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Influence of areca nut nanofiller on mechanical and tribological properties of coir fibre reinforced epoxy based polymer composite

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Abstract. The present study was aimed at investigating the effect of incorporating areca nut nanofiller on the tribological behaviour of coir reinforced epoxy based polymer matrix composite. Areca nut nanofiller was produced by grinding followed by ball milling. Particle size analyser confirmed that the size of nanofillers obtained was in the range of 20–100 nm. Composites with different weight percentages of nanofiller (0%, 5%, and 10%) were studied for their mechanical and tribological behaviours using pin on disc rig. Tensile, flexural, and inter-laminar shear and impact tests were carried out on the proposed composite. Taguchi's technique was used to analyse the effects of various factors on the tribological behaviour of the composite. The results showed that inclusion of areca nut nanofiller enhanced the micro hardness of the composite. Moreover, it first increased the tensile strength by up to 5% and then, there was a decrease in tensile strength. Flexural strength significantly increased with increase in filler percentage from 0% to 5%, but the variation of flexural strength from 5% to 10% was negligible. Inclusion of filler had negligible effect on the inter-laminar shear strength of composites. Impact strength and wear resistance of the composite were enhanced with incorporation of filler.

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1. Introduction

Composites based on natural fibres are finding applications in almost all fields of engineering, since natural fibres have better potential to be a reinforcing material for polymer matrix composites [1]. The varieties of natural fibres like jute, coir, hemp, sisal, banana, and bamboo have established their potential as substitutes for synthetic fibres in many engineering applications owing to their advantages over the synthetic fibres [2–7]. Coir is the most commonly used natural fibre obtained from coconut fruit and it is abundantly available in Southern parts of India. This fibre has multiple features such as low weight and cost and high specific strength [8–11], which make it a potential candidate for substituting synthetic fibres. Many researchers suggested using coir fibres as a suitable reinforcement in composites due to its excellent mechanical properties. The coconut shell based nanofiller was used in hybrid kenaf/coir reinforced vinyl ester composite and it was reported that incorporation of the filler enhanced the mechanical properties of the composite [12].

Studies carried out by various researchers confirm that the wear resistance of polymer is enhanced when it is reinforced with fibres [13] or used with

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lubricants [14,15]. In addition, some other studies have shown that the incorporation of nano particles enhances the tribological properties of polymers [16–20]. To achieve better wear properties, many researchers have modified the polymers using different fillers [21–24]. Addition of CuO and Pb_3O_4 as fillers enhanced the wear resistance of high-density polyethylene (HDPE) [21]. ZrO_2 and TiO_2 as fillers reduced the loss of mass due to wear in polytetrofluroethylene (PTFE) [22]. Compounds of copper, like CuO and CuS, resulted in enhanced wear resistance of polyether ether ketone (PEEK), PTFE, nylon, and HDPE [24]. Most of the studies of fillers cited above deal with metallic fillers. The effect of natural plant based fillers on the tribological behaviour of polymer matrix composites has less been explored. Controlling the length of abaca fibre, the wear resistance of the friction composite can be significantly enhanced [25].

Natural areca nut (areca catechu) is inexpensive and abundantly available. Karnataka is largest areca nut producing state in India, which has a share of around 50% of the whole areca production in the country. Chemically treated areca fibre reinforced epoxy composite provides better impact properties [26]. Physio-mechanical properties of hybrid betel nut (areca catechu) short fibre/seaweed polypropylene composite has been studied previously. It was reported that with increase in filler percentage, the tensile strength decreased [27].

Although abundant research on the tribological behaviour of composites has been carried out, the use of natural plant based fillers in polymer matrix composite has less been addressed. Few researchers have reported the use of areca nut as a fibre in the composite, but its use as a filler in composites for tribological applications is not reported. In this regard, the present work is focused on exploring the use of plant based areca nut (areca catechu) as filler in polymer matrix and its effect on the tribological properties of the composite.

2. Taguchi's Design of Experiments (DoE)

Taguchi's Design of Experiments (DoE) statistical technique is a very useful and powerful tool for designing the experiments. It involves a systematic way of collecting data, analysing them, and interpreting the results [28,29]. The advantage of using Taguchi technique is that more amount of information can be obtained with minimum experimentation and hence, the time and cost are reduced. The performance characteristics can be optimized through this method. However, the selection of factors is the critical stage in this methodology. Taguchi method makes use of orthogonal array to incorporate various factors and defines the plan of experiments. The responses obtained are analysed through mean, Signal to Noise (SN) ratio, and Analysis of Variance (ANOVA) to which the regression model can be fitted.

3. Materials and methods

3.1. Composite preparation

The proposed composite makes use of areca nut nanofiller in the epoxy matrix, which is reinforced by coir fibre. L12 epoxy with K6 hardener is used for its better resistance to alkali and good adhesive property. The coir fibres in the form of woven mat and the areca nut were procured from the local suppliers of Tumakuru district, Karnataka, India. Areca nut nanofiller is produced by grinding followed by ball milling. Particle size analyser confirmed that the size of nanofillers obtained was in the range of 20–100 nm. The composition and weight fraction of the composite with varied fillers, fibres, and matrices are shown in Table 1.

The composites were fabricated adopting hand layup technique and cured at room temperature for 24 hours. The specimen conforming to ASTM G99-95 standard was cut from the cured laminate.

3.2. Test of density, micro hardness, tensile, flexural, and impact properties

3.2.1. Density

Theoretical density can be found by using Eq. (1) according to [30]:

$$\rho_{th} = \frac{1}{\left(\frac{W_f}{\rho_f} + \frac{W_m}{\rho_m}\right)}, \tag{1}$$

where W is the weight fraction and ρ is the density. The suffixes m, f, and th stand for matrix, fibre, and theoretical, respectively. Since the present study makes use of filler, Eq. (1) can be modified by Eq. (2):

$$\rho_{th} = \frac{1}{\left(\frac{W_f}{\rho_f} + \frac{W_m}{\rho_m} + \frac{W_p}{\rho_p}\right)}. \tag{2}$$

The void percentage is determined using Eq. (3):

$$Void\ content\,(\%) = \left(\frac{\rho_{th} - \rho_a}{\rho_{th}}\right) \times 100, \tag{3}$$

where ρ_{th} is theoretical density and ρ_a is actual density calculated using Archimedes principle.

Table 1. Composition and weight fraction of the composites with varied fillers, fibres, and matrices.

Composite designation	Weight percentage		
	Arecanut powder	Coir	Epoxy
C1	0	40	60
C2	5	40	55
C3	10	40	50

3.2.2. Micro hardness

Micro hardness testing machine was used to calculate the Vickers harness number of the composite. In the present study, the load of 29.43 N was applied to the specimen. Vickers hardness number is calculated using Eqs. (4) and (5):

$$H_v = 0.1889 \times \frac{F}{L^2}, \tag{4}$$

$$L = \frac{X+Y}{2}, \tag{5}$$

where F is the applied Newtonian load, L is the diagonal of square impression in millimeters, X is the horizontal length in millimeters, and Y is the vertical length in millimeters.

3.2.3. Tensile strength

The tensile test was carried out by applying a uniaxial load according to ASTM D3039-76. The crosshead speed of the universal testing machine was maintained at 10 mm/min and the results were analysed to find the tensile strength of the composites. Five specimens of each composite configuration were tested and the averages were considered.

3.2.4. Inter-laminar shear and flexural strength

To find the inter-laminar shear strength, short beam shear tests were performed according to ASTM D2344-84 standard and the flexural strength and inter-laminar shear strength were calculated using Eqs. (6) and (7), respectively. The dimensions of the specimen used were 60 mm × 10 mm × 4 mm. The crosshead speed and span length were maintained at 10 mm/min and 40 mm, respectively. Five samples of each composite configuration were tested separately for flexural and shear tests:

$$F_s = \frac{3PL}{2bt^2}, \tag{6}$$

$$I_s = \frac{3P}{4bt}, \tag{7}$$

where P is the maximum load and b is the width, t the thickness, and L the span length of the specimen.

3.2.5. Impact test

Pendulum-based low-velocity impact test was carried out according to ASTM D256 standard with V-notch and notch depth of 20 mm. Five specimens of each composite configuration were tested and the average values were considered. The specimens were fixed in a square support and impacted at their central point by a hemispherical bolt of 4 mm diameter. The respective values of impact energy for different specimens were recorded directly from the dial indicator.

3.2.6. Experimental design and wear runs

In order to investigate the dry sliding wear behaviour of the proposed composite, a pin on disc test apparatus was used. The specifications of the disc are given in Table 2.

The parameters considered to be varied were the speed, load, and distance. Before subjecting the composite specimens to testing, they were cleaned with acetone. The initial weight of the specimen was measured using precision weighing balance. During the test, pin was pressed against the counterpart rotating against the steel disc by applying load. After completing the specified sliding distance for the specimen, it was removed, cleaned with acetone, and dried. Then, its final weight was noted down to find the weight loss due to wear. The weight loss of the composite due to wear was calculated using Eq. (8):

$$\text{Weight loss} = \text{Initial weight - Final weight.} \tag{8}$$

Weight fraction of the filler in terms of composite designation, sliding speed, and load and sliding distances was considered variable in the present study. The factors and their levels chosen in the present study are represented in Table 3. Experimentations were conducted according to Taguchi's L9 orthogonal array in order to investigate the effect of the filler and other chosen parameters on the wear resistance of the composites. The plan of experiments is represented in Table 4.

4. Results and discussions

4.1. Mechanical properties

The theoretical and experimental densities of the composites with varied weight percentages of areca nut nanofiller are given in Table 5. The density of neat resin mixed with hardener with the ratio of 10:1 was found 1040 kg/m^3. As seen in Figure 1, with the addition of

Table 2. Specifications of the disc.

Material	HRC	Diameter	Thickness	Surface roughness
En-32 steel Hardened	62	135 mm	8 mm	0.8 μm

Table 3. Factors and levels used in the Taguchi study.

Factors	Level 1	Level 2	Level 3
Composite designation	C1	C2	C3
Sliding speed (m/s)	2	4	6
Load (N)	10	20	30
Sliding distance (m)	500	1000	1500

the filler, the density of the composite was reduced and the void percentage increased.

Table 6 provides the hardness value, tensile strength, flexural and interlaminar shear strength, and impact strength of the composites, which are also graphically represented in Figure 2.

The wear resistance of a composite mainly depends on its hardness. The microhardness test carried out showed that with the incorporation of areca nut nanofiller from 0% to 10%, the hardness of the coir reinforced epoxy composite increased from 18 Hv to 27 Hv, as presented in Table 6. Figure 2 shows the variation of hardness value of composite with varying filler percentage. It can be seen that with the addition of filler, the hardness values significantly increases. The variation of tensile strength of the coir-reinforced epoxy composite is presented in Table 6. From Figure 2, it can be concluded that with the inclusion of areca nut filler, tensile strength increases by up to 5% and after that, it experiences a decrease.

The flexural and inter-laminar shear strength of the coir reinforced composite with various filler loading percentages is presented in Table 6. The variation of flexural strength and inter-laminar shear strength of the composite with different percentages of

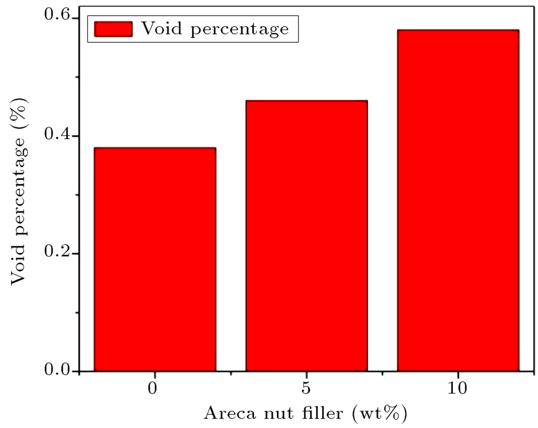

Figure 1. Variation of void percentage with inclusion of filler.

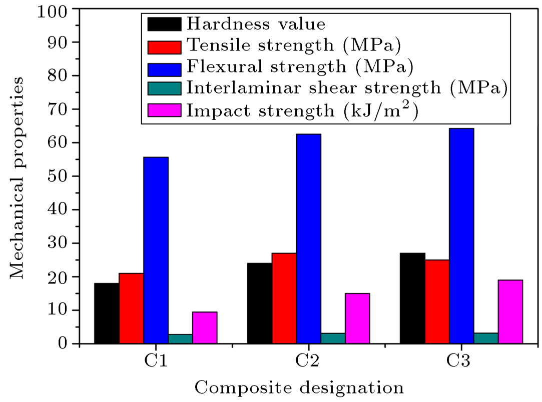

Figure 2. Variation of mechanical properties with inclusion of filler percentage.

Table 4. Plan of experiments according to Taguchi's L9 orthogonal array.

Composite designation	Sliding speed	Load	Sliding distance
1	1	1	1
1	2	2	2
1	3	3	3
2	1	2	3
2	2	3	1
2	3	1	2
3	1	3	2
3	2	1	3
3	3	2	1

Table 5. Theoretical and experimental densities of composites.

Composite designation	Weight percentage of filler	Theoretical density (kg/m^3)	Actual density (kg/m^3)	Void percentage
C1	0	1023.62	1019.73	0.38
C2	5	1004.50	999.87	0.46
C3	10	986.09	980.37	0.58

Table 6. Properties of composites for various percentages of filler.

Composite designation	Filler percentage	Hardness value	Tensile strength (MPa)	Maximum load (N)	Flexural strength (MPa)	Inter-laminar shear strength (MPa)	Impact strength (kJ/m^2)
C1	0	18	21	148.34	55.63	2.78	9.5
C2	5	24	27	166.77	62.54	3.12	15
C3	10	27	25	171.28	64.23	3.21	19

incorporated filler is also given in Figure 2. It can be observed that flexural strength significantly increases with increase in filler percentage from 0% to 5%, but its variation from 5% to 10% is negligible. Similarly, it can be observed that the inclusion of filler has negligible effect on the inter-laminar shear strength of composites.

The impact energy absorbed by the coir reinforced composite with various filler loading percentages is presented in Table 6. In Figure 2, it is evident that the impact strength of the composite is enhanced with the incorporation of the filler and with the addition of more amounts of filler, impact strength increases.

4.2. SN ratio

Table 7 gives the weight loss as a response to different combinations of factors along with SN ratios and means obtained for various runs of Taguchi's L9 orthogonal array.

The aim was to minimize the weight loss of the composite due to wear. Hence, the SN ratio was calculated to achieve the minimum weight loss with "the smaller the better" criterion as a logarithmic transformation of the loss function given by Eq. (9):

$$S/N = -10\ log_{10}\left((\Sigma y^2)/n\right), \tag{9}$$

where y is the response for a given factor level combination and n is the number of responses in the factor level combination.

The present study makes use of the commercially available statistical tool of MINITAB 17 for the computational practices. The 'weight loss' response of the composite was analysed and the effect of the factors on the response was studied. The responses for SN ratios are given in Table 8. Analysing the weight loss of the composites showed that composite designation at level 3 (10% weight fraction of areca nut nanofiller) with the highest SN ratio gave minimum wear loss. Moreover, it can be stated that the wear loss of the composite mainly depended on the filler percentage (composite designation) followed by load, sliding distance, and sliding speed. Thus, inclusion of the areca nut nanofiller enhanced the wear resistance of the composite.

Figure 3 shows the plots for the main effects of means and SN ratios. It is observable that the minimum wear loss was obtained when the filler percentage in the composite was high (level 3). According to the interaction plot for weight loss in Figure 4, since there are no parallel lines, there exists an interaction among all the factors considered for the weight loss of the composite, with filler percentage (composite designation) being the most significant factor.

4.3. ANOVA

ANOVA is a proper statistical means for finding the significant factors. It indicates to what extent a process parameter influences the response calculates its significance level. The ANOVA values for the weight loss of the composites are presented in Table 9.

According to the table, among the control fac-

Table 7. Signal to Noise (SN) ratios and means for various runs.

Runs	Composite designation	Sliding speed	Load	Sliding distance	Weight loss (gms)	SN ratio (dB)	Mean
1	1	1	1	1	0.28	11.0568	0.28
2	1	2	2	2	0.32	9.8970	0.32
3	1	3	3	3	0.35	9.1186	0.35
4	2	1	2	3	0.24	12.3958	0.24
5	2	2	3	1	0.20	13.9794	0.20
6	2	3	1	2	0.22	13.1515	0.22
7	3	1	3	2	0.20	13.9794	0.20
8	3	2	1	3	0.16	15.9176	0.16
9	3	3	2	1	0.18	14.8945	0.18

Table 8. Response table of Signal to Noise (SN) ratios for all three stacking sequences.

Levels	Composite designation	Sliding speed (m/s)	Load (N)	Sliding distance (m)
1	10.02	12.48	13.38	13.31
2	13.18	13.26	12.40	12.34
3	**14.93**	12.39	12.36	12.48
Delta	4.91	0.88	1.02	0.97
Rank	1	4	2	3

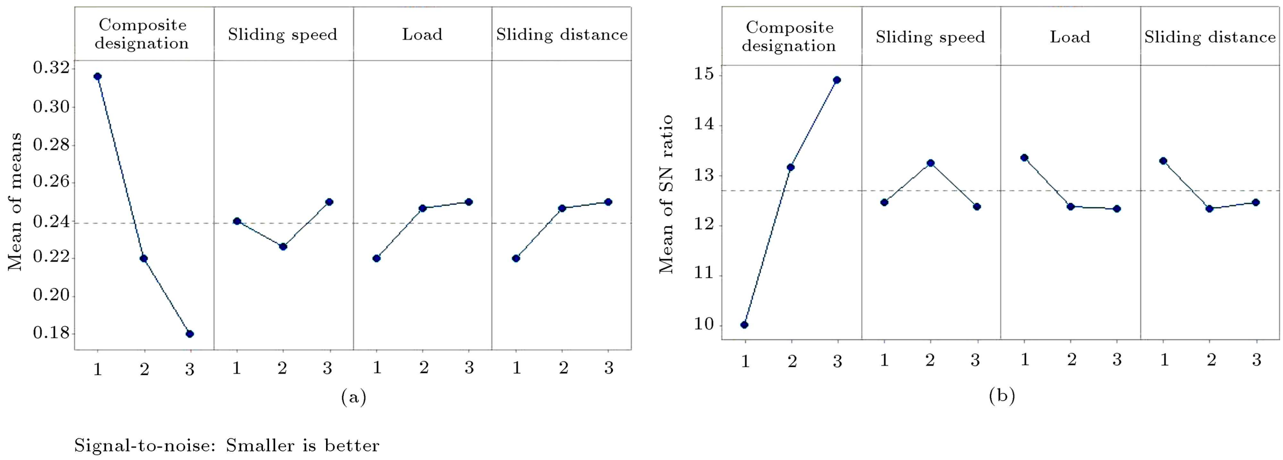

Figure 3. Main effect plot for (a) means and (b) Signal to Noise (SN) ratios.

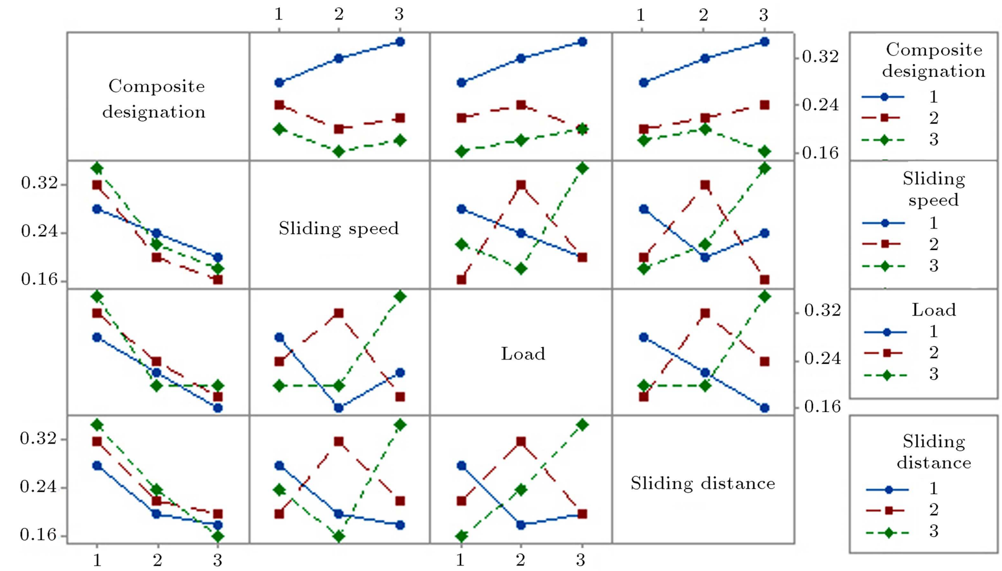

Figure 4. Interaction effect plot for weight loss of composites.

tors, composite designation (filler percentage) has the highest F value, followed by load/sliding distance and sliding speed. In addition, the percentage contribution, calculated using Eq. (10), is the highest for composite designation (filler percentage), which indicates that filler percentage has a significant effect on the weight loss of the composite due to erosion. The R-sq value indicates the coefficient of determination for an

Table 9. Analysis of variance (ANOVA) for weight loss of composites.

Source	DF	SS	MS	F value	P value	Contribution (%)
Composite designation	1	0.028017	0.028017	39.71	0.003	83.16
Sliding speed	1	0.000150	0.000150	0.21	0.669	0.44
Load	1	0.001350	0.001350	1.91	0.239	4
Sliding distance	1	0.001350	0.001350	1.91	0.239	4
Error	4	0.002822	0.000706			8.37
Total	8	0.033689				100

$S = 0.0265623$, R-sq = 91.67%, R-sq (adj) = 90.25%

equation. It is more than 90% in this study, which indicates that the developed model gives good results and helps to predict the weight loss values within experimental conditions.

$$\%contribution = \left(\frac{SS_f}{SS_t}\right) * 100, \quad (10)$$

where SS_f is the sum of squares of a factor and SS_t is the total sum of squares.

4.4. Regression analysis

Regression models are developed for the output responses and the regression equation developed for the composite is shown in Eq. (11):

$$\begin{aligned} Weight\ loss =& 0.3056 - 0.0683\ Composite\ designation \\ &+ 0.0050\ Sliding\ speed + 0.0150\ Load \\ &+ 0.0150\ Sliding\ distance. \quad (11) \end{aligned}$$

The coefficient associated with the control factors indicates that increase in filler percentage leads to a reduction in weight loss. Furthermore, filler percentage has the highest coefficient, indicating that the influence of filler on weight loss is significant compared to other factors.

In order to validate the developed model, the experimental results are compared with the predicted results in Table 10. he error percentage was found within 10%, indicating that the developed models were adequate and feasible to predict the weight loss due to wear within the range of experimental conditions. Figure 5 shows the comparison of experimental and calculated values of weight loss.

4.5. Contour plots

Figure 6 shows the contour plots for weight loss against various combinations of factors. It can be seen from all the graphs that the weight loss is maximum at the first level of composite designation (0% filler) and the third level of load (30 N) with sliding distance of 1500 m and sliding speed of 6 m/s. On the other hand, minimum weight loss is obtained at the third level of composite designation (10% filler), which means that weight loss is minimized with increase in filler percentage.

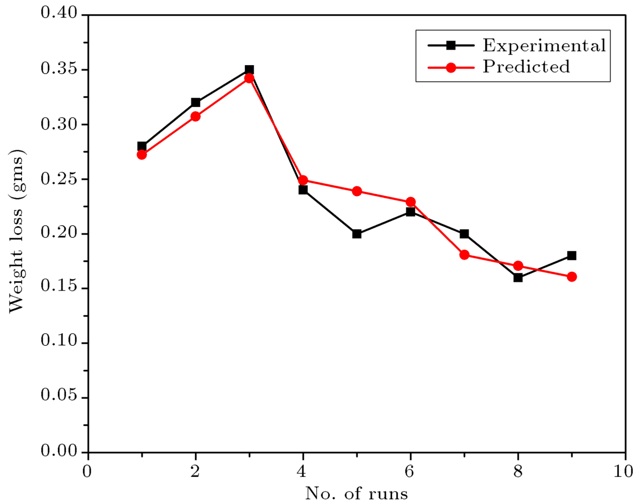

Figure 5. Comparison of experimental and predicted values of weight loss for composites.

5. Conclusions

In the present study, the effects of various percentages of areca nut nanofiller on the wear behaviour of the coir reinforced epoxy based polymer matrix composite were studied. The impacts of various factors on the mechanical and tribological properties of the composite were investigated. It was found that with the addition of the filler, the density of the composite was reduced and the void percentage and microhardness increased. The tensile strength of the composite was enhanced by the inclusion of the filler by up to 5% and after that, it decreased. Flexural strength significantly increased with increase in filler percentage from 0% to 5%, but its variation was negligible from 5% to

Table 10. Comparison of the experimental and calculated values of weight loss for composites.

Composite designation	Sliding speed	Load	Sliding distance	Experimental weight loss (gms)	Predicted weight loss (gms)	Percentage error
1	1	1	1	0.28	0.2723	2.75
1	2	2	2	0.32	0.3073	3.96
1	3	3	3	0.35	0.3423	2.20
2	1	2	3	0.24	0.249	–3.75
2	2	3	1	0.20	0.239	–19.50
2	3	1	2	0.22	0.229	–4.09
3	1	3	2	0.20	0.1807	9.65
3	2	1	3	0.16	0.1707	–6.68
3	3	2	1	0.18	0.1607	10.72

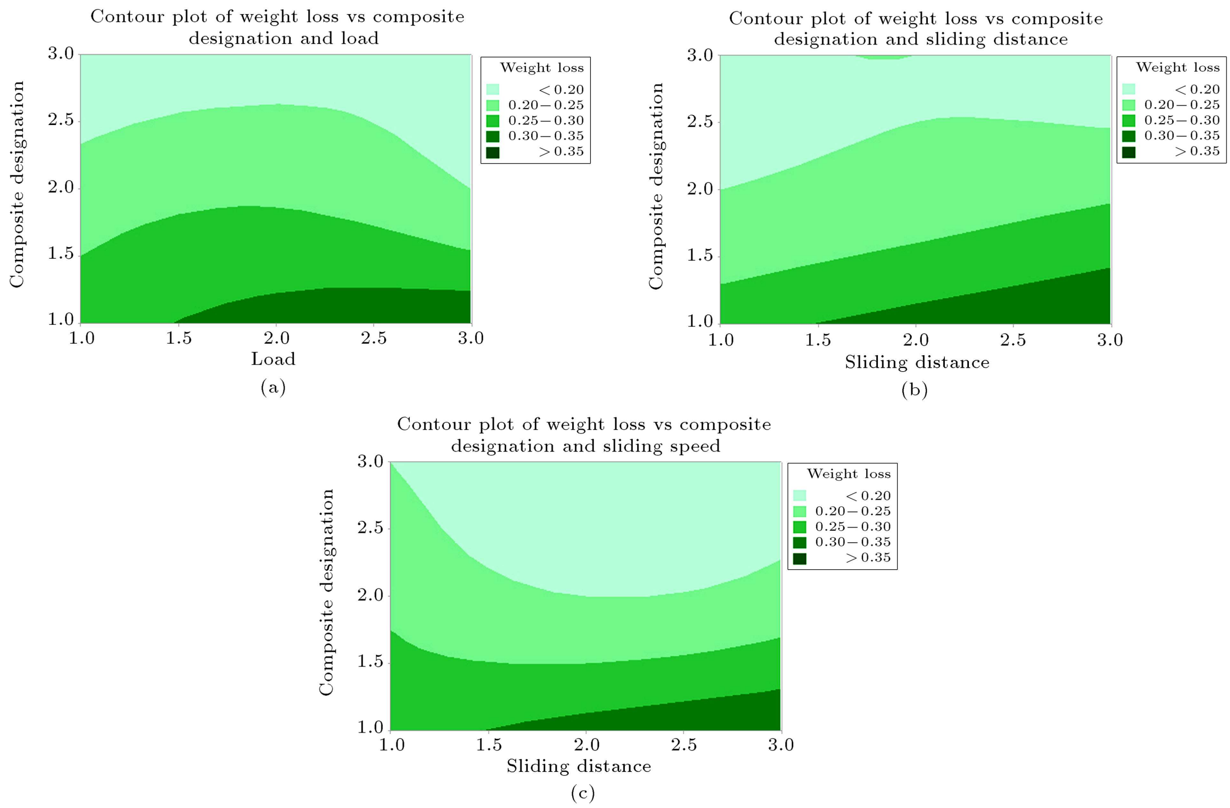

Figure 6. Contour plots of weight loss against various combinations of factors.

10%. It was observed that the inclusion of filler had negligible effect on the inter-laminar shear strength of the composites. Impact strength of the composite was enhanced with the incorporation of the filler. It was concluded that the incorporation of areca nut nanofiller significantly enhanced the wear resistance of the composite. Incorporating 10% weight fraction of areca nut nanofiller gave lower wear loss than 5% and no incorporation. It was found that the wear loss of the composite mainly depended on the filler percentage followed by load, sliding distance, and sliding speed. Analysis of variance (ANOVA) showed that the percentage contribution of filler loading was the highest (83.16%), followed by load/sliding distance (4%) and sliding speed (0.44%). Moreover, regression model was developed and validated. It was observed that the error percentage was within 10%, indicating that the developed models were adequate and feasible to predict the weight loss due to wear within the range of experimental conditions.

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