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RESEARCH

Wear Behaviour of Sansevieria and Carbon Fiber Reinforced Epoxy with Nanofillers: Taguchi Method

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A B S T R A C T

Sliding wear behaviour of combined chemically modified Sansevieria and Carbon fiber reinforced epoxy (TSria/CF-E) and its multi-phase nanocomposite coupons has been carried out by Taguchi design of experiments. A Taguchi L16 orthogonal array and ANOVA were used to examine the consequence of tribo-system parameters on the tribological behaviour of hybrid TSria/CF-E composites filled with nano-$CaCO_3$ and nano-SiC. The specific wear rate was calculated for each experiment for nano-$CaCO_3$ and nano-SiC multi-phase composites. The normal load was found to have the highest significant influence of 42.15 % on the SWR of the nano-$CaCO_3$ composite, whereas in case of the nano-SiC filled composites, the filler loading was found to have the highest significance of 62.34 % on the SWR. The improvement in the wear resistance was attributed to high strength, high hardness, and good dispersion of nano-SiC particles, which can enhance the interfacial bonding of nanofiller and the epoxy matrix. The lowest specific wear rate of composite was 0.06 × 10^{-4} mm^3/N m. Examination of the worn micrographs of the confirmation experiment shown adhesive and abrasive wear as the prevailing mechanisms.

1. INTRODUCTION

Plant fibers-reinforced polymer composites (PFRPCs) has risen as a possible eco-friendly and sensibly evaluated alternative to artificial fibers-strengthened composites. Unlike the conventional synthetic fibers (glass, carbon and aramid) lignocellulose fibers can give certain advantages to the composites, for example, low density, higher modulus, low cost, renewability, biodegradability and high level of flexibility during fabrication [1]. Cellulosic fibers namely sisal, coir, flax, and bamboo in their characteristic structure just as a few waste cellulosic items, for example, coconut shell flour, wood flour and pulp have been utilized as fortifying operators of various thermosetting and thermoplastic composites [2]. Materials demand and environmental impact of herbal fibers, their utilization has been restricted to non-bearing applications because of their poor strength and stiffness in contrast with synthetic fiber reinforced composites [3]. The poor mechanical properties of bio-composites can be overcome by way of modifying the polymer with partial synthetic fibers and nanofillers. During the closing few years, a collection of works have been performed to replace the conventional artificial fiber with natural fiber-reinforced composites [4]. For instant, hemp, sisal, jute, broom, cotton and flax are the most frequently used fibers to improve polymers like polyolefins, unsaturated polyester (USP), polystyrene, and epoxy resins. Also, fibers similar to jute, sisal, coir, bamboo, oil palm, flax straw and wheat, banana and waste silk [5] have demonstrated as promising reinforcements in the thermoset/ thermoplastics. Nevertheless, certain elements of natural fiber strengthened composite behaviour nevertheless scantily unstated such as their interfacial adhesion and tribological properties. Modest data regarding the tribological properties of natural fiber strengthened composite has been expressed. In this circumstance, long plant fibers, like flax, hemp [6] and bamboo have good sized viable in the manufacture of echo-friendly bio-based composite materials.

Mylsamy et al. [7] studied agave-fiber-reinforced epoxy composites by varying the fiber length (3, 5, and 7 mm). The incorporation of 5 mm fiber length, because of higher aspect ratio, improved the wear resistance in agave-fiber/epoxy composites. Yousif et al. [8] used betelnut fiber to increase the wear resistance and reduce the friction coefficient of unsaturated polyester. Shao et al. [9] prepared epoxy filled TiO_2-SiO_2 composites and subjected to adhesive wear tests. Results indicated that the TiO_2-SiO_2 particles had improved resistance to wear of the composites. Law et al. [10] investigated recycled carbon fiber and epoxy, bought as two varieties, cryogenic treated and as received condition. Results indicated wear resistance improved and coefficient of friction decreased in the cryogenic treated carbon fiber in the epoxy matrix. Shakuntala et al. [11] analyzed wear characteristics of carbon black and raw wood apple shell particles crammed in epoxy. Results showed the lower wear rate for carbon black particulate composites. Vijay et al. [12] selected locally available plant fibers to study the tribological behaviour of PFRPCs. They used three varieties of plant fibers (flax, jute, and hemp) be reinforced in epoxy to produce hybrid composites using hand layup method. Inclusion of natural fibers in epoxy enhanced the wear properties of the prepared PFRPCs in comparison to pristine epoxy. From the prepared composites, jute/epoxy composite showed the maximum friction coefficient and specific wear rate.

In addition, the influence of lower wt.% of E-glass fiber on wear behaviour of sansevieria-polyester hybrid composites by Munisamy et al. [13]. The findings showed that the polyester with 30 wt.% sansevieria fiber and 20 wt.% glass fiber composite showed better wear resistance. Qing et al. [14] studied the friction and wear behaviour of short carbon fiber-reinforced epoxy composites with varying nano-SiO_2, and observed that 6 wt.% carbon fiber and 4 wt.% nano-SiO_2 has superior wear properties. Suresha et al. [15] observed that alkali treated coir fiber and silanated aramid fiber with coconut shell particles in vinyl ester influence the friction and wear behaviour of composites. Hemalata et al. [16] investigated solid particle erosion wear behavior of bamboo fibre reinforced epoxy composites filled with cenosphere particles. Final results indicated that increase in the loading of cenosphere decreased the erosion rate of the composite. Yousif et al. [17] investigated kenaf fiber-reinforced epoxy composites for bearing application, and observed better wear behavior of composite in the normal orientation of fibers. Bajpai et al. [18] incorporated three different types of natural fibers (nettle, grewia optiva, and sisal) into polylactic acid. The results indicate that the addition of natural fiber mats into PLA matrix substantially improved the wear resistance. Atiqah et al. [19] studied the friction and wear behavior of coir fiber-reinforced composites with varying fiber volume fraction, and observed that 5% volume fraction of fiber has optimum physico-mechanical as well as wear properties.

The design of experiments (DoE) by Taguchi method has been effectively utilized by specialists in investigation of the wear behaviour of composites materials. The DoE provides an orderly way to collect, analyze, and interpret data to satisfy the scope of the study. Sahin et al. [20] analyzed wear performance of polytetrafluoroethylene (PTFE) with glass filled composites and carbon filled composites using Taguchi's L9 orthogonal array. The results indicated that increasing load, grit size, and sliding distance, the specific wear rate decreased. Sandep et al. [21] studied epoxy bolstered novel bast fiber and sisal fiber composites and characterized for adhesive wear properties as per Taguchi's L16 array. Results highlighted SWR was influenced by means of the fiber content basically followed with sliding distance, normal load and sliding velocity. Amar et al. [22] studied adhesive wear characteristics of polyester reinforced with flakes of pine-bark stuffed with kiln-dust by Taguchi method. Results indicated that specific wear rate is very much impacted by few control factors. Chandra et al. [23] analyzed wear characteristics for untreated and treated dust coir filled with epoxy matrix by adopting design of experiments approach. Influence of untreated and treated coir dust quantity by 10, 20, 30 by wt.%, loads being 10, 20, 30 N and sliding velocities are 3, 4, 5 m/s on the specific wear rate.

Likewise, Sansevieria (Sria) fibers may also have considerable potential as reinforcement in thermosetting polymers to provide benefits when used as a substitute for glass fiber. To the author best knowledge, no work yet has been reported in the literature on the tribological behaviour of Sria fiber reinforced epoxy with nano-fillers loading. It is thought-provoking to know how the composite performance varies with the combination of natural fiber with partial synthetic fiber and nanofillers under various experimental conditions.

The utilization of nanoparticles in polymers for improvement of the wear properties is drawing more and more consideration because of their exceptional properties resulting from the nano-scale structures. The incredibly high specific surface area is one of the most appealing attributes of nanoparticles on the grounds that it expedites in creating an interphase in a composite, and thus a strong interaction between the fillers and the matrix results even at rather low nanofiller loading [24]. Fillers influence the strength of the composite according to their padding characteristics, size and interfacial bonding [25]. At the point when a molecule size is decreased to a nano-scale, the wear behaviour of composites may be significantly different from that of micrometer-sized filler filled systems [26]. Concerning the tribological performance, the utilization of nanoparticles is accepted to have two points of interest: (i) the abrasiveness of hard nanoparticles diminishes astoundingly because of the reduction in angularity in contrast with microparticles and (ii) the amount of material removed in nanocomposites will be significantly less than that of conventional composites on the grounds that the fillers are of the same size as the segments of the surrounding polymer chains [27, 28]. At present, different scientists have reported that the incorporation of inorganic nanoparticles such as Al_2O_3, TiO_2, ZnO, ZrO_2, SiC, SiO_2, and $CaCO_3$ in polyphenylene sulphide, polyetheretherketone, epoxy, PMMA, and PTFE matrices has improve nanocomposites wear performance [29-35]. The use of hard and abrasive nanoparticle fillers in polymers has been found to have mixed results on wearing.

Various scientists have contemplated the impact of nano-$CaCO_3$ on mechanical and tribological properties of polymer based nanocomposites. Applying the nano-$CaCO_3$ in polymers improved tensile, flexural, and impact strengths [36-39]. Zhang et al. [40] utilized nano-$CaCO_3$ to increase the wear resistance of polyalphaolefin. The results showed that nano-$CaCO_3$ can dramatically improve wear resistance and reduce friction. The consolidation of nano-$CaCO_3$ particles, because of their crystal nucleation and lubrication effects, improved the wear resistance in PA6-$CaCO_3$ nanocomposites as revealed by Mohsenzadeh et al. [41].

Silicon carbide (SiC) is one such ceramic material that can possibly be utilized as filler in different polymer matrices. Silicon carbide is made of tetrahedral carbon and silicon atoms with strong bonds in the precious stone cross section. This delivers exceptionally hard and solid material. It is not assaulted by any acids, soluble bases, or liquid salts up to 800 ^{0}C. The high thermal conductivity combined with low coefficient of thermal expansion and high strength gives this material exceptional thermal shock resistant properties. Silicon carbide has low density of about 3.1 g cm^{-3}, low thermal expansion, high elastic modulus, high strength, high thermal conductivity, high hardness, excellent thermal shock resistance, and superior chemical inertness [42,43].

The inclusion of nanofillers to thermosetting polymer matrix composites has considerable effect on its wear behaviour. Nano-sized calcium carbonate (nano-$CaCO_3$) and nano-SiC can be effectively used as potential fillers in fiber-reinforced composites. Although the literature is rich with studies on the mechanical properties of nanofiller filled epoxy composites, no analysis has been reported on the dry sliding wear behaviour of nano-$CaCO_3$ and nano-SiC filled with hybrid fiber-reinforced epoxy composites. This study investigates epoxy wear of hybrid composites in which chemically treated unidirectional Sria and carbon fibers are used as hybrid fiber reinforcement, and nano-$CaCO_3$ and nano-SiC and secondary fillers. The fabricated composite coupons were subjected to adhesive wear tests as per ASTM G99-17 [44]. The specific wear rate (SWR) for each composite with varying fiber length and fiber orientation mutually with other tribo-parameters (i.e., load, speed, and sliding distance) had been introduced and compared. The motive to utilize Sria fiber as reinforcement is due to its abundant availability, low cost, fast growth rate, low density and low energy consumption in fabrication, and biodegradability.

2. EXPERIMENT DESCRIPTION

2.1 Test Materials

In the present work, unidirectional sansevieria (Sria) plant fibers were collected from a farm near Annur, Tamil Nadu, India, by using a mechanical process called decortication. The obtained plant fibers are weaved in mat form such that all fibers are arranged unidirectional. Unidirectional carbon fibers (mat-form, CFs) were purchased from Master Micron International, Bengaluru, India. Nano-sized calcium carbonate ($CaCO_3$) and silicon carbide (SiC) fillers were purchased from Global nanotech, Kolkata, India. The epoxy resin (Lapox-12) with an amine hardener (K6), supplied by Yuje Enterprises, Bengaluru, India.

2.2 Chemical Treatment of Sansevieria Fiber

Sansevieria (Sria) plant fibers were collected from a farm near Annur and the extracted fibers were hand washed and dried at room temperature for 72 h.

To enhance the interfacial adhesion between fiber and matrix, Sria fibers were subjected to alkali treatment [45]. The cleaned fibers were soaked in 4.5 wt.% NaOH solution for 24 h at room temperature, then washed with distilled water and dried in an air circulated oven at 40 ^{0}C for 24 h. Later, these fibers were neutralized in 0.2 wt.% dilute acetic acid solution followed by thoroughly washing with distilled water. The neutralized fibers were then dried in an oven at 40 °C for 6 h. Further, the NaOH treated Sria fibers had been subjected to potassium permanganate treatment. The Sria fibers had been soaked in 0.5 wt.% potassium permanganate for 45 min [46]. Then the fibers have been washed with distilled water and dried in an air circulated oven at 40˚C for 6 h [46,47].

2.3 Specimen Preparation

Figure 1 display the pictorial diagram of processability of nanofiller filled TSria/CF-Ep composites through step by step hand lay-up method followed by compression moulding using hot press. Chemically treated UD Sria fiber mat and UD CF mat of 300 mm × 300 mm size were utilized to prepare the final composite slabs. A stainless steel mould with dimensions of 350 × 350 × 25 mm^3 was used. Initially, fiber mats were placed in a mould where a thin layer of releasing agent (silicon spray) was used to facilitate easy removal of the composite slab form the mould after curing. The composite laminate comprises of three layers in which CF mats are situated at the top and bottom of Sria mat. The preforms of fiber mats are manufactured through spreading the necessary amount of epoxy and hardener mix over the stacked mats and rollers are utilized to disperse the resin mix and to remove the air bubbles entrapped.

Similarly the hybrid composite slabs have been prepared using the same process by incorporating the required quantity of nanoparticles (nano-$CaCO_3$ and nano-SiC) in the epoxy resin and mixed uniformly using a high speed mechanical mixer.

Finally, the stacked composite was cured in a hydraulic press for 24 h before it removed from the mould. The cast of each composite slab was post cured in the air for another 24 h after removing out from the mould. Coupons for wear testing were cut using a diamond tipped cutter.

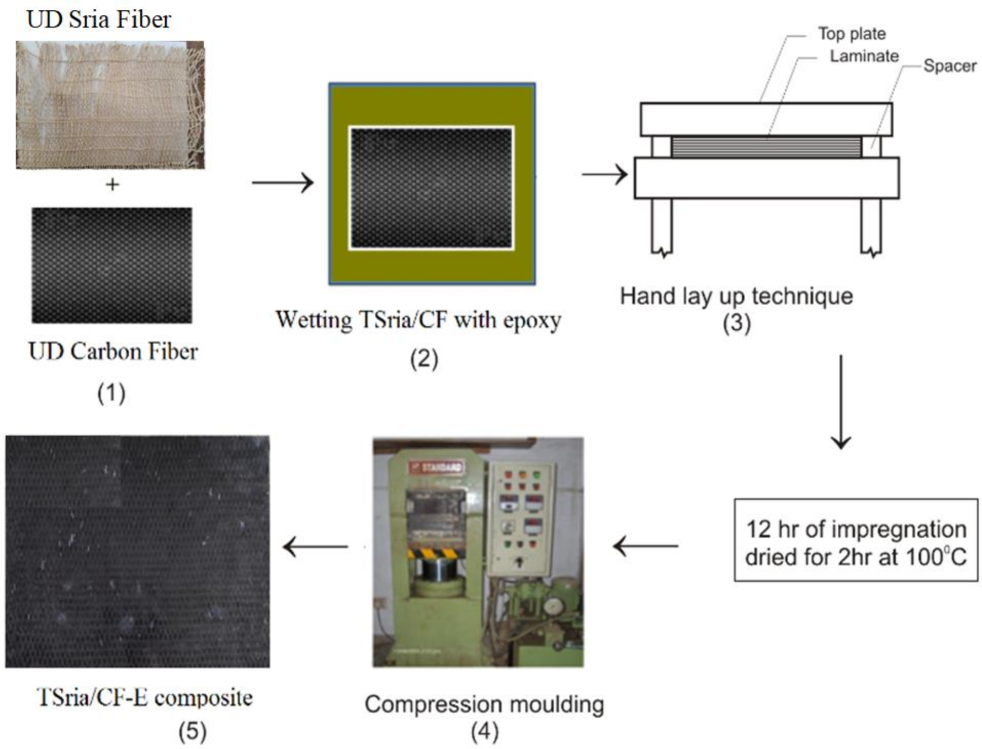


Fig. 1. Hand layup followed by compression moulding.

Table 1. Sria based hybrid epoxy composites and their codes.

Sl. No.	Description					Sample Code
	Epoxy (wt. %)	Treated Sria (wt. %)	Carbon fiber (wt. %)	Nano-$CaCO_3$ (wt. %)	Nano-SiC (wt. %)	
1	65	30	5	--	--	TSria/CF-E
2	63.5	30	5	1.5	--	H1
3	62	30	5	3.0	--	H2
4	60.5	30	5	4.5	--	H3
5	63.5	30	5	--	1.5	H4
6	62	30	5	--	3.0	H5
7	60.5	30	5	--	4.5	H6

2.4 Wear testing

Adhesive wear tests of prepared composite slabs are carried out using pin-on-disk (PoD) test rig, manufactured by Magnum Engineers, Bangalore, India. The counter disk is made of ground hardened steel, (EN-32) with 64 Rockwell hardness number. The surface roughness of disc was maintained at 0.5 Ra, for all wear tests. Before each test, the steel disc was polished with 1200 grit water proof SiC emery paper. Then the steel disc is cleaned with acetone followed by exposure to air. The composite coupons (6.5 mm × 6. 5 mm × 4.5 mm) with parallel fiber orientation were slid against the steel counter surface. Wear test is performed as per ASTM G99-17 standard [44]. The tribo-test parameters include; sliding distance, applied normal load, and sliding velocity. The specific wear rate (**K_s**) (m^3/Nm) [44], which values the wear performance of was deduced using the relation (1):

$$K_s = \frac{\Delta m}{F_n \times \rho \times D} \tag{1}$$

where, **Δm** is the loss of mass (g), **ρ** is the density in g m^{-3} and **D** is the sliding distance in m, **F_n** is the applied normal load (N).

2.5 Full factorial design approach

An orthogonal array and analysis of variance (ANOVA) was implemented to investigate the effects of tribo-parameters on specific wear rate (SWR) of all the composites. The full factorial Taguchi design of experiments eradicates the necessity for repeated experiments and in turn saves the material, time, as well as cost. The full factorial experimental plan with orthogonal array L_{16} was chosen and the experimental design includes three wear-testing conditions, such as applied normal load (NL), sliding velocity (SV) and filler loading (FL). The codes and levels of control parameters are however summarized in Tables 2 and 3. Each combination of experiments was repeated thrice, so as to acquire more accurate end results. In the Taguchi approach, the experimental data obtained are transformed into signal-to-noise (S/N) ratio and there exists several available S/N ratios, depending on the type of characteristics considered.

Table 2. Experimental parameters.

Test parameter	Levels			
	I	II	III	IV
Filler Loading, FL (wt.%)	0	1.5	3	4.5
Normal Load, NL (N)	20	40	60	80
Sliding Velocity, SV (m/s)	0.5	1	1.5	2

Table 3. L_{16} Orthogonal arrays.

Exp. No.	FL (wt. %)	NL (N)	SV (m/s)
1	0	20	0.5
2	0	40	1
3	0	60	1.5
4	0	80	2
5	1.5	20	1
6	1.5	40	0.5
7	1.5	60	2
8	1.5	80	1.5
9	3	20	1.5
10	3	40	2
11	3	60	0.5
12	3	80	1
13	4.5	20	2
14	4.5	40	1.5
15	4.5	60	1
16	4.5	80	0.5

3. RESULTS AND DISCUSSION

3.1 Analysis of wear results of nano-$CaCO_3$ filled hybrid composites

Table 4 lists the SWR of the nano-$CaCO_3$ filled TSria/CF-E composites. The test data in Table 4 are related to a fixed sliding distance of 4 km and wear control parameters are as per Table 3. From Table 4, the overall mean for the S/N ratio of the SWR was found to be 87.534 dB. Fig. 2 displays graphically the effect of the three control factors on SWR. The analysis was made using the most popular software specifically used for design of experimental applications known as MINITAB R17.

Table 4. Specific wear rate of nano-$CaCO_3$/ nano-SiC filled TSria/CF-E composites.

Exp. number	FL (wt. %)	NL (N)	SV (m/s)	$CaCO_3$		SiC	
				SWR x 10^{-4} (mm^3/Nm)	S/N Ratio (dB)	SWR x 10^{-4} (mm^3/Nm)	S/N Ratio (dB)
1	0	20	0.5	1.220	78.2728	1.220	78.2728
2	0	40	1	1.100	79.1721	1.100	79.1721
3	0	60	1.5	0.880	81.1103	0.880	81.1103
4	0	80	2	0.864	81.2697	0.864	81.2697
5	1.5	20	1	1.350	77.3933	0.910	80.8191
6	1.5	40	0.5	0.650	83.7417	0.530	85.5144
7	1.5	60	2	0.820	81.7237	0.550	85.1927
8	1.5	80	1.5	0.600	84.4370	0.445	87.0328
9	3	20	1.5	1.100	79.1721	0.480	86.3751
10	3	40	2	0.950	80.4455	0.420	87.5350
11	3	60	0.5	0.450	86.9357	0.170	95.3910
12	3	80	1	0.46	86.7448	0.150	96.4781
13	4.5	20	2	1.100	79.1721	0.530	85.5144
14	4.5	40	1.5	0.485	86.2852	0.265	91.5350
15	4.5	60	1	0.250	92.0412	0.180	94.8945
16	4.5	80	0.5	0.150	96.4782	0.060	104.437

Mean of SWR as a function of FL, NL, as well as SV of nano-$CaCO_3$ filled TSria/CF-E composites coupons are plotted in Fig. 2. Observations from these results revealed that mean of SWR decreases with increasing nano-$CaCO_3$ loading and NL. However, the marginal increase in SWR with increase in the SV was found for nano-$CaCO_3$ filled TSria/CF-E composites. Through Taguchi analysis, it was found that among all the considered wear parameters, the greatest impact on the SWR has the NL, and then the FL. The optimization of the wear parameters as per the selected factors and levels, with respect to the criterion smaller is better, results in the combination of control factors namely; NL, FL and SV, as can be seen from Fig. 2.

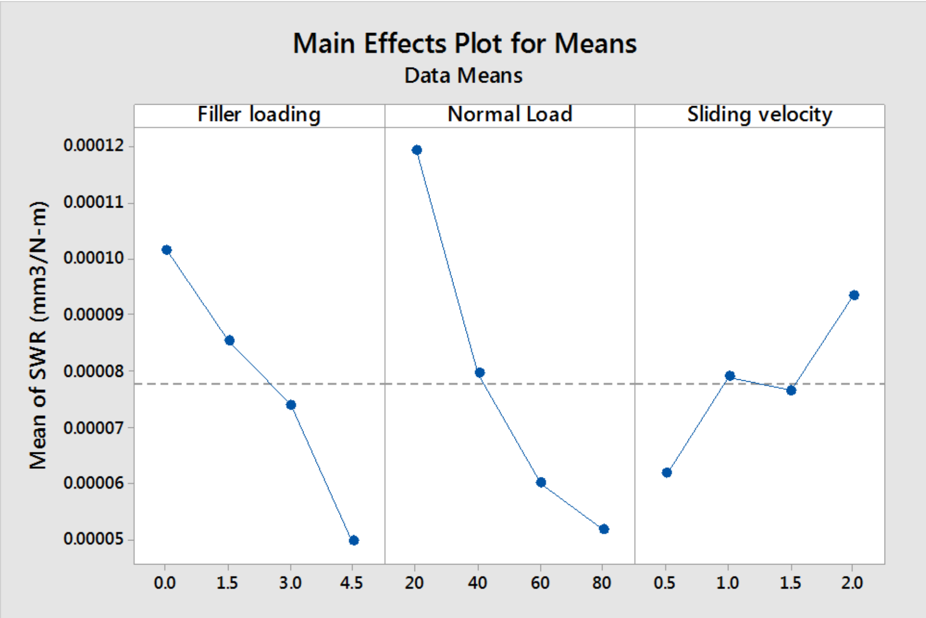


Fig. 2. Mean of SWR for nano-$CaCO_3$ filled TSria/CF-E composites.

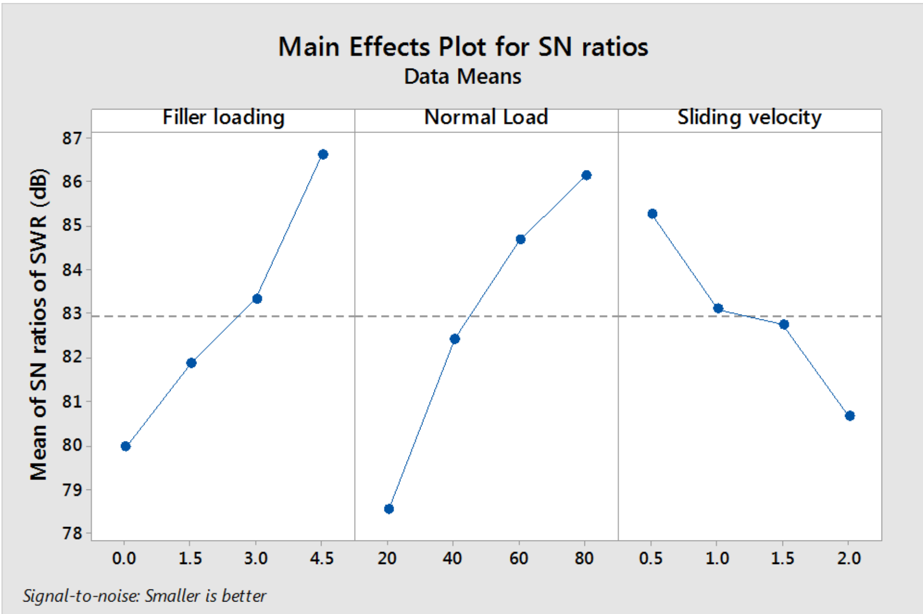


Fig. 3. Effect of control factors of nano-$CaCO_3$ filled TSria/CF-E composites.

The SWR decreases sharply when the NL is increased from 20 N to 40 N (Fig. 2); and thereafter it decreases steadily. However, with an increment in NL, from 40 N to 80 N, the SWR decreases on the ground that the area of contact between the coupon and the rotating disc are significantly increased at higher loads. Due to amplified contact surface, it allows maximum particles to come across the interface and takes the stress [48]. This prompts a steady state or decrease in the SWR, especially in the NL of 60 to 80 N. The SWR firmly relies upon the NL for all the tested composite materials. The effect of NL has more grounded impact on the wear behaviour of PTFE composites than the SV. In the present study, the SWR values are in agreement with the literature [49].

Next prevailing factor on the wear behaviour of composites is FL. A less decreasing trend in SWR was observed with increasing FL from 1.5 to 3 wt.%. However, it decreases sharply with changing the FL from 3 to 4.5 wt.%. Whereas, with an increment in filler stacking from 1.5 to 4.5 wt.%, there is a decrease in the SWR because as the nano-$CaCO_3$ stacking builds, the quantity of nano-$CaCO_3$ filler particles in the transfer film additionally increments, and it causes the interruption of transfer film because of the more number of hard particles [50].

Last factor which affects the wear behaviour of composite materials is SV. With increase in SV from 0.5 to 1 m/s and 1.5 to 2 m/ s, there is a marginal increase in SWR; while from 1 to 1.5 m/s SV, there is a slight reduction in the SWR.

Experimental perceptions are changed into signal to noise ratio (S/N= μ) using the equation:

$$\mu = -10\log_{10}\left(\sum_{i=0}^{n} y_i^2\right) \quad (2)$$

The μ ratio is determined using the equation 2 for each of 16 experiments and are tabulated in Table 4. Analysis of the result (Fig. 3) leads to the conclusion that the control factor combination of FL (4.5), NL (80) and SV (0.5), contributes to minimum SWR. The factors FL and NL have greater contribution on output performance, and the control factor SV has the least effect on the SWR. From this analysis, we can conclude that factors like FL and NL have a significant effect on SWR and the SV has the least effect on SWR (Table 4). It can also be seen from Table 4 that the least and highest S/N ratios are estimated at 77.39 and 96.48 dB, corresponding to test runs 5 and run 16, respectively.

3.2 Analysis of variance (ANOVA)

The results from ANOVA for the adhesive wear behaviour of hybrid TSria/CF-E composites are calculated from S/N ratios of SWR (Table 4 and Fig. 3) and summarized in Table 5. The percentage contribution of control parameters on the SWR of $CaCO_3$ filled TSria/CF-E hybrid composites is illustrated in Fig. 4.

ANOVA was carried out for a level of significance of 10 %, which is, involving a level of confidence of 90 %. Table 5 reveals that factors FL, NL, and SV has statistical and physical importance on the SWR. As shown in the column P (%) of ANOVA table, P of each individual factor on overall variations shows the level of influence on the results of wear. It is seen that the factor FL (p = 38.73 %), factor NL (p = 42.15 %), and factor SV (p = 16.24 %) have the effects on the sliding SWR, but the factor SV showed very less effect on SWR. The error seen from ANOVA table for the mean S/N ratio is approximately 2.88 %.

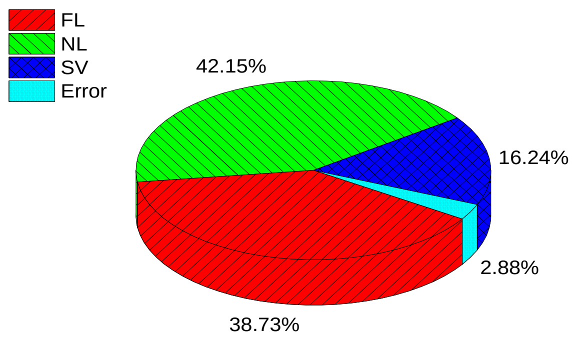


Fig. 4. Contribution of control parameters on the SWR of $CaCO_3$ filled TSria/CF-E composites.

The correlation of R2 (adj.) was 92.82 %. It is realized that that smaller the p-value, more prominent the criticality of the factor interaction relating to it. The table of ANOVA for S/N ratio showed that the filer loading (p= 0.001), normal load (p=0.001), and sliding velocity (p = 0.007) in this order the noteworthy control factors are effecting the SWR. It means that the normal load is the major factor, followed by the filler loading and sliding velocity.

3.3 Linear regression analysis

Linear regression analysis was carried out in order to ascertain the correlation significant factors determined by the ANOVA analysis which were NL and FL. Relations of linear regression model with the experimental values are shown in Fig. 5.

Table 5. ANOVA table of S/N ratio of SWR of nano-$CaCO_3$ filled TSria/CF-E composites.

Source	DF	Adj SS	Adj MS	F-Value	P-Value	P (%)	Rank
FL	3	161.20	53.73	26.97	0.001	38.73	**2**
NL	3	175.45	58.48	29.35	0.001	42.15	**1**
SV	3	67.62	22.53	11.31	0.007	16.24	**3**
Error	6	11.96	1.99	-	-	02.88	-
Total	15	416.23	-	-	-	100	-

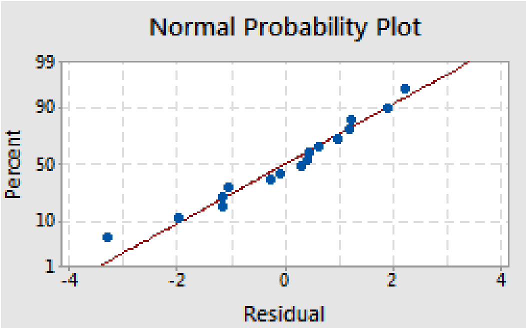
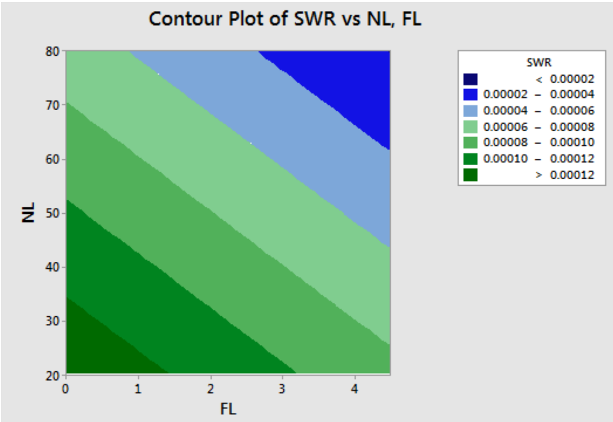
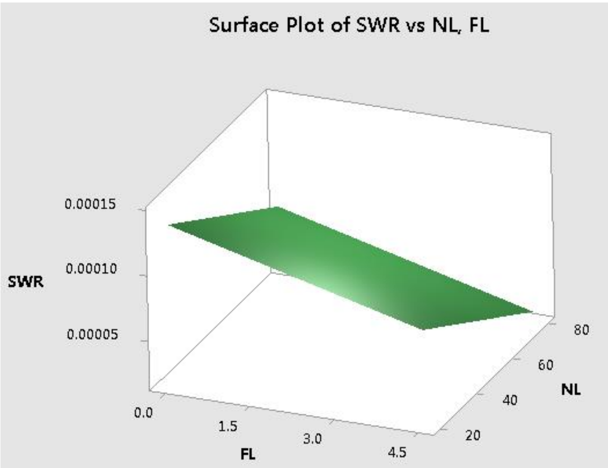


Fig. 5. Relation of linear regression model with the experimental values.



(a)

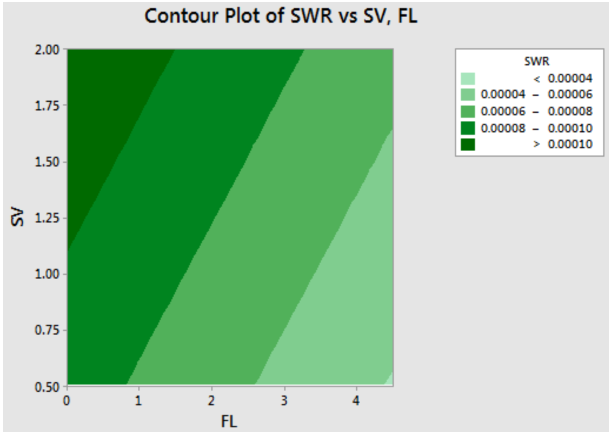


(b)

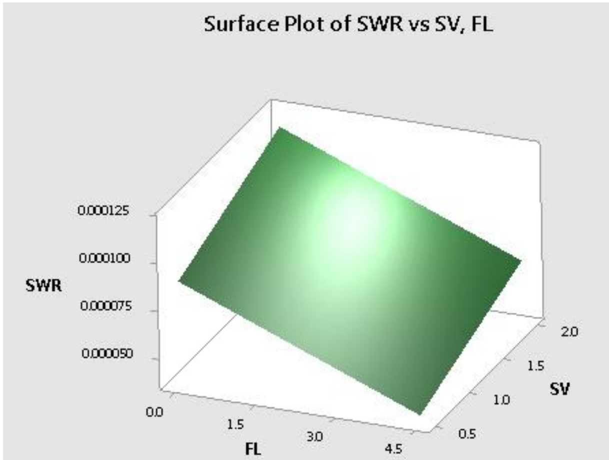
Fig. 6. Dependence of SWR on normal load and filler loading (a) contour plot (b) surface plot.

Figure 6a depicts the contour plot of the SWR for NL and FL for nano-$CaCO_3$ filled TSria/CF-E composites. Curves suggest the existence of interaction between NL and FL. Conversely, in Fig. 6b, the surface plot of SWR depending on the combination of factors NL and FL. Based on the analysis and graphs, minimal SWR is obtained with the highest load (80 N) and the highest filler loading (4.5wt.%$CaCO_3$), which means that the impact of highest load in composite material with higher $CaCO_3$ content have great impact. Its influence demonstrates with higher load values, i.e., 60 N and 80 N.

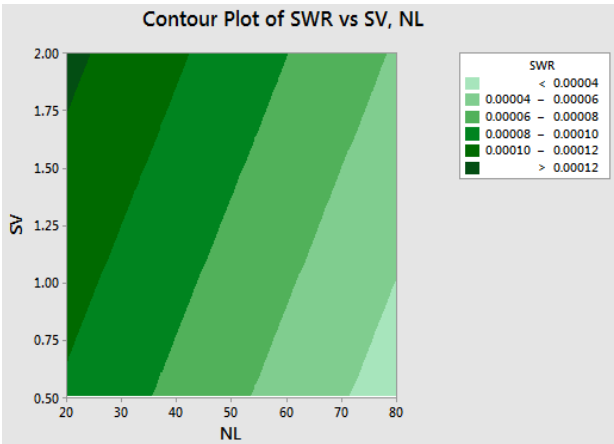
Figure 7a presents the contour plot of the SWR for FL and SV for nano-$CaCO_3$ filled TSria/CF-E composites. Curves suggest the presence of interaction between FL and SV. Conversely, in Fig. 7b, the surface plot of SWR depending on combination of factors namely NL and SV.



(a)



(b)



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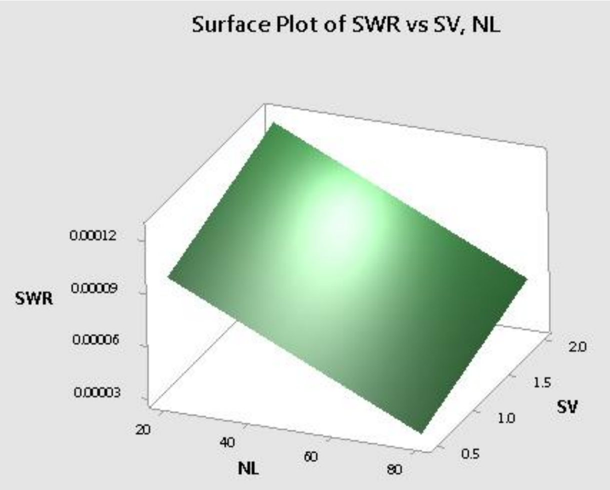


(d)

Fig. 7. Contour and surface plots of SWR:
(a) dependence on filler loading and sliding velocity
(b) dependence on normal load and sliding velocity.

On the basis of the conducted analysis and given graphs, minimal values of SWR are achieved with the highest load (80 N) in the considered range of SV (0.5, 1, 1.5, and 2 m/s). This suggests that the impact of SV for the composites with maximum load does not have great impact. Furthermore, the SWR gradually upsurges to 20 N when the SV vary from 1.5 m/s to 2 m/s.

Multiple linear regression model is realized using the software "Minitab R16". This model gives the ratio between the input and output by setting the regression equation [51,52]. A regression equation generated establishes the relationship between significant conditions obtained from the ANOVA analysis, i.e. FL, NL, and SV. The regression equation developed for *SWR* of nano-$CaCO_3$ filled TSria/CF-E composites is as follows:

$$SWR = 0.000135 - 0.000011 \times FL - 0.000001 \times NL + 0.000018 \times SV \quad (3)$$

From equation (3), it is seen that NL play a major role on minimum SWR and it is highly influenced followed by FL.

3.4 Confirmation test

The finishing step in Taguchi based design of experiments is to check the improvement of the quality characteristic utilizing the optimal levels of design parameters (FL4.5 NL80 SV0.5). The S/N ratio is determined using the equation (4).

$$\hat{\eta} = \eta_m + \sum_{i=0}^{n} (\eta_i - \eta_m) \quad (4)$$

where η_i, the mean S/N ratio of the result at the optimum level **n** is the number of design parameters and η_m is the total of mean S/N ratio.

For nano-$CaCO_3$/nano-SiC filled TSria/CF-E composites, confirmation experiments were conducted by utilizing the levels of the optimal process parameters (FL4.5, NL80, and SV0.5). The results of experimental confirmation using optimal wear parameters and comparison of the predicted SWR with the experimental SWR are listed in Table 6.

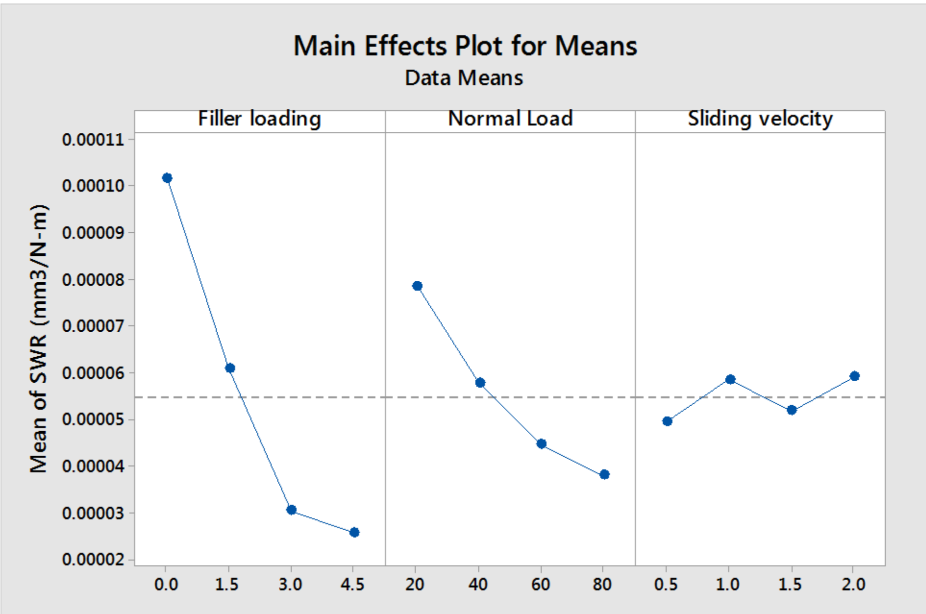


Fig. 8. Mean of SWR for nano-SiC filled TSria/CF-E composites.

Table 6. Confirmation test data for SWR of nano-$CaCO_3$ and nano-SiC TSria/CF-E composites.

$CaCO_3$ filled	**Estimation**	**Experimental value**	**Error (%)**
Level	NL80FL4.5SV0.5	NL80FL4.5SV0.5	
SWR (mm^3/N m)	0.15	0.144	4
SiC filled	**Estimation**	**Experimental value**	**Error (%)**
Level	NL80FL4.5SV0.5	NL80FL4.5SV0.5	
SWR (mm^3/N m)	0.06	0.058	3.3

Good agreement seems to take place between the estimated and experimental SWR. The SWR is decreased by 4 % and 3.3 % for nano-$CaCO_3$ and nano-SiC TSria/CF-E composites respectively. Thus the wear performance (reciprocal of SWR) is prominently improved by using Taguchi design of experiments.

3.5 Analysis of wear results of nano-SiC filled hybrid composites

Table 4 lists the SWR of the nano-SiC filled TSria/CF-E composites. The test data in Table 4 are related to a fixed sliding distance of 4 km and wear control parameters are as per Table 3. From Table 4, the overall mean for the S/N ratio of the SWR was found to be 104.437 dB. Fig. 8 displays graphically the effect of the three control factors on SWR.

Mean of SWR as a function of FL, NL, as well as SV of nano-SiC filled TSria/CF-E composites coupons are plotted in Fig. 8. Observations from these results revealed that mean of SWR decreases with increasing nano-SiC loading and NL. However, the marginal increase in SWR with increase in the SV was found for nano-SiC filled TSria/CF-E composites. Through Taguchi analysis, it was found that among all the considered wear parameters, the greatest impact on the SWR has the FL, and then the NL. The optimization of the wear parameters as per the selected factors and levels, with respect to the criterion smaller is better, results in the combination of control factors namely; FL, NL and SV, as can be seen from Fig. 8.

The SWR decreases sharply when the load in increased from 20 N to 60 N (Fig. 8) at maximum filler loading (FL4.5); and thereafter it decreases gradually. However, with an increment in NL, from 60 N to 80 N, the SWR significantly decreases on the ground that the area of contact between the coupon and the rotating disc are significantly increased at higher loads. Due to amplified contact surface, it allows maximum SiC particles to come across the interface and resist the shear loads. This prompts decrease in the SWR, especially when the NL is 80 N. The decrease in the SWR could be attributed to the modification of counter face by the debris of transfer film from the nano-SiC composite coupon.

FL. whereas, with an increment in FL from 3 to 4.5 wt.%, there is decrease in the SWR because of the nano-SiC, which is hard and having high strength with good between the filler and matrix in the final composites.

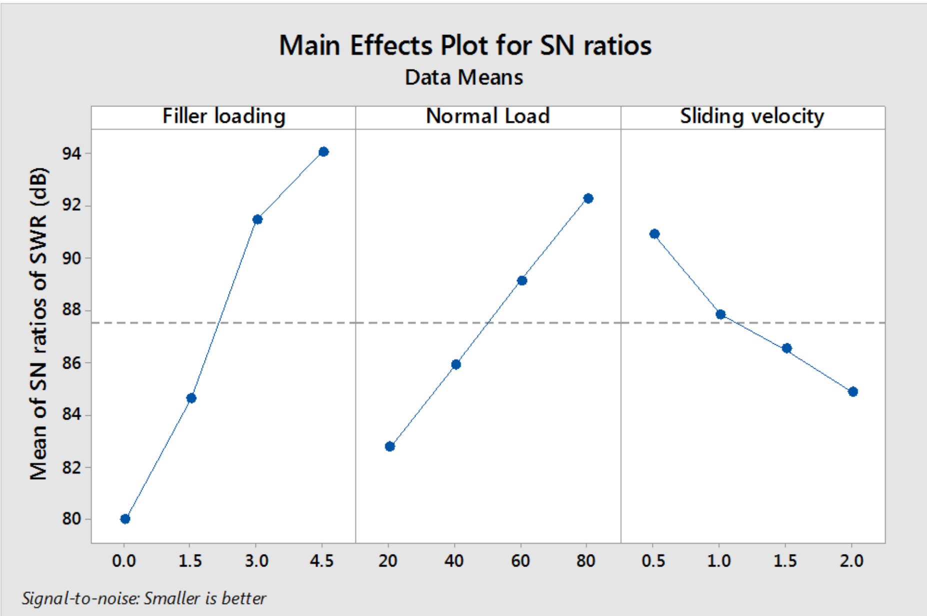


Fig. 9. Mean of SN Ratio of SWR for nano-SiC filled TSria/CF-E composites.

Next prevailing factor on the wear behaviour of composites is NL. A less decreasing trend in SWR was observed with increasing NL from 60 to 80 N. However, it increases sharply with increasing

Last factor which affects the wear behaviour of composite materials is SV. With increase in SV from 0.5 to 1 m/s and 1.5 to 2 m/s, there is a marginal increase in SWR; while from 1 to 1.5 m/s SV, there is a slight reduction in the SWR.

The μ ratio is determined using the equation 2 for each of 16 experiments and are tabulated in Table 4. Analysis of the result (Fig. 9) leads to the conclusion that the control factor combination of FL (4.5), NL (80) and SV (0.5), contributes to minimum SWR. The factors FL and NL have greater contribution on output performance, and the control factor SV has the least effect on the SWR. From this analysis, we can conclude that factors like filler loading (FL), and applied normal load (NL) have a significant effect on SWR and the sliding velocity (SV) has the least effect on SWR (Table 4). It can also be seen from Table 4 that the least and highest S/N ratios are estimated at 78.278 and 104.437 dB, corresponding to test runs 1 and run 16, respectively.

3.6 Analysis of variance (ANOVA)

The results from ANOVA for the adhesive wear behaviour of hybrid TSria/CF-E composites are calculated from S/N ratios of SWR (Table 4 and Fig. 9) and summarized in Table 7. The percentage contribution of control parameters on the SWR of $CaCO_3$ filled TSria/CF-E hybrid composites is illustrated in Fig. 10.

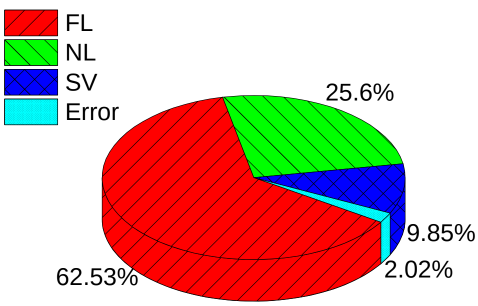


Fig. 10. Contribution of control parameters on the SWR of nano-SiC filled TSria/CF-E composites.

ANOVA was carried out for a level of significance of 10 %, which is, involving a level of confidence of 90 %. Table 7 reveals that factors FL, NL, and SV has statistical and physical importance on the SWR. As shown in the column P (%) of ANOVA table, P of each individual factor on overall variations shows the level of influence on the results of wear. It is seen that the factor FL (p = 62.5 %), factor NL (p = 25.6 %), and factor SV (p = 9.85 %) have the effects on the sliding SWR, but the factor SV showed very less effect on SWR. The error seen from ANOVA table for the mean S/N ratio is approximately 2.02 %.

The correlation of R2 (adj.) was 92.82 %. It is realized that that smaller the p-value, more prominent the criticalness of the factor interaction relating to it. The table of ANOVA for S/N ratio showed that the FL (p= 0.000), NL (p=0.001), and SV (p = 0.001) in this order the noteworthy control factors are effecting the SWR. It means that the FL is the major factor, followed by the NL and SV.

Table 7. ANOVA Table of S/N ratio of SWR for nano-SiC filled TSria/CF-E composites.

Source	DF	Adj SS	Adj MS	F-Value	P-Value	P (%)	Rank
FL	3	496.57	165.525	61.87	0.000	62.53	**1**
NL	3	203.33	67.778	25.33	0.001	25.60	**2**
SV	3	78.18	26.061	9.74	0.010	9.85	**3**
Error	6	16.05	2.675	-	-	2.02	-
Total	15	794.14	-	-	-	100	-

3.7 Linear regression analysis

Linear regression analysis was carried out in order to ascertain the correlation significant factors determined by the ANOVA analysis which were NL and FL. Relations of linear regression model with the experimental values are as shown in Fig. 11.

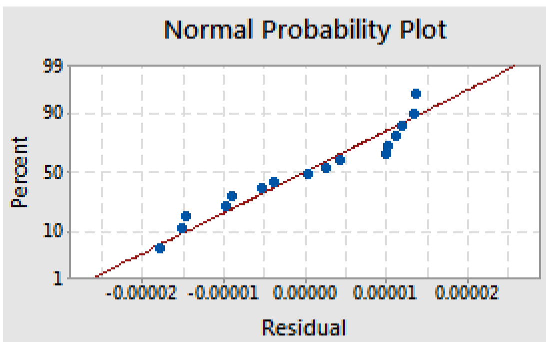
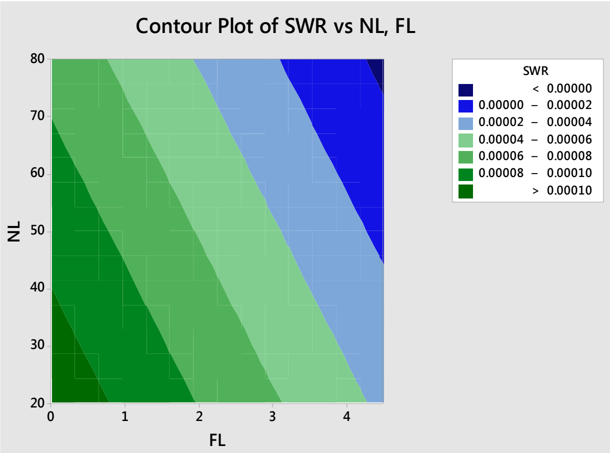
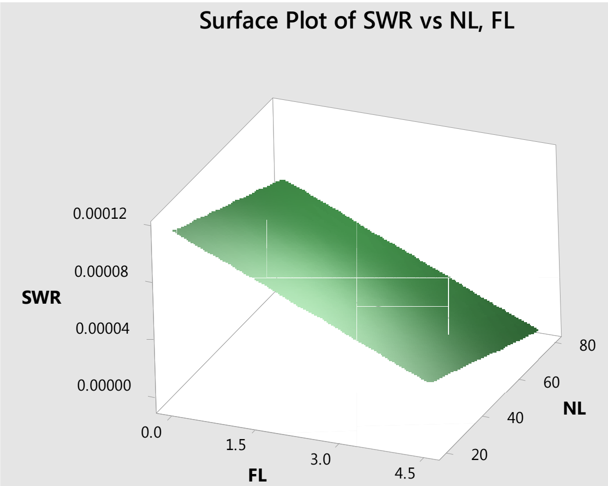


Fig. 11. Relation of linear regression model with the experimental values



(a)

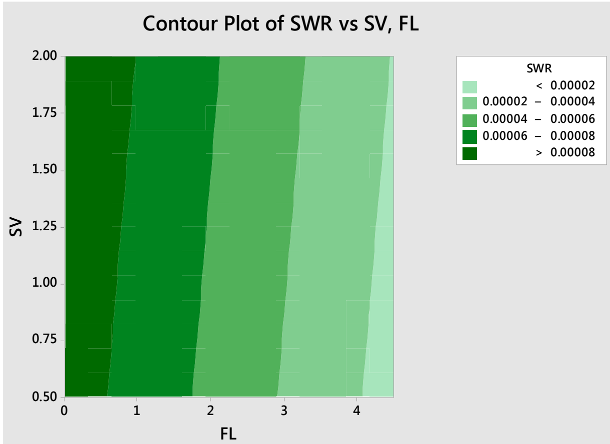


(b)

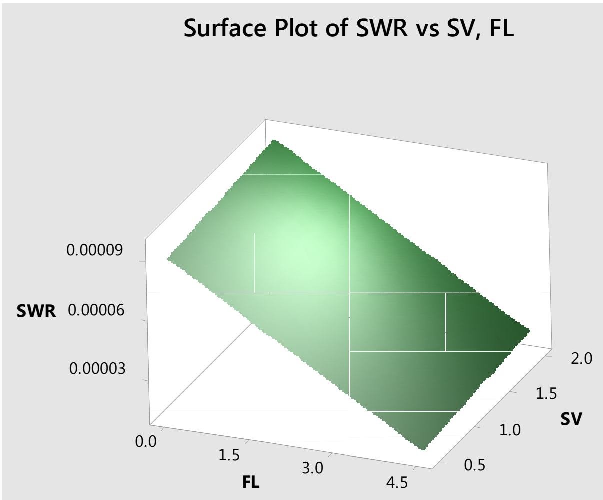
Fig. 12. Dependence of SWR on normal load and filler loading: (a) contour plot (b) surface plot.

Figure 12a depicts the contour plot of the SWR for NL and FL for nano-SiC filled TSria/CF-E composites. Curves suggest the existence of interaction between FL and NL. Conversely, in Fig. 12b, surface plot of SWR depending on the combination of factors FL and NL. Based on the analysis and graphs, minimal SWR is obtained with the highest FL (4.5 wt.% SiC), and at the NL (80 N), which means that the impact of highest filler loading in composite material with higher load have great impact. Its influence demonstrates with higher filler loading value (4.5 wt.%).

Figure 13a presents the contour plot of the SWR for FL and SV for nano-SiC filled TSria/CF-E composites. Curves suggest the presence of interaction between FL and SV. Conversely, in Fig. 13b the surface plot of SWR depending on combination of factors namely NL and SV. On the basis of the conducted analysis and given graphs, minimal values of SWR are achieved with highest load (80 N) in the considered range of SV (1.5 m/s to 2 m/s). This suggests that the impact of SV for the composites with maximum load does not have great impact. Furthermore, the SWR gradually upsurges to 20 N when SV vary from 1.5 m/s to 2 m/s.



(a)



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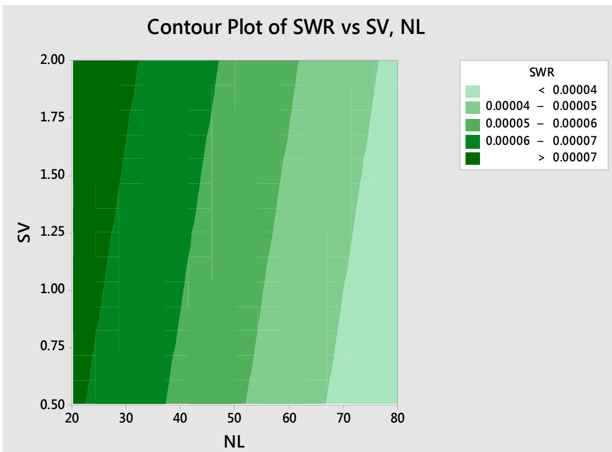

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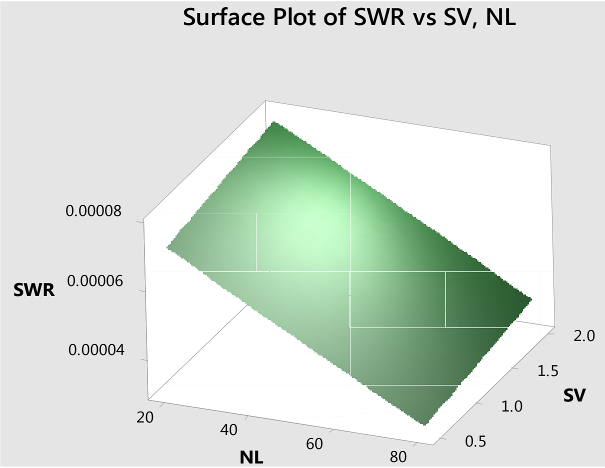


(d)

Fig. 13. Contour and surface plots of SWR:
(a) dependence on filler loading and sliding velocity
(b) dependence on normal load and sliding velocity.

A regression equation generated establishes the relationship between significant conditions obtained from the ANOVA analysis, i.e. FL, NL, and SV. The regression equation developed for SWR of nano-SiC filled TSria/CF-E composites is as shown in equation (5):

$$SWR = 0.000122 - 0.000017 \times FL - 0.000001 \times NL + 0.000004 \times SV \quad (5)$$

For nano-SiC filled TSria/CF-E composites, confirmation experiments were conducted by utilizing the levels of the optimal process parameters (FL4.5, NL80, and SV0.5). The results of experimental confirmation using optimal wear parameters and comparison of the predicted SWR with the experimental SWR are listed in Table 6.

In general fibrous materials, either short or long, carbon fibers are able to reduce the specific wear rate and friction coefficient of thermoset/thermoplastics predominantly due to their notable load carrying capability [53-55]. Likewise, nano-SiC filler can also decrease the SWR of epoxy by changing the wear mechanism from severe abrasion to mild sliding wear [54, 55]. When TSria, CF and nano-SiC reinforcing materials are incorporated into the epoxy composites, a desirable synergetic effect appears (Table 4). It is fascinating to find that the composites with 4.5 wt.% nano-SiC in TSria/CF-E have the lowest SWR; the enhancement is more significant when compared unfilled and $CaCO_3$ filled TSria/CF-E composites. In general, hardness is one of the most important factors that direct the materials wear resistance. Harder surface would have higher wear resistance.

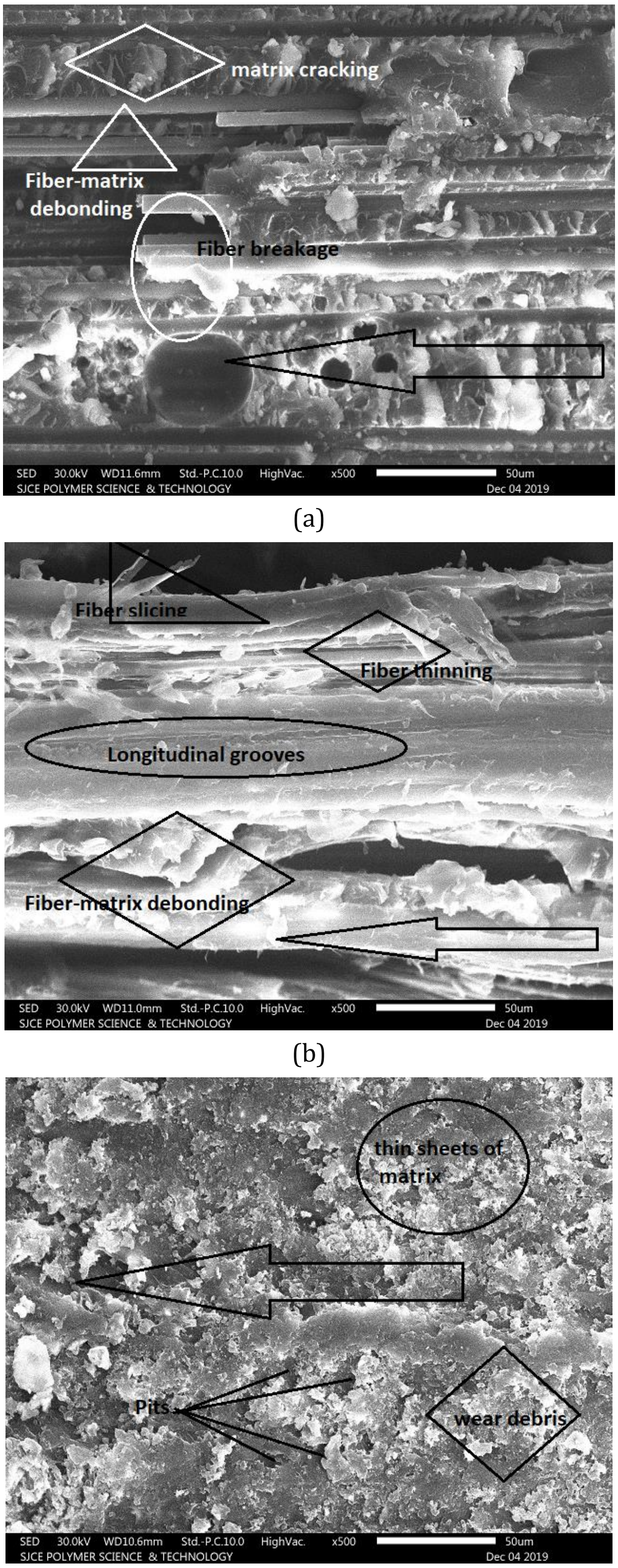
To understand the synergetic effect revealed in Table 4, the SiC has higher hardness and the TSria/CF fibers dispersed in the epoxy matrix, which increases hardness, tensile strength and creep resistance of the composites and reduces the SWR [55]. The hardness of TSria/CF-E with nano-SiC (4.5 wt.%) is almost the highest among other nano-SiC/ nano-$CaCO_3$ filled TSria/CF-E composites. Furthermore, TSria/CF-E filled with nano-SiC, the wear resistance tended to increase with increasing loading of nano-SiC. Compared with the SWR of the nano-SiC filled TSria/CF-E, TSria/CF-E composites were similarly affected by the filling of nanometer $CaCO_3$. Interestingly, the largest wear resistance was observed nano-SiC filled the TSria/CF-E composites.

3.8 Worn surface morphology

Scanning electron microscope (SEM) images of the worn surfaces of unfilled and nano-filler filled TSria/CF-E composite coupons at an applied load of 80 N are illustrated in Fig. 14. The wear track depths increased in unfilled TSria/CF-E composites (Fig. 14a) when compared to nanofiller filled TSria/CF-E composites at an applied load of 80 N (Figs. 14b and 14c).

Unfilled TSria/CF-E composite achieved highest SWR and could be justified by SEM micrograph (Fig. 14a) at 2 m/s sliding velocity, and at an applied load 80 N. At higher load, matrix cracking, fiber-matrix debonding, fiber breakage and pull-out of fiber from the matrix are clearly seen in the SEM micrograph of TSria/CF-E composite. The highest SWR of these composites could be due the increased load which in turn increases the interface temperature between the disc and the composite coupon which weakened the fiber-matrix interfacial bonding and increased the SWR.

Figure 14b shows the worn surface morphology of nano-$CaCO_3$ filled TSria/CF-E composite coupon after wear testing. From the micrograph, it is observed that the worn surface mainly composed of longitudinal grooves caused by micro-cutting and micro-ploughing. At increased load and sliding velocity, the SWR reduces with increase in filler loading. This is because, the interface temperature increases with the high load and velocity, hence the composite surface becomes soft caused by frictional heat at the interface, thus the reduction in the SWR. Other microscopic features include thinning of fiber, fiber slicing, and debonding of fiber from the epoxy matrix.



(c)

Fig. 14. Worn surface images of (a) TSria/CF-E, (b) Nano-$CaCO_3$ filled, (c) Nano-SiC filled TSria/CF-E composites at 80 N load.

Figure 14c depicts worn surface micrograph of nano-SiC filled TSria/CF-E composite coupon. Fatigue wear is the key mechanism accountable for material removal in the form of thin slips. Bound wear debris is found in some areas, which aids to guard the bulk composite from severe wear. Manifestly, the bond between the filler and matrix increases. Other microscopic features include: low wear track depth, pits, and more matrix debris. Good structural integrity among filler/fiber-epoxy matrix in nano-SiC filled TSria/CF-E resulted in lowest SWR among the developed composites.

4. CONCLUSIONS

Adhesive wear behaviour of TSria/CF-E and its multi-phase composite coupons has been carried out by Taguchi design of experiments. The analysis was carried out for nano-$CaCO_3$ and nano-SiC multi-phase composite coupons under different sections. On the basis of the results obtained from the present study, the following conclusions were made.

- Both nano-$CACO_3$ and nano-SiC fillers inclusion in TSria/CF-E hybrid composites, the SWR decrease with increase in filler loading, load and increase in sliding velocity.
- The combination of control parameters and their levels for optimum SWR and hence, for optimum SWR of nano-$CaCO_3$ and nano-SiC filled TSria/Cf-E hybrid composites under constant sliding distance are FLNLSV (filler loading of 4.5 wt.%, normal load of 80 N and sliding velocity of 0.5m/s). The test results reveals that the TSria/CF-E hybrid composite with 4.5 wt.% nano-SiC filler showed superior wear resistance when compared to unfilled and nano-$CaCO_3$ filled TSria/CF-E hybrid composites.
- The contributions of FL, NL, and SV to the quality characteristic i.e. SWR of TSria/CF-E with nano-$CaCO_3$ is 38.73 %, 42.15 %, and 16.24 % respectively.
- The contributions of FL, NL, and SV to the quality characteristic i.e. SWR of TSria/CF-E with nano-SiC is 62.53 %, 25.6 %, and 9.85 % respectively.

- Regression equations generated for the 4.5 wt.% $CaCO_3$/SiC filled TSria/Cf-E hybrid composites and are used to predict the SWR of the composites for intermediate condition with reasonable accuracy.
- Worn surface micrographs are utilized to support the SWR data of the hybrid composites. Matrix cracking, grooves, fiber-matrix debonding, fiber breakage and pull-out of fiber from the matrix are primary wear mechanism for unfilled TSria/Cf-E hybrid composites; conversely nanofillers reduces groves and fiber breakage, low wear track depth, few pits, and more matrix debris.

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