	Response characteristics		
				Specific wear rate $\times 10^{-13}$ mm^3/N-m	COF	Shore-D hardness
1	15	1	25	0.8067	0.3320	76.20
2	15	1	35	0.7052	0.3167	81.31
3	15	1	45	0.5333	0.2933	85.23
4	15	2	25	1.3890	0.4333	76.20
5	15	2	35	1.1900	0.4133	81.31
6	15	2	45	0.9025	0.3933	85.23
7	15	3	25	4.3921	0.6227	76.20
8	15	3	35	3.4380	0.6147	81.31
9	15	3	45	2.5435	0.6000	85.23
10	20	1	25	0.6722	0.3425	76.20
11	20	1	35	0.5619	0.3100	81.31
12	20	1	45	0.4307	0.3000	85.23
13	20	2	25	1.1090	0.4300	76.20
14	20	2	35	0.9586	0.4150	81.31
15	20	2	45	0.8615	0.4000	85.23
16	20	3	25	6.1848	0.6550	76.20
17	20	3	35	3.6363	0.6425	81.31
18	20	3	45	2.7076	0.6100	85.23
19	25	1	25	0.5916	0.3800	76.20
20	25	1	35	0.4496	0.3680	81.31
21	25	1	45	0.3446	0.3600	85.23
22	25	2	25	0.9412	0.4480	76.20
23	25	2	35	0.8198	0.4400	81.31
24	25	2	45	0.7138	0.4360	85.23
25	25	3	25	5.3243	0.6488	76.20
26	25	3	35	3.9669	0.6480	81.31
27	25	3	45	2.4615	0.6360	85.23

COF – Coefficient of friction.

performance of the fabricated composites but Taguchi would be the top amongst all, as it is very helpful system for single objective optimization utilized for addressing various design issues. More importantly, Taguchi's DOE is a time saver and focused only on the few set of experimental data which would be essential and enough for analysing entire system. In simple words, it eliminates the unnecessary experiments, which is unrealistic to conduct, thereby saving the time. Furthermore, the optimization of the performance characteristics is accomplished all the way by following the sophisticated systematic approach starting from the data collection to its analysis. First step in the T-DOE technique is the conversion of the experimental data in to signal to noise ratio (S/N) which is classified as the higher the better (HB), the lower the better (LB) and the nominal the better (NB) as per the criterion of the individual characteristic responses selected. As far as the present wear is concerned, characteristic responses such as wear rate, SWR and COF, follow lower the better (LB), whereas hardness of the composites follows higher the better (HB) in order to find out their optimal wear performances. Criterion such as HB and LB which has been expressed in terms of loss function is computed by using following equations:

$$L_{HB} = \frac{1}{p}\sum_{i=1}^{n} \frac{1}{Y_{hrd}^2} \quad \ldots(4)$$

$$L_{LB} = \frac{1}{p}\sum_{i=1}^{n} \frac{1}{Y_{SWR}^2} \quad \ldots(5)$$

$$L_{HB} = \frac{1}{p}\sum_{i=1}^{n} \frac{1}{Y_{COF}^2} \quad \ldots(6)$$

where y^2_{hrd}, y^2_{swr} and y^2_{COF} represent the response for hardness, specific wear rate and coefficient of friction respectively; and 'p' denotes number of experiments.

In T-DOE, responses can be articulated in the form of S/N ratio by the help of logarithmic transformation, as shown in the following equations:

S/N ratio for (Shore-D) hardness = $-10 \log_{10} (L_{HB})$ ... (7)

S/N ratio for specific wear rate = $-10 \log_{10} (L_{LB})$ ... (8)

S/N ratio for coefficient of friction = $-10 \log_{10} (L_{LB})$ ...(9)

2.3.2.2 Grey Relational Analysis (GRA)

GRA was first created by Ju-long (1982) and the step-wise procedure involved in GRA-optimization has been discussed here. At an outset, GRA normalizes the experimental data between 0 to 1 and such process is called as grey relational generation. Following next step is to compute the grey relational coefficient (GRC) from the normalized data to arrive the interrelationship among the desired and actual experimental data. In GRA, individual experimental run (consist of the multi response factor) is converted to single response objective problem based on the grey relational grade (GRG), which is determined by considering the average GRCs of the multi response factors. Analysis of variance (ANOVA) is employed to the GRG data, so that significant factor affects the wear performance of the silane treated F-E composites. The following steps are involved in determination of the GRG:

(i) With respect to present investigation, grey relational generation related to the normalized data of WR/SWR and COF pertaining to LB criterion, as expressed in following equation:

$$Y_i(k) = \frac{\max Q_i(k) - Q_i(k)}{\max Q_i(k) - \min Q_i(k)} \quad \ldots(10)$$

HRD (Shore D hardness) should follow higher the better criterion, which can be determined by using following equation:

$$Y_i(k) = \frac{Q_i(k) - \min Q_i(k)}{\max Q_i(k) - \min Q_i(k)} \quad \ldots(11)$$

where $Q_i(k)$ is the obtained value; min Q_i, the minimum value for the k^{th} response; and max $Q_i(k)$, the maximum value for the k^{th} response.

(ii) To calculate the deviation sequence for all the corresponding process, following equation was used:

$$\Delta_{0i} = \left| Q_0(k) - Q_i(k) \right| \quad \ldots(12)$$

where, Q_o (k) and Q_i (k) are reference and comparability sequence respectively. Δ_{0i} is the deviation sequence of the aforesaid sequences.

(iii) GRC of the L_{27} experimental run is calculated using following equation:

$$\xi(k) = \frac{\Delta_{\min} + \beta\Delta_{\max}}{\Delta_{0i}(k) + \beta\Delta_{\max}} \quad \ldots(13)$$

where β is the identification coefficient value and it is considered as 0.5 when equal weightage is given to the process parameters.

(iv) In final step, calculation of GRG is carried out using following equation:

$$\psi_i(GRG) = \frac{1}{n_r}\sum_{k=1}^{n}\xi_i(k) \quad \ldots(14)$$

(v) PCA incorporated to assign the weight value of individual response characteristics, then GRG for coupled PCA will be calculated as per the Eq.(15). As discussed above, GRG is the summation of the GRCs and PCA, coupled with GRA so that determined weightage of each response characteristics is multiplied with GRC of the same response characteristics. The value of the GRG generally lies between 0 and 1. Larger GRG conveys better relation among the combination of the process parameter and this is also referred to as optimal level of combination. Equation (15) is given below:

$$\psi_i(GRG) = \sum_{k=1}^{n}\omega_k\xi_i(k) \quad \ldots(15)$$

where ψ_i represents the GRG of the ith experiment and n_r represents the number of the response characteristics selected for the study. In actual practical application, effect of each factor is not same. Therefore, Eq.(14) is modified to Eq.(15), where ω_k indicates the weighted value of the factor k which is computed from principal component analysis.

2.3.2.3 Principal Component Analysis (PCA)

PCA, a multivariate statistical tool, was first invented by Pearson in 1901 and then further modified by Hotelling in 1933. In the present work, PCA was carried out to find out the weightage of the individual response characteristics, pertaining to the tribological performance of the F-E composites.

The procedure involved in the PCA are as follows:

(i) First step in PCA is to construct the variance and covariance matrix 'J_i', normalized value used are as follows. In following equation, $J_i(x)$, i varied between 1 and *m*, *x* is varied between 1 and *n*:

$$J_i = \begin{bmatrix} J_1(1) & J_1(2) & \ldots\ldots & J_1(n) \\ J_2(1) & J_2(2) & \ldots\ldots & J_2(n) \\ \ldots\ldots & \ldots\ldots & \ldots\ldots & \ldots\ldots \\ \ldots\ldots & \ldots\ldots & \ldots\ldots & \ldots\ldots \\ J_m(1) & J_m(2) & \ldots\ldots & J_m(n) \end{bmatrix} \quad \ldots(16)$$

where *m* denotes number of the experimental run; n represents number of the response characteristics considered for the investigation; and *J* indicates the GRC of an individual performance characteristic. For the present studies on dry adhesion and abrasion wear, *m*=27, and *n* = 3.

(ii) The second step in PCA involves computation of correlation coefficient arrays which is computed by using following equation:

$$V_{xL} = \left(\frac{\mathrm{cov}(J_i(x), J_i(L))}{\sigma_{Ji}(x)\times\sigma_{Ji}(L)}\right) \quad \ldots(17)$$

where cov ($J_i(x)$, $J_i(L)$) are covariance of the sequence $J_i(x)$, and $J_i(L)$ respectively; and $\sigma_{Ji}(x)$ and $\sigma_{Ji}(L)$ denote the S.D. of the sequence $J_i(x)$, and $J_i(L)$ respectively.

(iii) Computation of the Eigen values and Eigen vectors using the correlation coefficient arrays with the help of following equation:

$$(V-\lambda_k I_m)P_{ik} = 0 \quad \ldots(18)$$

where λ_k denotes the Eigen values; $\sum_{k=1}^{n}\lambda_k = n$, where k=1, 2, 3, …………*n*; and P_{ik} represents $P_{ik} = [a_{k1}, a_{k2}, a_{k3}, \ldots\ldots\ldots a_{km}]^T$ Eigen vectors corresponding to Eigen values λ_k.

(iv) Thus, finally principal components are computed using the following equation:

$$E_{mk} = \sum_{i}^{n} J_m(i)\times P_{ik} \quad \ldots(19)$$

where E_{m1} represents as first component; E_{m2} as second principal components and so on.

Finally, confirmation test was performed to validate the experimental results obtained.

3 Results and Discussion

3.1 Effect of Load and Sliding Velocity on Wear Loss

Wear loss of the silane treated F-E composites as a function of applied load and sliding velocities is depicted in Fig. 2. Wear loss in terms of grams increases with increases in the applied loads and sliding velocities. However, neat epoxy resin shows higher wear loss and its resistance improved after it is reinforced with silane treated flax fibre. It is observed that wear loss gradually decreases with respect to

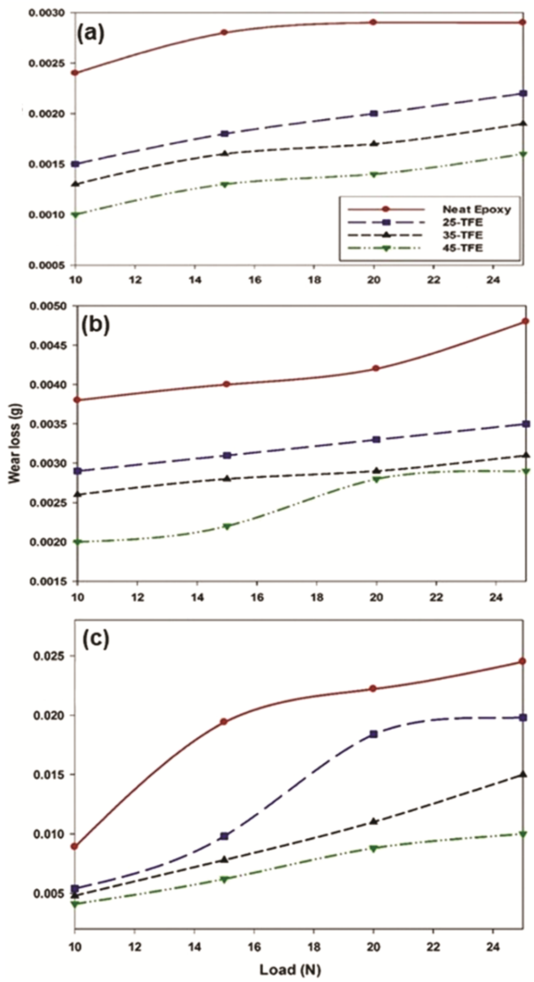

Fig. 2 — Variation in wear loss with respect to applied load for all developed F-E composites at (a) 1 m/s, (b) 2 m/s, and (c) 3 m/s sliding velocities

fibre content increases. Furthermore, wear loss of F-E composites increases with increases in the velocity. Of the fabricated composites, 45-TFE shows better resistance to the wear loss under tested condition.

From the Fig. 2, it is noticed that 45-TFE exhibits higher resistance to wear loss at a sliding speed of 1 m/s and 10 N applied load condition. This is ascribed to strong interfacial bonding of fibre-matrix and also because of the higher aerial density (578 g/m^2) of the irregular basket (4×2) woven fabric used[16]. About 58.3 % and 44.84 % improvement of wear resistance are noticed for 45-TFE as compared to that of neat epoxy composites at 10 N and 25 N respectively. As sliding speed increases, wear resistance decreases, which implies increase in wear loss. At 2 m/s, 45-TFE exhibits 47.36% and 39.58 % improved wear resistance than neat epoxy at 10N and 25 N respectively. Similarly, at 3 m/s, its resistance to wear is enhanced to 53.93% and 59.18 % at 10 N and 25 N respectively.

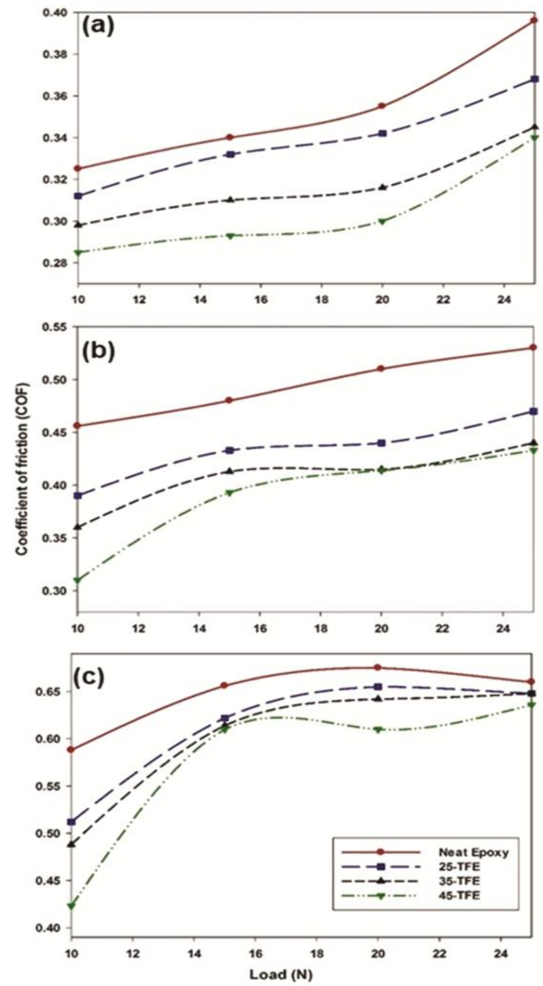

Fig. 3 — Variation of COF with respect to the applied load for all the developed F-E composites at (a)1 m/s, (b) 2 m/s and (c) 3 m/s sliding velocities

Increasing applied normal load increases wear loss. This may be due to the higher heat generation at the interface (disc and specimen), leading to softening of epoxy matrix[16]. As a result, the fibres of flax woven fabrics get pulled out and eventually F-E composites experience material removal. At higher load (25 N), both epoxy and silane treated F-E composites exhibit higher material removal, as the lubricated film formed on the worn surface is easily vulnerable for the detachment [16,17].

3.2 Effect of Load and Sliding Velocity on Coefficient of Friction

Coefficient of friction (COF) with respect to applied normal loads for the silane treated F-E composites at various sliding velocities is depicted in Fig. 3. Neat epoxy exhibits higher COF than F-E composites. Variation in COF strongly depends on the matrix film formation and strength of the fibre used. It is observed that the increase in load, increases the COF. This may be due to the thin film formation by the epoxy matrix phase[10]. At sliding velocity of 1 m/s, COF increases with increase in applied load, as depicted in Fig. 3. Also, increasing fibre content decreases the COF of the silane treated F-E composites. Compared to neat-epoxy composites, 45-TFE composites exhibit 12.14-14.41 % of friction improvement at 10 N and 25 N loading condition respectively. Lower coefficient of friction of 0.285 is found for 45-TFE at 10 N.

COF plotted against the normal load for the velocity of 2 m/s is shown in Fig. 3. Increasing the applied load increases the COF. Lower friction value of 0.31 is noted for 45-TFE composites at 10 N loading condition. Furthermore, compared to neat epoxy, silane treated F-E composites show lower COF for increasing load condition. Friction improvement from 32.5% to 35.5% is noticed between neat epoxy and 45-TFE composites at 10 N and 25 N respectively. Moreover, mere increment of friction value is exhibited by 35-TFE and 45-TFE composites when the load is increased from 20 N to 25 N. This may be attributed to the soaring interface temperature, that paves the way for the formation of partial thin plastic film between F-E specimen and counter face. Thus, it can be deduced that COF variation, whether it is increase, decrease, or constant, depends on the nature of fibre employed including fibre content (wt.%) and formation of thin polymer film.

At 3 m/s, variation of COF of the F-E composites and epoxy with respect to the varying load is shown in Fig. 3. In this sliding condition, friction value of the composites increases with increase in the applied load. Increasing of treated fibre loading in the F-E composites has improved their friction property. Improvement in COF exhibited by the F-E composites tested under 3 m/s sliding velocity has found to be inferior with those tested at 1 m/s and 2 m/s. Nearly 7-3% improvement in friction is noted between neat epoxy and 45-TFE, when load is increased from 15 N to 25 N, respectively. However, at lower load 10 N, it is increased to 28%. Neat epoxy shows higher COF than F-E composites at different sliding velocities. It implies that, the increase of velocity has increased the COF for both neat epoxy and F-E composites. Increasing COF for neat epoxy may be due to the fact that the rough film at the counter face increases the interlocking with asperities or fine wear particles when it contacts with specimen as a third body interaction. Hence, it could be accountable for upsurge in COF. Generally, with increase in applied load the contact pressure exerting at the F-E composite and steel disc interface also increases, causing the escalation of its interface temperature, which eventually results in increased frictional force[14,18]. However, it is also evident that the friction value is decreased in the F-E composites as fibre content increases. This may be due to the improvement in thermal resistance of the F-E composites, which restricts the material deformation at interface[14]. Besides, the presence of treated fibres produces smoother thin film with steel counterface, which could be the possible reason for the reduction of COF in case of 45-TFE composites.

3.3 Effect of Load and Sliding Velocity on Specific Wear Rate

Figure 4 presents the specific wear rate (SWR) of the neat epoxy and silane treated F-E composites at

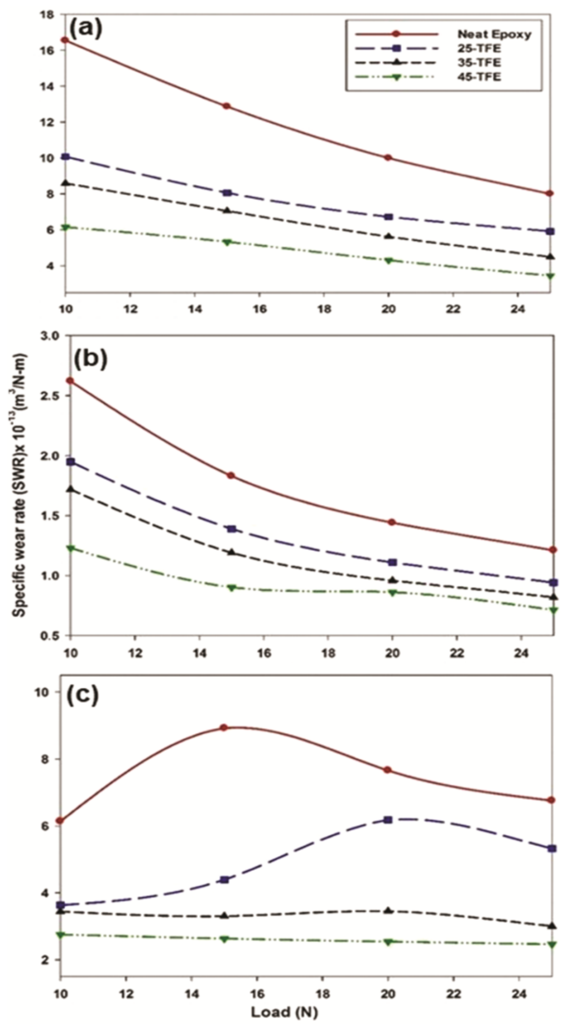

Fig. 4 — Variation of SWR with respect to applied load for all developed F-E composites at (a) 1 m/s, (b) 2 m/s and (c) 3 m/s sliding velocities

different applied loads (N) under varying siding velocities (m/s). Increasing the normal load has decreased the SWR of the epoxy and F-E composites. Further increase in the sliding velocity increases SWR in the F-E composites. Figure 4 (a) shows that drastic decrease in the SWR of the epoxy, as the reinforcement of chemically treated flax fibre content increases. It means that the woven flax fabric improves the wear resistance of the F-E composites. This may be due to the fact that the alkali-silane treatment improves the interfacial adhesion between matrix and fibre, which has increased the load bearing capacity of the fibre and eventually protects the resinous region of the composites[10]. SWR of the neat epoxy is reduced by 62.5 % as compared to that of 45-TFE at 25 N load condition. At other loading conditions, 38-45% reduction in SWR is noted at sliding velocity of 1m/s. Decrease in SWR of the neat epoxy may be due to the high brittle nature of the epoxy. Besides, it is also important to note that the combining effect of velocity and load increases the temperature at the interface, as it enhances the softening process, which further intensifies material removal rate and hinders the thin film formation at the counterface[19].

Figure 4 shows the effect of applied normal load on SWR of neat epoxy and silane treated F-E composites at 2 m/s. SWR at 2m/s of the aforesaid materials is found similar to that of 1m/s. However, the increase in SWR is noticed in all the materials, as velocity is increased from 1 m/s to 2 m/s. Almost 36% increase in the SWR is noted for neat epoxy, whereas 50 % surge is observed for F-E composite at 10 N loading condition. This seems to be applicable for all applied loading conditions. As the load increases, SWR decreases and this trend is more significant at 1 m/s and 2 m/s sliding velocity condition. However, at higher load, due to the higher pressure at the interface (composites specimen) and steel disc, there is a high chance for the thin film transfer debris to get transferred back to F-E composite's surface, which may contribute to lower SWR. It is very interesting to note that SWR of the neat epoxy and treated F-E tested under varying applied loads at 3 m/s is found to be higher than that of SWR determined at 1 m/s and 2 m/s. This may be owing to the fact that higher velocity and load combination increases the temperature, which enhances the softening process of matrix, resulting in higher material removal from the sliding surface of the sample. However, reinforcement of the flax woven fibre in to the epoxy phase improves the wear resistance of the composites. The interfacial adhesion property of the flax fibre has improved as result of chemical treatment, which may reduce the fibres to get pulled out during sliding action. Besides, load bearing capacity of the fibre improves and thus protects the resinous region of the composites, thereby enhancing the wear resistance property of the F-E.

3.4 Multi-Response Optimization of Dry Sliding Wear Property – A Hybrid Statistical Approach

In the current research, control factors, namely applied normal (N), sliding velocity (m/s), and fibre content (wt.%) have been considered as the prime control factors, affecting the wear process. Obviously, specific wear rate ($\times 10^{-13}$, mm^3/N-m) and coefficient of friction (COF) are supposed to be the output responses of wear experiment. However, literature work suggested that, wear performance of the composites is significantly affected by the hardness of the material. Therefore, hardness property of the silane treated F-E composites is considered in the present statistical analysis. Moreover, aim of the present study is to minimize the wear rate and COF and maximizing the hardness of the F-E composites. Based on the Taguchi's factorial design approach, for three factors each at three levels with accordance to design of OAs, L_{27} has been selected to establish the process inter relationships. According to that, output responses of the wear such as specific wear rate (SWR), COF and hardness of the F-E composites are presented in Table 3

Firstly, normalization of each response characteristics is carried out using Eqs (10) and (11), followed by the determination of the deviation sequence for the normalized data using Eq. (12). Normalized and deviation sequence values of each output response characteristics is shown in Table 4. The GRC values are computed for the deviation data using Eq.(13). Table 5 shows the Eigen value and explained variation of principal components for dry sliding wear test.

To the T-GRA analysis, principal component analysis (PCA) is introduced in order to determine the weighted value of each performance characteristics. Calculated Eigen vectors and contribution of individual performance characteristics for the wear behavior of the F-E composites is shown in Table 6. GRG values are computed by introducing the weighted value of each response characteristics using Eq.(15). GRG is a very important function in this statistical analysis. Because it is a powerful tool to convert the multiple

Table 4 — Normalized, deviation and GRC data of the dry sliding wear response characteristics

Experiment No	Normalized value			Deviation sequence			Grey relational coefficient (GRC)		
	Specific wear rate mm^3/N-m	COF	Shore-D hardness	Specific wear rate mm^3/N-m	COF	Shore-D hardness	Specific wear rate mm^3/N-m	COF	Shore-D hardness
1	0.9209	0.8930	0.0000	0.0791	0.1070	1.0000	0.8901	0.8237	0.3333
2	0.9383	0.9354	0.5659	0.0617	0.0646	0.4341	0.9393	0.8856	0.5353
3	0.9677	0.9999	1.0000	0.0323	0.0001	0.0000	0.7366	0.9998	1.0000
4	0.8212	0.6128	0.0000	0.1788	0.3872	1.0000	0.7755	0.5636	0.3333
5	0.8552	0.6681	0.5659	0.1448	0.3319	0.4341	0.8396	0.6011	0.5353
6	0.9045	0.7234	1.0000	0.0955	0.2766	0.0000	0.4191	0.6439	1.0000
7	0.3070	0.0894	0.0000	0.6930	0.9106	1.0000	0.4856	0.3545	0.3333
8	0.4703	0.1115	0.5659	0.5297	0.8885	0.4341	0.5704	0.3601	0.5353
9	0.6235	0.1521	1.0000	0.3765	0.8479	0.0000	0.8991	0.3709	1.0000
10	0.9439	0.8640	0.0000	0.0561	0.1360	1.0000	0.9307	0.7861	0.3333
11	0.9628	0.9538	0.5659	0.0372	0.0462	0.4341	0.9714	0.9155	0.5353
12	0.9853	0.9815	1.0000	0.0147	0.0185	0.0000	0.7925	0.9643	1.0000
13	0.8691	0.6221	0.0000	0.1309	0.3779	1.0000	0.8263	0.5695	0.3333
14	0.8949	0.6635	0.5659	0.1051	0.3365	0.4341	0.8496	0.5978	0.5353
15	0.9115	0.7050	1.0000	0.0885	0.2950	0.0000	0.3333	0.6289	1.0000
16	0.0000	0.0000	0.0000	1.0000	1.0000	1.0000	0.4701	0.3333	0.3333
17	0.4364	0.0346	0.5659	0.5636	0.9654	0.4341	0.5527	0.3412	0.5353
18	0.5954	0.1244	1.0000	0.4046	0.8756	0.0000	0.9220	0.3635	1.0000
19	0.9577	0.7603	0.0000	0.0423	0.2397	1.0000	0.9653	0.6759	0.3333
20	0.9820	0.7935	0.5659	0.0180	0.2065	0.4341	1.0000	0.7077	0.5353
21	1.0000	0.8156	1.0000	0.0000	0.1844	0.0000	0.8304	0.7306	1.0000
22	0.8979	0.5723	0.0000	0.1021	0.4277	1.0000	0.8600	0.5390	0.3333
23	0.9186	0.5944	0.5659	0.0814	0.4056	0.4341	0.8878	0.5521	0.5353
24	0.9368	0.6055	1.0000	0.0632	0.3945	0.0000	0.3696	0.5590	1.0000
25	0.1473	0.0171	0.0000	0.8527	0.9829	1.0000	0.4463	0.3372	0.3333
26	0.3798	0.0194	0.5659	0.6202	0.9806	0.4341	0.5797	0.3377	0.5353
27	0.6375	0.0525	1.0000	0.3625	0.9475	0.0000	1.0000	0.3454	1.0000

Table 5 — Eigen value and explained variation for dry sliding wear test

Principal component	Eigen value	Explained variation, %
PC1	1.9678	65.59
PC2	0.9471	31.57
PC3	0.0851	2.836

quality characteristics of the system in to single representative and rank is assigned for GRG from higher level to lower level. Table 7 shows the higher value of GRG; first rank has been assigned for the experiment number 11 (GRG-0.3054). Higher values of GRG declares that experimental values are in close agreement with the ideal ones.

In other words, combinations of the control parameters have approached closer to optimal condition. From the GRG (Table 7), it can be concluded that experiment number 11 gives the multiple response of lowest value of SWR and COF and highest hardness for the wear behavior of silane treated F-E composites.

Influence of GRG on the selected control factors is shown in Fig. 5. Wear responses in terms of GRG for dry sliding wear test is detailed in Table 8, from where the best combination of the levels of input parameter with respect to the highest value of GRG is given as A1 (15 N), B1(1 m/s), and C3 (45 wt.% of fibre). These factors are turned out to be as a best optimal combination for the multiple response characteristics. From the response table of GRG, it is also evident that higher value of GRG is found for sliding velocity followed by fibre content and applied load. It simply implies that, sliding velocity plays a

Table 6 — Eigen vectors and contribution of the individual response characteristics for the principal components under dry sliding wear test

Output response characteristics	Eigen vectors			Contributed/weighted value
	PC1	PC2	PC3	
Specific wear rate	0.695	0.083	-0.714	0.483
COF	0.699	0.291	0.684	0.4886
Shore-D hardness	-0.265	0.953	-0.147	0.0702

pivotal role towards the adhesive wear performance of the F-E composites.

3.4.1 Analysis of Variance (ANOVA) for Dry Sliding Wear

ANOVA tells about the significant contribution of the individual control factors for optimizing the wear performance of the system. ANOVA for GRG has been determined using MINITAB (V.16) and values are tabulated in Table 9, where it is evident that sliding velocity has influenced the wear behavior of the composites by 95.5 % followed by fibre content (4.1%) and applied load (N) (0.5%). The last step in this statistical analysis is conformation test. After identifying the optimal wear factors, validation of the experimental results is carried out through confirmation test. It can be executed using predicted GRG Eq.(14). After conducting the confirmation test, it is apparent that predicated (0.4425) and experimental (0.4663) values are in close agreement (95%), which implies that T-GRA integrated with PCA has been successfully implemented for multi-response optimization of wear behavior of the silane treated F-E composites under dry sliding wear condition.

Table 7 — GRG value and its rank for dry sliding wear test of F-E composites

Exp. No	Grey relational grade (GRG)	Rank
1	0.2739	7
2	0.2958	2
3	0.2911	4
4	0.2167	18
5	0.2373	12
6	0.1869	19
7	0.1388	25
8	0.158	22
9	0.2235	17
10	0.2749	6
11	**0.3054**	**1**
12	0.2948	3
13	0.2257	16
14	0.2384	10
15	0.1708	20
16	0.1332	26
17	0.1524	24
18	0.226	15
19	0.264	9
20	0.279	5
21	0.266	8
22	0.2266	14
23	0.2378	11
24	0.1663	21
25	0.1299	27
26	0.1562	23
27	0.2359	13

3.5 Worn Morphology of F-E composites

Figures 6 (a) – (d) show the FESEM images of the silane treated F-E composites, which are worn out for the test conducted at optimal input combination. With increase in the sliding velocity, the wear rate of F-E is increased and results in uneven surfaces**.** The formation of uneven surface may be due to the severe damage of the composite layers. Moreover, it is interesting to note that the composites may have experienced higher wear rate with increase in the sliding velocities owing to the detachment of flax fibre as a result of poor interfacial bonding between fibre and matrix. From the wear analysis it is clear that, when F-E composites are subjected to dry sliding wear at higher load and sliding velocity, temperature at the sliding interface is increased, which results in higher wear rate. Due to this process, thermal

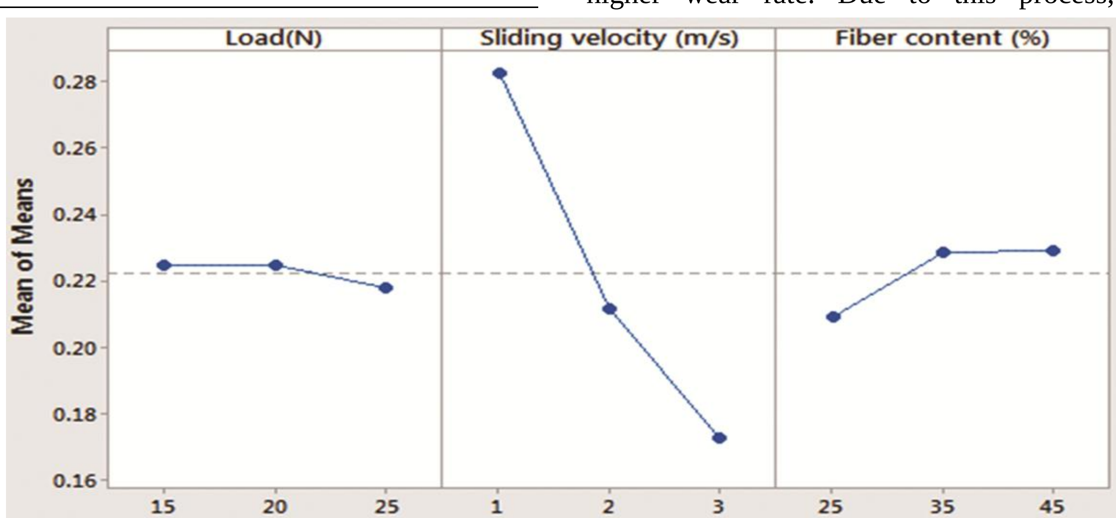

Fig. 5 — Influence of GRG on selected control factors

Table 8 — Response for GRG under dry sliding wear

Symbol	Control factors	Grey relational grade (GRG)			Main effect (Max-Min)	Rank
		Level-1	Level-2	Level-3		
A	Applied normal load, N	**0.2248**	0.2247	0.218	0.0067	3
B	Sliding velocity, m/s	**0.2828**	0.2119	0.1727	**0.1101**	1
C	Fibre content, wt.%	0.2094	0.229	**0.2291**	0.0197	2

Table 9 — ANOVA of GRG for dry sliding wear

Source	DF	Adj SS	Adj MS	F-value	P-value	Contribution, %
Applied load, N	2	0.00027	0.00013	0.13	0.88	0.46
Sliding velocity, m/s	2	0.05609	0.02804	26.97	0	95.5
Fibre content, wt.%	2	0.00232	0.00116	1.12	0.347	4.12
Total	26	0.07948				100

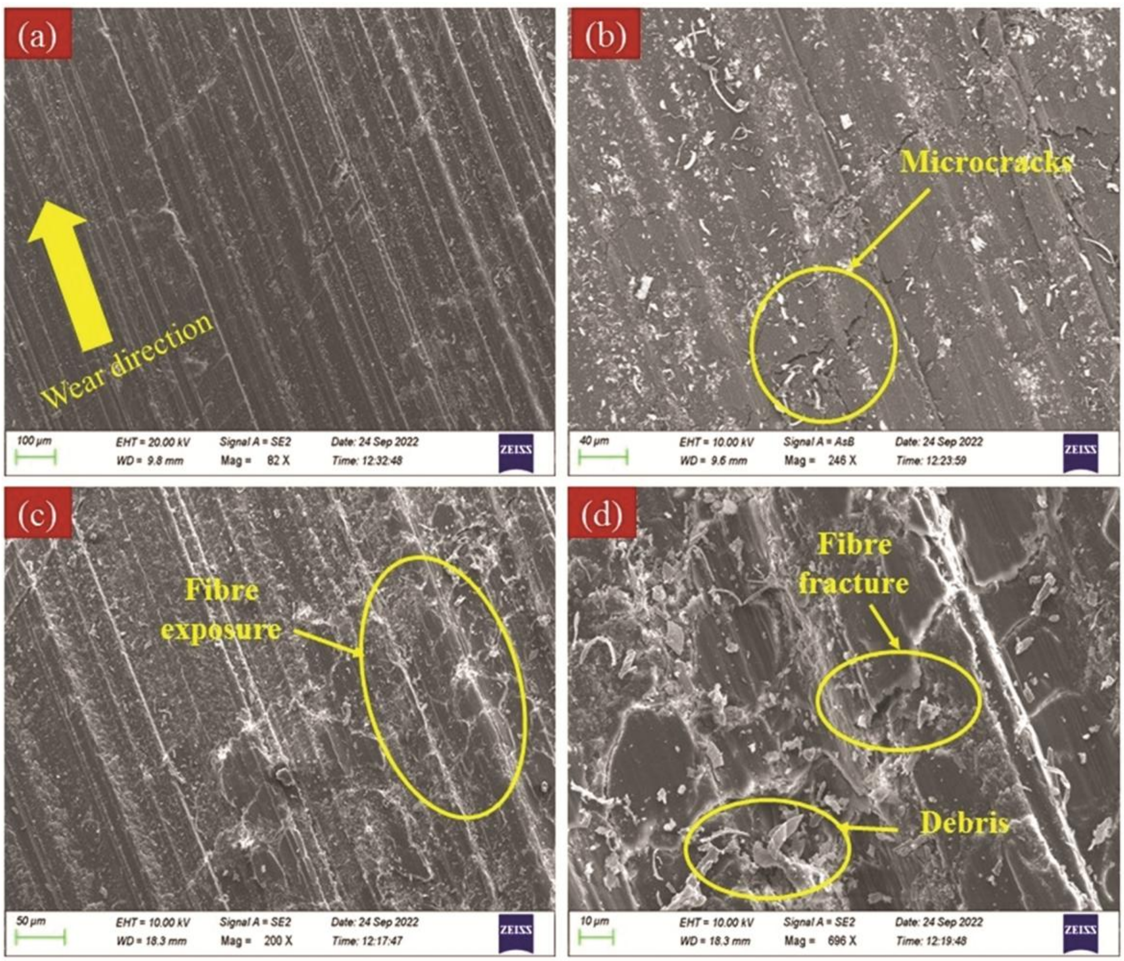

Fig. 6 — Worn out FESEM images of the silane treated F-E composites at optimal input parametric condition

softening or crack occurs at the fibre-matrix, which could be the main reason for the poor wear behaviour of the F-E composites, as indicated in Fig.6(d).

4 Conclusion

The major outcomes of this research work have been summarized below:

4.1 Wear rate of the F-E composites improves as fibre content increases. This may be owing to the improvement in load bearing capacity of the composites, as chemical treatment enhances the interfacial bonding of fibre-matrix. Increase in applied load and sliding velocity has increased the wear loss and COF of the composites. 45-TFE composite shows lowest COF (0.27) sliding at 1 m/s sliding velocity at 10 N loading condition.

4.2 SWR of the F-E composites decreases with increase in the applied load, which is attributed to the transfer of the wear debris back on to the composite surface because of the intense pressure at the composite-disc interface at higher loading. Reinforcement of silane treated woven fabric found to be very effective in reducing the wear rate of the composites. However, increase in the sliding velocity from 1 m/s to 3 m/s has increased the SWR of the F-E composites. This may be owing to the fact that higher velocity and load combination raise temperature, which enhances the softening process of matrix, resulting in a higher material removal from the sliding surface of the sample

4.3 Hybrid optimization technique effectively optimized the sliding wear parameters with multiple response attributes. Optimum combination of wear process parameters responsible for higher hardness of F-E composites to exhibit minimum COF and SWR, is found at A1 B1 C3 combinations (15 N load, 1 m/s sliding velocity, and 45wt.% fibre content).

4.4 ANOVA results indicate that sliding velocity is the most influenced factor which significantly contributes on the wear performance of the silane treated F-E composites followed by fibre content and applied load. Amongst the fabricated F-E composites, 45-TFE shows minimum wear rate at optimized wear test condition.

4.5 Confirmation test shows that predicated and experimental values are in close range and hence test conducted by adopting hybrid optimization technique may found to be satisfactory. However, the prime reason of microcracks and matrix erosion of the F-E composite is attributed to thermal softening as exposed by FESEM.

References

1 Nayak S & Mohanty J, *Compos Commun*, 18 (2020) 19. doi:10.1016/j.coco.2020.01.006.

2 Shettahalli Mantaiah V K, *J Nat Fibers*,19 (2022) 12415. doi: 10.1080/15440478.2022.2060404

3 Nirmal U, Hashim J& Ahmad M M, *Tribol Int*, 83 (2015) 77. doi:10.1016/j.triboint.2014.11.003.

4 Fiore V, Di Bella G & Valenza A, *Compos [Part B]: Engg*, 68 (2015) 14. doi: 10.1016/j.compositesb.2014.08.025.

5 Hassaini L, Kaci M, Touati N, Pillin I, Kervoelen A & Bruzaud S, *J Nat Fibers*, 23 (2018) 123. 1doi.org/10.1080/15440478.2018.1550461.

6 Gang D, *Surf Interface Anal*, 50 (2018) 304. doi.org/10.1002/sia.6369.

7 Liu Y, Xie J, Wu N, Wang L, Ma Y & Tong J, *Tribol Int*, 131 (2019) 398. doi.org/10.1016/j.triboint.2018.11.004.

8 Rajesh Kumar G, *J Ind Text*, 51 (2020) 2819S. doi: 10.1177/1528083720918948

9 Behera S, Gautam R K, Mohan S & Chattopadhyay A, *J Nat Fibers*, 19 (2021) 6134. doi: 10.1080/15440478.2021.1904483

10 Gupta M K & Srivastava R, *Am J Polym Sci Eng*, 3 (2015) 198.

11 Maurya H O, Jha K & Tyagi Y, *Poly Poly Compos*, 25 (2017) 215. doi.org/10.1177/096739111702500306.

12 Kumar R & Anand A, *Mater Res Express*, 6 (2018) 015309. doi.org/10.1088/2053-1591/aae69b.

13 Chaudhary V, Bajpai P K & Maheshwari S, *Fiber Polym*, 19 (2018) 403. doi.org/10.1007/s12221-018-7759-6.

14 Rajini N, Mayandi K, Siengchin S, Ayrilmis N, Bennet C & Ismail S O, *J Nat Fibers*, 18 (2021) 261. doi.org/10.1080/15440478.2019.1621232.

15 Pradhan S, Acharya S K & Prakash V, *J Appl Polym Sci*, 138 (2021) 50077.doi.org/10.1002/app.50077

16 Rashid B, Leman Z, Jawaid M, Ghazali M, Ishak M & Abdelgnei M, *Wear*, 380 (2017) 26. doi.org/10.1016/j.wear.2017.03.011.

17 Nirmal U, Hashim J & Low K, *Tribol Int*, 47 (2012) 122. doi.org/10.1016/j.triboint.2011.10.012

18 Ahmed K S, Khalid S S, Mallinatha V & Kumar S A, *Mater Design*, 36 (2012) 306. doi.org/10.1016/j.matdes.2011.11.010.

19 Yousif B & El-Tayeb N, *Surf Rev Lett*, 14 (6) (2007) 1095. doi.org/10.1142/S0218625X07010561.