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Mechanical and Dry Sliding Wear Behavior of Particulate Fillers $CaCO_3$ and $CaSO_4$ Filled Vinyl ester Composites

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Abstract The improved performance of polymers and their composites in industries and many other applications by the addition of particulate fillers has shown great advantages and so has lately been the subject of considerable interest. In this paper, mechanical and tribological behavior of particulate fillers $CaCO_3$ and $CaSO_4$ filled vinyl ester composites have been presented. Wear tests were carried out in dry sliding conditions on a pin-on-disc friction and wear test rig. (DUCOM) at room temperature under sliding velocity (1.57, 2.62 and 3.67 m/sec.), normal load (20, 40 and 60 N), filler content (0, 10 and 20 wt.%) and sliding distance (1000, 3000 and 5000 m). The plans of experiments is based on the Taguchi technique, was performed to acquire data in a controlled way. An orthogonal array and analysis of variance (ANOVA) were applied to investigate the influence of process parameters on the coefficient of friction and sliding wear behaviour of these composites. The coefficient of friction and specific wear rate were significantly influenced with increase in both the filler content. The results show that for pure vinyl ester the coefficient of friction and specific wear rate increases with the increase of normal load, sliding velocity and sliding distance. The coefficient of friction and specific wear rate for $CaCO_3$ filler decreases with the increase of filler content. But, for filler $CaSO_4$ the coefficient of friction and specific wear rate decreases at 10 wt.% and then increases at 20 wt.%. It is believed that a thin film formed on stainless steel counterface was seems to be effective in improving the tribological characteristics. The worn surfaces examined through SEM to elucidate the mechanism of friction and wear behaviour.

Keywords Sliding Wear, Polymer-Matrix Composites, Fillers, Scanning Electron Microscopy

1. Introduction

An informal number of papers dealing with the tribological behaviors of polymer materials have been published. That is why the polymers are extending over a great area used in sliding components like such as gears, cams, breaks, clutches, bearings, wheels and bushes. Adhesive wear includes galling, fretting, scuffing and surface fatigue. This refers to the damage produced when two mating surfaces move relative to each other under a normal load. Surface asperities interact and very high stresses, strain, and strain rates are generated in localized regions[1]. This type of wear occurs in bearings, piston rings, cylinders and in electrical contacts. In recent years attention has been focused on the sliding wear behavior of polymers and their composites due to their increasing use as bushings and seals in machinery.

The research by various authors[2-4] reported that the friction between polymers can be attributed by the two main mechanisms i.e, deformation and adhesion. The deformation mechanism involves dissipation of energy in the contact area. The adhesion components of friction of polymer results from the breakage of bonds between the polymer and mating sliding surface[3]. Franklin[5] studied the friction and wear behavior of POM polymer and reported that the effect of sliding speed on the wear of polymers does not always follow the generally accepted engineering rule of "higher sliding speed, the higher wear rate".

From the point of view of the coefficient of friction, Brentnall and Lancaster[6] reported that the friction coefficient of polymers rubbing against metals decreases with the increase in load. Some researchers[7-9] reported that the coefficient off friction value increases with the increase in load. Finally, Byett and Allen[10] and Friedrich et al.[11] have reported that the coefficient off friction value increases with the increase in load. Many polymeric materials have an excellent strength-to-weight ratio, good corrosion resistance, wider choice of the materials, dimensional stability, high impact strength, light weight and ease to manufacturing. Some polymers also possess excellent tribological properties[12]. The polymers can be considered to be one of the competitive materials for tribological applications because of their low friction values against steel counterparts, good damping properties, and self

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lubricating abilities.

It has been observed that by assimilating filler particles in the polymer based composites, synergistic effects may be achieved in the form of higher modulus and reduction of the material cost[13-15]. The inclusion of such particles into polymers for commercial applications is focused at the cost reduction and stiffness improvement[16, 17]. Various kinds of polymers and polymer-matrix composites with different types of fillers such as Al_2O_3, SiC, TiO_2, fly ash etc. have been the subject of extensive research in recent years as found from the literature[18-21]. But the potential of particulate fillers $CaCO_3$ and $CaSO_4$ in vinyl ester matrix has not been reported so far. So, in this paper we are using these fillers ($CaCO_3$ and $CaSO_4$) with vinyl ester matrix and studying the effect of such fillers on mechanical and dry sliding wear behavior of the composites. Polymer composites containing different fillers and/or reinforcements that are frequently used for applications like automotive parts, gear assemblies, tub/ shower industries etc. in which friction and wear are critical issues. Calcium carbonate ($CaCO_3$) and calcium sulfate ($CaSO_4$) are the fillers, which are used in the automobile parts and tub/ shower industries respectively. However, study of the effect of such filler addition is necessary to ensure that the mechanical properties of the composites are not affected adversely by the addition of such fillers.

The importance of mechanical and tribological properties has convinced many researchers to study the friction and wear behaviour and to improve the wear resistance of polymer based composites. From the above mentioned literature it is understood that there is tremendous potentials of these fillers used for research on vinyl ester composites and studied under dry sliding conditions. Since, the purpose of this paper is to study the mechanical and dry sliding wear behaviour of fillers $CaCO_3$ and $CaSO_4$ filled vinyl ester composites. Taguchi method is used to optimize the process parameters of sliding wear in order to reduce the number of experiments without sacrificing the information.

2. Experimental Details

2.1. Specimen Preparation

The matrix used in this work is vinyl ester resin (density 1.28 gm/cc) was supplied by Northern Polymer Pvt. Ltd. New Delhi. Methyl ethyl ketone peroxide (MEKP- 1.5%), Cobalt Naphthenate (1.5%) was used as catalyst and accelerator respectively. Three different types of composites are prepared for the study. The Calcium Sulfate ($CaSO_4$) and Calcium Carbonate ($CaCO_3$) having particle size 1.813 μm and 1.620 μm respectively were used as a filler material collected from the Pioneer Chemical Corporation, Delhi.

The composites are made homogeneously. Firstly they are properly sterilized in a jar and then simply pour into the 10mm diameter test tubes. There are three different types of composites are made for the current study with 0, 10 and 20 wt.% of the filler content. The accelerator Cobalt Naphthenate 1.5% is mixed thoroughly in vinyl ester resin and then catalyst 1.5% Methyl ethyl ketone peroxide (MEKP) was mixed in the resins prior to reinforcement. Before pouring the composite solution in the test tube, the test tube is sprayed with a release agent (Silicon spray) to ensure that the part will not adhere to the test tube after the curing of the composites samples. The cast of each composite is cured for 24 hour at room temperature before it is removed from the test tube. The other composite samples with fillers $CaCO_3$ and $CaSO_4$ of fixed weights (10 wt.% and 20 wt.%) percentage were fabricated by the same technique. The fillers $CaSO_4$ and $CaCO_3$ were mixed thoroughly in the vinyl ester resin mechanically before pouring into the test tubes. The composites prepared for this study are designated as $WCGV_1$, $WCGV_2$, $WCGV_3$, $WCGV_4$, and $WCGV_5$ respectively. The composition and designation of the composites prepared for this study are listed in Table 1.

Table 1. Composition and designation of the composites

Designation	Detail composition
$WCGV_1$	Vinyl ester
$WCGV_2$	Vinyl ester +10 wt.% $CaSO_4$
$WCGV_3$	Vinyl ester +20 wt.% $CaSO_4$
$WCGV_4$	Vinyl ester +10 wt.% $CaCO_3$
$WCGV_5$	Vinyl ester +20 wt.% $CaCO_3$

2.2. Friction and Wear Test Apparatus

Dry sliding wear tests were conducted on a pin-on-disc friction and wear monitoring test rig (DUCOM) as per ASTM G 99. The cylindrical pin specimens of 10mm diameter and 30mm length were tested against a disc made of hardened ground steel (EN-32, hardness 72HRC, surface roughness R_a= 0.07 μm). The specimen was held stationary and the disc was rotated while a normal force was applied through a lever mechanism. The schematic diagram of the pin-on-disc apparatus is shown in the Figure 1. During the test, frictional force was measured by the transducer mounted on the loading arm. The frictional force readings were taken as the average of 100 readings of every 40 seconds for the required time period. For this purpose a microprocessor controlled data acquisition system was used. The average mass loss was used to calculate to the specific wear rate (K_s). The tests were conducted with sliding velocity (1.57, 2.62, 3.67 m/sec.), normal load (20, 40, 60 N), filler content (0, 10, 20 %) for the sliding distance of 1000, 3000 and 5000 m. Sliding wear data reported here is the average of two runs. The initial weight before run and final weight after run is measured using a precision electronic balance with an accuracy of ± 0.01 mg. The specific wear rate (mm^3/Nm) is then expressed on 'volume loss' basis.

$$K_s = \frac{\Delta m}{L\, \rho\, F_n}$$

Table 2. Parameters setting and levels for various control factors for wear test

Control Factors	Symbols	Units	Levels		
			I	II	III
Velocity	A	m/sec.	1.57	2.62	3.67
Normal Load	B	N	20	40	60
Filler Content	C	%	0	10	20
Sliding Distance	D	m	1000	3000	5000

Where, K_s is the specific wear rate (mm^3/Nm), Δm is the mass loss in the test duration in gm, ρ is the density of the composite (gm/cm^3), F_n is the applied normal load (N), L is the sliding distance (m). The parameters setting and levels for various control factors for wear test are shown in the Table 2.

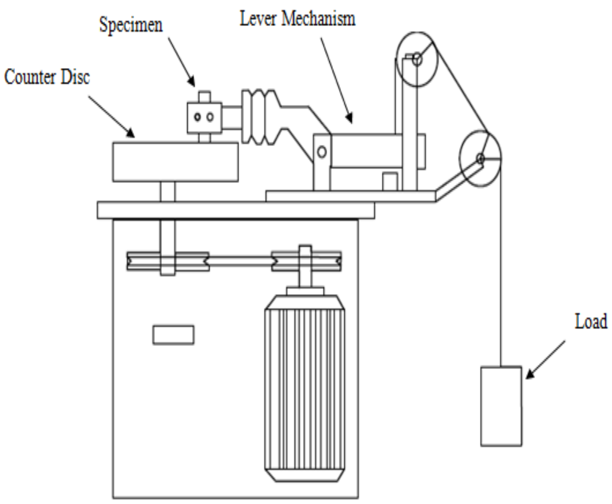

Figure 1. Schematic diagram of pin-on disc apparatus

2.3. Scanning Electron Microscopy

A FEI quanta FEG450 was used to analyze the worn surfaces of the polymer composites. The composite samples are mounted on stubs with gold plating. To enhance the conductivity of the samples, thin films of platinum are vacuum evaporated onto them before the photomicrographs were taken.

2.4. Experimental Design

Taguchi design of experiment is a powerful analysis tool which is adopted for optimizing design parameters. Taguchi method provides the designer with a systematic and efficient approach for experimentation to determine near optimum settings of design parameters for performance, quality and cost[22-25]. The most important stage in the design of experiment lies in the selection of the control factors. In the present work, the impact of the four such factors are studied using L_{27} (3^{13}) orthogonal array which has 27 rows corresponding to the number of tests (20 degree of freedom) with 13 columns at three levels. The operating conditions under which sliding wear tests carried out are given in the Table 2.

In conventional full factorial experimental design, it would require $3^4 = 81$ runs to study four factors each at three levels whereas, Taguchi's factorial experiment approach reduces it to only 27 runs offering a great advantage in terms of experimental time and cost. The experimental observations are transformed into a signal-to-noise (S/N) ratio. There are three S/N ratios available depending upon the type of characteristics (smaller-the-better, larger-the-better, nominal-the better). The S/N ratio for minimum (friction and wear rate) coming under smaller is better characteristic, which can be calculated as logarithmic transformation of the loss function as shown below[26]

$$\frac{S}{N} = -10 \log \left[\frac{1}{n} (y_1^2 + y_2^2 + \cdots y_n^2 \right] \quad (1)$$

Where 'n' is the repeated number trial conditions and y_1, y_2........y_n are the response of the friction and sliding wear characteristics. "Lower is better" (LB) characteristic, with the above S/N ratio transformation is suitable for minimizations of coefficient of friction and specific wear rate. The standard linear graph is used to assign the factors and interactions to various columns of the orthogonal array (OA).

The plan of experiments is as follows: the first column is assigned to the velocity (A), the second column to normal load (B), the fifth column to filler content (C) and the ninth column to sliding distance (D) the third and fourth column are assigned to $(A\times B)_1$ and $(A\times B)_2$ respectively to estimate interaction between the velocity (A) and the normal load (B), the sixth and seventh column are to $(B\times C)_1$ and $(B\times C)_2$ respectively to estimate the interaction between the normal load (B) and the filler content (C), the eight and eleventh column are assigned to $(A\times C)_1$ and $(A\times C)_2$ respectively to estimate the interaction between the velocity (A) and the filler content (C) and the remaining columns are used to estimate the experimental error. The linear graph for L_{27} array is shown in the Figure 2.

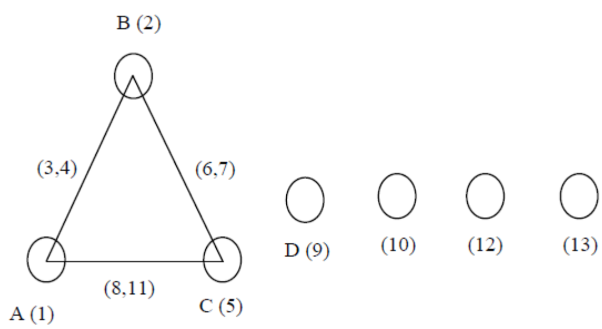

Figure 2. Linear graphs for L_{27} array

3. Results and Discussion

The characterization of the composites reveals that inclusion of any particulate filler has very strong influence not only on the mechanical properties of composites but also on their sliding wear behavior. By incorporating these particulate fillers into the vinyl ester matrix, synergistic effects, as expected were achieved in the form of modified mechanical properties and improved sliding wear resistance. A comparative study of modified behavior of the composites against the two different types of fillers is presented.

3.1. Density

The composite under this investigation consists of three components such as matrix, fiber and particulate filler. Hence, the density of the composite can be calculated using rule-of-mixture as shown in the following expression Agarwal and Broutman[27].

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

Where, W and ρ represents the weight fraction and density respectively. The suffix m, f, and p stand for the matrix, fiber and particulate filler respectively.

The actual or experimental density ρ_{exp} of the composite, however, can be determined by simple water immersion technique (Archimedes principle). The volume fraction of voids (V_v) in the composites is calculated using the following equation:

$$V_v = \frac{\rho_{ct} - \rho_{exp}}{\rho_{exp}} \quad (3)$$

Table 3. Composite designations and their experimental and theoretical densities

Composite Designation	Composite Compositions	Experimental Density (g/cm^3)	Theoretical Density (g/cm^3)	Void Fraction
WCGV$_1$	Vinyl ester	1.2738	1.2800	0.4844
WCGV$_2$	Vinyl ester + 10 wt.% $CaSO_4$	1.3307	1.3570	1.9381
WCGV$_3$	Vinyl ester + 20 wt.% $CaSO_4$	1.3891	1.4439	3.7953
WCGV$_4$	Vinyl ester + 10 wt.% $CaCO_3$	1.3400	1.3513	0.8362
WCGV$_5$	Vinyl ester + 20 wt.% $CaCO_3$	1.4022	1.4311	2.0194

It can be noticed from Table 3 that composite density values calculated from weight fractions using Eq. (2) are not in agreement with the experimentally determined values. The difference between the composite or theoretical density and the actual or experimental density is a measure of voids and pores present in the composites. It is clear from the Table 3 that volume fraction of voids is small in WCGV$_1$ due to absence of particulate fillers in it. As the filler content of $CaSO_4$ and $CaCO_3$ composites (WCGV$_2$-WCGV$_5$) increases from 10 wt.% to 20 wt.% the volume fractions of voids increases as shown in the Table 3. The voids are more in the $CaSO_4$ filler as comparison to the $CaCO_3$ filler. This may due to the particle size variations, because the particle size of the $CaSO_4$ (1.813 μm) and $CaCO_3$ (1.620 μm) is higher respectively. The voids significantly affect some of the mechanical properties and even the performance of composites. Higher void contents usually mean lower fatigue strength, greater susceptibility to water penetration and weathering[27]. The knowledge of the void content is usually important for the estimation of the quality of the composites.

3.2. Mechanical Properties

The experimental values of the properties of the particulate filled $CaCO_3$ and $CaSO_4$ composites under this investigation are presented in Table 4.

Table 4. Mechanical properties of the composites

Composites	Tensile strength (MPa)	Tensile Modulus (GPa)	Flexural strength (MPa)	Compressive Strength (MPa)	Hardness (HRB)
WCGV$_1$	39.04	0.583	103.15	9.51	17.5
WCGV$_2$	21.65	0.797	69.45	8.81	21.3
WCGV$_3$	18.58	0.808	59.0	6.27	10.2
WCGV$_4$	24.81	0.698	101.75	8.05	22.15
WCGV$_5$	20.77	0.744	107.6	10.52	19.6

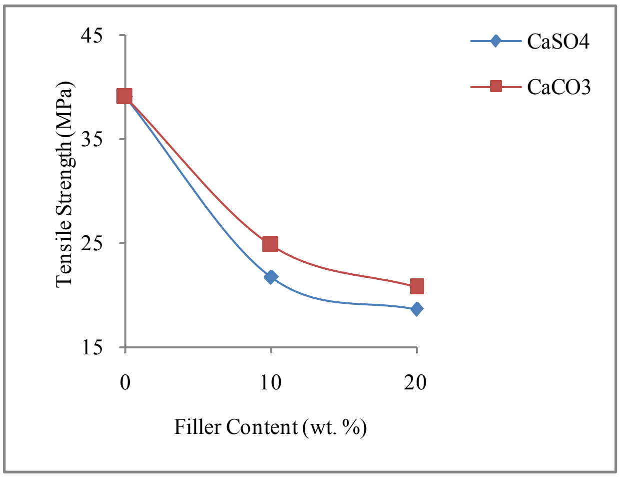

Figure 3. Variation of tensile strength of composites with filler type and content

The tensile test is performed on the universal testing machine (UTM) Hounsfield H25KS as per ASTM standard D 3039-76[28]. It is seen that in all the samples irrespective of the filler material the tensile strength of the composite decreases with increase in filler content. The pure vinyl ester has strength of 39.04 MPa in tension and it may be seen from Table 4 that this value drops to 21.65 MPa and 18.58 MPa with addition of 10 wt.% and 20 wt.% of $CaSO_4$ filler respectively. The same phenomenon has been seen in $CaCO_3$ filler, the values are drops to 24.81 MPa to 20.77 MPa with the addition of the 10 wt.% and 20 wt.% of $CaCO_3$ filler.

Among the two fillers taken in this study, the inclusion of $CaSO_4$ filler causes maximum reduction in the composite strength. It may occurs due to the interface bonding between the vinyl ester matrix and $CaSO_4$ filler is not good to transfer the tensile stress as that in $CaCO_3$ filler content. The one more reason is that the corner points of the irregular shaped particulates result in stress concentration in the vinyl ester matrix.

Now, it is interesting to note that the properties for tensile modulus is increasing with the addition of the both the filler $CaSO_4$ and $CaCO_3$ at 10 wt.% and 20 wt.% respectively as shown in Figure 4.

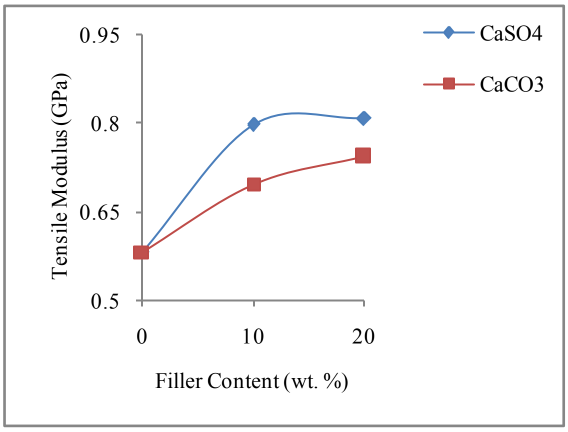

Figure 4. Variation of tensile modulus of composites with filler type and content

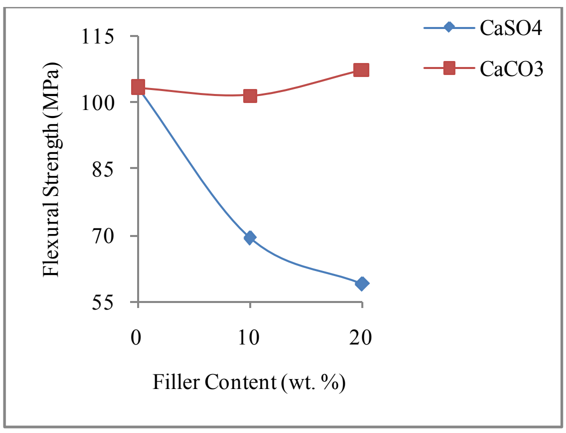

Figure 5. Variation of flexural strength of composites with filler type and content

The flexural test is conducted on the same UTM as per ASTM standard D 2344-84[29]. Figure 5 shows the comparison of flexural strengths of the composites obtained experimentally from the 3-point bend tests for composites ($WCGV_1$-$WCGV_5$). The flexural strength for the filler $CaCO_3$ is increasing with the addition of the 10 wt.% to 20 wt.% filler content. But, for the filler $CaSO_4$ it decreases with the addition of the filler content. Now, from the results it may now be suggested that $CaCO_3$ is potential candidate to be used as filler in making high flexural strength composites with the increase of the reinforcement of the filler in comparison to $CaSO_4$ filler. The flexural properties are of great importance for any structural element. Composite materials used in structures are prone to fail in bending and therefore the development of new composites with improved flexural characteristics is essential. From the results it may now be suggested that $CaCO_3$ is potential candidate to be used as filler in making high flexural strength composites with the increase of the reinforcement of the filler in comparison to $CaSO_4$ filler. $CaSO_4$ holds good flexural strength at 10 wt.% filler content also more than $WCGV_1$ and $WCGV_4$ composites as shown in Figure 5. There may be one reason for this that the voids in $WCGV_2$ (1.9381) is more in comparison to $WCGV_4$ (0.8362).

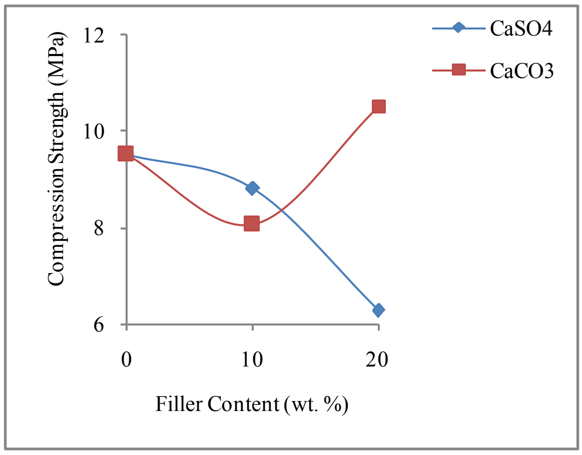

Figure 6. Variation of compression strength of composites with filler type and content

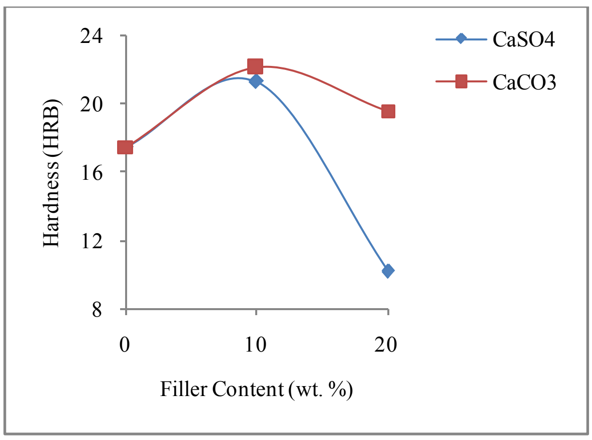

Figure 7. Variation of Rockwell hardness of composites with filler type and content

Figure 6 shows the compressive strengths of the composites obtained experimentally for the composites ($WCGV_1$-$WCGV_5$). The compressive strength for filler $CaSO_4$ decreases continuously with the addition of the 10 wt.% and 20 wt.% filler content and for filler $CaCO_3$, it firstly decreases at the addition of 10 wt.% filler content and then increases with the addition of 20 wt.% filler content as shown in the Figure 6.

Figure 7 shows the hardness of the composites ($WCGV_1$-$WCGV_5$) as obtained from the experiments. For composite $WCGV_1$, the hardness is recorded as 17.5 HRB while for $WCGV_2$ and $WCGV_3$ the values are recorded as 21.3 HRB and 10.2 HRB respectively. On the other hand the values for composites $WCGV_4$ and $WCGV_5$ are recorded as 22.15 HRB and 19.6 HRB respectively. From the analysis of the results we observed that the hardness of the $CaCO_3$ filler is more as comparison to $CaSO_4$.

3.3. Analysis of Experimental Results

The experimental data for coefficient of friction and specific wear rate (K_s) for $CaCO_3$ and $CaSO_4$ fillers is reported in the Table 5 and 8 respectively. The data reported here is the average of two runs. From Table 5 the overall mean for the S/N ratio of the coefficient of friction and the specific wear rate for $CaCO_3$ are found to be 4.1395 db and 92.5339 db respectively.

On the other hand, from Table 8 the overall mean for the S/N ratio of the coefficient of friction and the specific wear rate for $CaSO_4$ are found to be 3.3670 db and 89.8949 db respectively. Here, we saw that the overall mean for the S/N ratio of the coefficient of friction and specific wear rate is more in $CaCO_3$ in comparison to $CaSO_4$ which means that the coefficient of friction and specific wear rate is less in $CaCO_3$. The analysis of the experimental data is carried using the software MINITAB 16 specially used for the design of experiment applications. Before analyzing the experimental data using this software for predicting the measure of performance, the possible interaction between control factors are considered. Thus factorial design incorporates a simple means of testing for the presence of the interaction effects.

Table 5. Experimental design for $CaCO_3$ using L_{27} array

Velocity (m/sec.)	Normal Load (N)	Filler Content (%)	Sliding Distance (m)	COF (μ)	S/N Ratio (db)	Wear (mm^3/Nm)	S/N Ratio (db)
1.57	20	0	1000	0.70	3.09804	0.0000771	82.259
1.57	20	10	3000	0.59	4.58296	0.0000462	86.707
1.57	20	20	5000	0.54	5.35212	0.0000384	88.313
1.57	40	0	3000	0.75	2.49877	0.0000853	81.381
1.57	40	10	5000	0.58	4.73144	0.0000386	88.268
1.57	40	20	1000	0.45	6.93575	0.0000273	91.277
1.57	60	0	5000	0.80	1.93820	0.0000932	80.612
1.57	60	10	1000	0.47	6.55804	0.0000312	90.117
1.57	60	20	3000	0.39	8.17871	0.0000262	91.634
2.62	20	0	3000	0.78	2.15811	0.0000825	81.671
2.62	20	10	5000	0.69	3.22302	0.0000474	86.484
2.62	20	20	1000	0.64	3.87640	0.0000333	89.551
2.62	40	0	5000	0.83	1.61844	0.0000952	80.427
2.62	40	10	1000	0.59	4.58296	0.00000822	101.703
2.62	40	20	3000	0.50	6.02060	0.00000493	106.143
2.62	60	0	1000	0.75	2.49877	0.0000858	81.330
2.62	60	10	3000	0.48	6.37518	0.00000792	102.025
2.62	60	20	5000	0.46	6.74484	0.00000423	107.473
3.67	20	0	5000	0.85	1.41162	0.0000982	80.158
3.67	20	10	1000	0.76	2.38373	0.00000863	101.280
3.67	20	20	3000	0.61	4.29340	0.00000561	105.021
3.67	40	0	1000	0.78	2.15811	0.0000832	81.598
3.67	40	10	3000	0.66	3.60912	0.00000606	104.351
3.67	40	20	5000	0.60	4.43697	0.00000451	106.916
3.67	60	0	3000	0.80	1.93820	0.0000942	80.519
3.67	60	10	5000	0.57	4.88250	0.00000411	107.723
3.67	60	20	1000	0.52	5.67993	0.00000212	113.473

Figure 8 and 9 for $CaCO_3$ shows graphically the effect of four control factors on coefficient of friction and specific wear rate of the composite specimens $WCGV_1$, $WCGV_4$ and $WCGV_5$. The analysis of the results gives the combination factors resulting in minimum coefficient of friction and specific wear rate of the composites. Analysis of these results leads to the conclusion that factors combination A_1, B_3, C_3 and D_2 gives minimum coefficient of friction as shown in the Figure 8. It is observed that the interaction B×C shows significant effect on the coefficient of friction. Similarly the combination factors A_3, B_3, C_3 and D_2 gives minimum specific wear rate as shown in the Figure 9. It is observed that interaction A×C has significant effect on the specific wear rate.

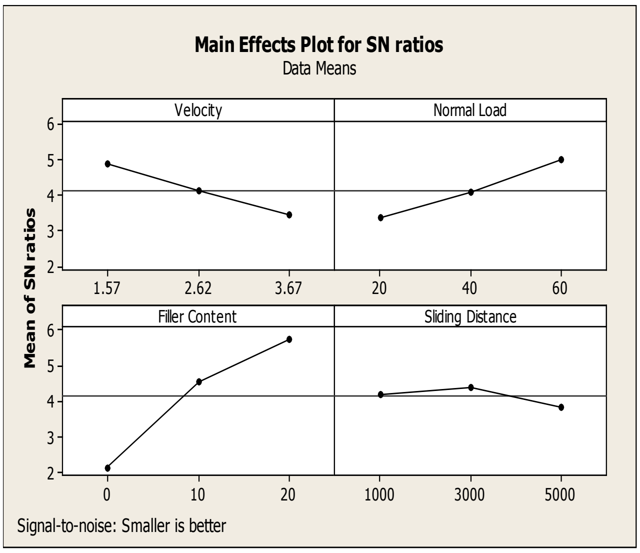

Figure 8. Effect of control factor on Coefficient of friction. (For $CaCO_3$)

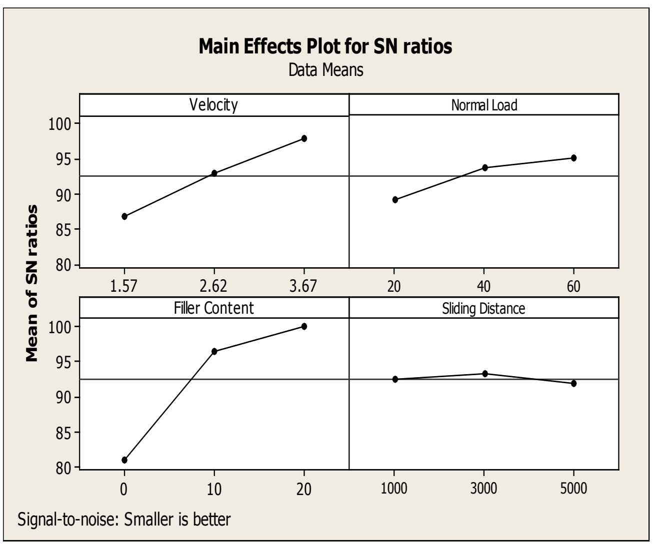

Figure 9. Effect of control factor on Specific wear rate. (For $CaCO_3$)

For $CaSO_4$ Figure 10 and 11 shows graphically the effect of four control factors on coefficient of friction and specific wear rate of the composite specimens $WCGV_1$, $WCGV_2$ and $WCGV_3$. Analysis of these results leads to the conclusion that factors combination A_1, B_3, C_2 and D_1 gives minimum coefficient of friction as shown in the Figure 10. It is observed that the interaction B×C again shows significant effect on the coefficient of friction as in case of $CaCO_3$ filler. The combination factors A_3, B_3, C_2 and D_1 gives minimum specific wear rate as shown in the Figure 11. It is observed that interaction A×C has significant effect on the specific wear rate.

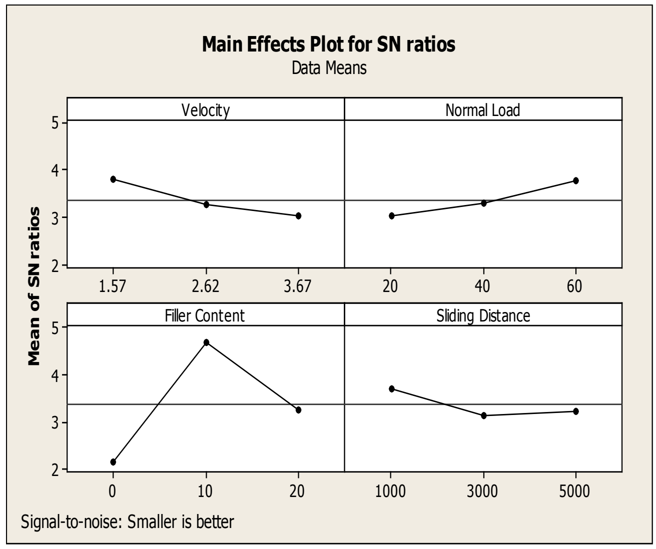

Figure 10. Effects of control factor for coefficient of friction. (For $CaSO_4$)

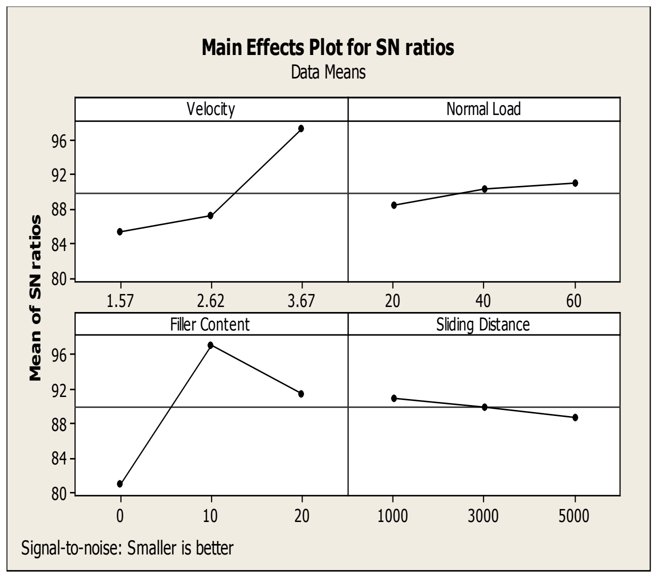

Figure 11. Effect of control factor on Specific wear rate. (For $CaSO_4$)

3.4. ANOVA and Effects of Factors

In order to understand the impact of various control factors like velocity (A), normal load (B), filler content (C) and sliding distance (D) and interaction on the response of experimental data it is desirable to develop the analysis of variance (ANOVA) to find the significant factors as well as interactions. ANOVA allows analyzing the influence of each variable on the total variance of the results. For $CaCO_3$, Table 6a shows the results of ANOVA for the specific wear rate and Table 7a shows the results of ANOVA for coefficient of friction and for $CaSO_4$, Table 9a shows the results of ANOVA for the specific wear rate and Table 10a shows the results of ANOVA for coefficient of friction. The analyses are performed with a level of significance 5% means at 95% level of confidence. In ANOVA table, the column shows the percentage contribution (P) of each variable in the total variation indicating the influence of specific wear rate and coefficient of friction.

For filler $CaCO_3$ it can be observed from the ANOVA Table 6a for specific wear rate that the filler content (P=57.981%), velocity (P=17.981%), normal load (P=5.523%) and the interactions A×C (P=10.218%), B×C (P=3.283) and A×B (P=3.097%) has significant influence on the specific wear rate. However, the control factor sliding distance (P=0.303%) does not have a significant effect (both physically and statistically) on specific wear rate as their values are quit smaller than error (P=1.613%) so they are neglected. From the analysis of ANOVA and response Table 6b of the S/N ratio of specific wear rate, it is observed that the control factor filler content (C) has major impact on the specific wear rate followed by velocity (A), normal load (B) and sliding distance (D). It means that with increasing the filler content, velocity and normal load the specific wear rate decreases i.e., increase the wear resistance as observed from the Figure 9.

Table 6a. ANOVA table for Specific wear rate. (For $CaCO_3$)

Source	DF	Seq SS	Adj SS	Adj MS	F	P (%)
A	2	563.46	563.46	281.732	33.45	17.981
B	2	173.08	173.08	86.540	10.27	5.523
C	2	1816.92	1816.92	908.462	107.86	57.981
D	2	9.51	9.51	4.754	0.56	0.303
A*B	4	97.06	97.06	24.266	2.88	3.097
A*C	4	320.19	320.19	80.047	9.50	10.218
B*C	4	102.87	102.87	25.718	3.05	3.283
Residual Error	6	50.54	50.54	8.423		1.613
Total	26	3133.63				100.00

Table 6b. Response table for Specific wear rate. (For $CaCO_3$)

Level	A	B	C	D
1	86.73	89.05	81.11	92.51
2	92.98	93.56	96.52	93.27
3	97.89	94.99	99.98	91.82
Delta	11.16	5.94	18.87	1.45
Rank	2	3	1	4

Table 7a. ANOVA table for Coefficient of friction. (For $CaCO_3$)

Source	DF	Seq SS	Adj SS	Adj MS	F	P (%)
A	2	9.5096	9.5096	4.7548	33.39	10.265
B	2	11.6173	11.6173	5.8086	40.79	12.540
C	2	59.8532	59.8532	29.9266	210.15	64.607
D	2	1.6144	1.6144	0.8072	5.67	1.743
A*B	4	0.6990	0.6990	0.1748	1.23	0.755
A*C	4	1.7495	1.7495	0.4374	3.07	1.889
B*C	4	6.7443	6.7443	1.6861	11.84	7.279
Residual Error	6	0.8544	0.8544	0.1424		0.922
Total	26	92.6416				100.00

Table 7b. Response table for coefficient of friction. (For $CaCO_3$)

Level	A	B	C	D
1	4.875	3.375	2.146	4.197
2	4.122	4.066	4.548	4.406
3	3.422	4.977	5.724	3.815
Delta	1.453	1.602	3.578	0.591
Rank	3	2	1	4

In the same way from the ANOVA Table 7a for coefficient of friction the filler content (P=64.607%), normal load (P=12.540%), velocity (P=10.265%), sliding distance (P=1.743%) and the interactions B×C (P=7.279%), A×C (P=1.889%) has significant effect on the coefficient of friction. But, interaction A×B (P=0.755%) does not have a significant effect (both physically and statistically) on coefficient of friction as its value is quit smaller than error (P=0.922%) so it may neglected. So, from the analysis of ANOVA and response Table 7b of the S/N ratio of coefficient of friction, it is observed that the filler content (C) has major influence followed by normal load (B), velocity (A) and sliding distance (D) as for the coefficient of friction.

For filler $CaSO_4$, it can be observed from the ANOVA Table 9a for specific wear rate that filler content (P=44.375%), velocity (P=27.788%) and the interactions A×C (P=20.423%) and B×C (P=2.474%) has significant effect on specific wear rate. However, the control factors normal load (P=1.326%), sliding distance (P=0.815%) and interaction A×B (P=0.859%) do not have a significant effect (both physically and statistically) on specific wear rate as their values are quit smaller than residual error (P=1.941%) so they are neglected. From the analysis of ANOVA and response Table 9b of the S/N ratio for specific wear rate, it is observed that the control factor filler content (C) has major impact on the specific wear rate followed by velocity (A), the normal load (B) and sliding distance (D). It means that for filler $CaSO_4$, with the increases of the filler content, velocity and normal load the specific wear rate decreases i.e., the wear resistance is good as observed from the Figure 11. But from the Figure 11 we also observed that for $CaSO_4$ the filler content plays adverse effect when filler content is increases from 10 wt.% to 20 wt.%. At 10 wt.% for $CaSO_4$ the specific wear rate is decreased and for 20 wt.%, it further increases.

Table 8. Experimental design for $CaSO_4$ using L_{27} array

Velocity (m/sec.)	Normal Load (N)	Filler Content (%)	Sliding Distance (m)	COF (μ)	S/N Ratio (db)	Wear (mm^3/Nm)	S/N Ratio (db)
1.57	20	0	1000	0.70	3.09804	0.0000771	82.259
1.57	20	10	3000	0.60	4.43697	0.0000432	87.290
1.57	20	20	5000	0.63	4.01319	0.0000483	86.321
1.57	40	0	3000	0.75	2.49877	0.0000853	81.381
1.57	40	10	5000	0.57	4.88250	0.0000373	88.566
1.57	40	20	1000	0.64	3.87640	0.0000498	86.055
1.57	60	0	5000	0.80	1.93820	0.0000932	80.612
1.57	60	10	1000	0.48	6.37518	0.0000325	89.762
1.57	60	20	3000	0.70	3.09804	0.0000535	85.433
2.62	20	0	3000	0.78	2.15811	0.0000825	81.671
2.62	20	10	5000	0.68	3.34982	0.0000458	86.783
2.62	20	20	1000	0.72	2.85335	0.0000588	84.612
2.62	40	0	5000	0.83	1.61844	0.0000952	80.427
2.62	40	10	1000	0.57	4.88250	0.00000812	101.809
2.62	40	20	3000	0.75	2.49877	0.0000685	83.286
2.62	60	0	1000	0.75	2.49877	0.0000858	81.330
2.62	60	10	3000	0.52	5.67993	0.00000783	102.125
2.62	60	20	5000	0.64	3.87640	0.0000786	82.092
3.67	20	0	5000	0.85	1.41162	0.0000982	80.158
3.67	20	10	1000	0.64	3.87640	0.00000652	103.715
3.67	20	20	3000	0.79	2.04746	0.00000785	102.103
3.67	40	0	1000	0.78	2.15811	0.0000832	81.598
3.67	40	10	3000	0.63	4.01319	0.00000558	105.067
3.67	40	20	5000	0.69	3.22302	0.00000582	104.702
3.67	60	0	3000	0.80	1.93820	0.0000942	80.519
3.67	60	10	5000	0.58	4.73144	0.00000333	109.551
3.67	60	20	1000	0.64	3.87640	0.00000401	107.937

Table 9a. ANOVA Table for Specific wear rate. (For $CaSO_4$)

Source	DF	Seq SS	Adj SS	Adj MS	F	P (%)
A	2	747.54	747.54	373.769	42.95	27.788
B	2	35.66	35.66	17.831	2.05	1.326
C	2	1193.77	1193.77	596.883	68.58	44.375
D	2	21.93	21.93	10.967	1.26	0.815
A*B	4	23.11	23.11	5.777	0.66	0.859
A*C	4	549.40	549.40	137.351	15.78	20.423
B*C	4	66.55	66.55	16.636	1.91	2.474
Residual Error	6	52.22	52.22	8.703		1.941
Total	26	2690.17				100.00

Table 9b. Response table for Specific wear rate. (For $CaSO_4$)

Level	A	B	C	D
1	85.30	88.32	81.11	91.01
2	87.13	90.32	97.19	89.88
3	97.26	91.04	91.39	88.80
Delta	11.96	2.72	16.08	2.21
Rank	2	3	1	4

Table 10a. ANOVA table for Coefficient of friction. (For $CaSO_4$)

Source	DF	Seq SS	Adj SS	Adj MS	F	P (%)
A	2	2.8080	2.8080	1.4040	6.47	6.655
B	2	2.6152	2.6152	1.3076	6.02	6.199
C	2	29.3058	29.3058	14.6529	67.47	69.460
D	2	1.7235	1.7235	0.8618	3.97	4.085
A*B	4	1.7612	1.7612	0.4403	2.03	4.174
A*C	4	0.1993	0.1993	0.0498	0.23	0.472
B*C	4	2.4749	2.4749	0.6187	2.85	5.866
Residual Error	6	1.3030	1.3030	0.2172		3.088
Total	26	42.1908				100

Table 10b. Response table for coefficient of friction. (For $CaSO_4$)

Level	A	B	C	D
1	3.802	3.027	2.146	3.722
2	3.268	3.295	4.692	3.152
3	3.031	3.779	3.263	3.227
Delta	0.771	0.752	2.546	0.570
Rank	2	3	1	4

In the same way from the ANOVA Table 10a for coefficient of friction the filler content (P=69.460%), velocity (P=6.655%), normal load (P=6.199%), sliding distance (P=4.085%) and the interactions B×C (P=5.866%) and A × B (P=4.174%) has significant effect on the coefficient of friction. However, the interaction A × C (P=0.472%) do not have significant effect on the coefficient of friction as their values are quite smaller than the residual error (P=3.088%), so it may neglected. From the analysis of the ANOVA and the response Table 10b for coefficient of friction it is observed that the filler content (C) has major influence followed by the velocity (A) normal load (B) and the sliding distance (D). From the Fig. 10 for $CaSO_4$, it is observed that the coefficient of friction is also increase with the increase of the filler content. It means that in 10 wt.% the coefficient of friction is less as comparison to 20 wt.% i.e., the reinforcement of the $CaSO_4$ filler at 10 wt.% is more wear resistance.

3.5. Surface Morphology

Figures 12a-c are the SEM pictures of composites for minimum, maximum and nominal wear test conditions for $CaCO_3$ filled vinyl ester composites. It has been found from the experimental analysis that the minimum wear occurs at 3.67 m/sec., 60 N, 20 wt. %, 1000 m test parameter conditions, the maximum wear occurs at 3.67 m/sec., 20 N, 0 wt. %, 5000 m test parameter conditions and the nominal wear occurs at 3.67 m/sec., 20 N, 10 wt. %, 1000 m test parameter conditions as shown in the Figures 12a-c.

The micrograph in Figure 12a shows the resinous and matrix region. The filler $CaCO_3$ covered the matrix region which results in less wear. Figure 12b shows the debris and wedge formation regions due to long sliding distance. Vinyl ester debris was adhered into the filler region and micro cracks were identified which increases the wear rate. Figure 12c showing the thin layer formation and debris which results the nominal wear.

Similarly, Figures 13a-c are the SEM pictures of composites for minimum, maximum and nominal wear test conditions for CaSO4 filled vinyl ester composites. It has been found from the experimental analysis that the minimum wear occurs at 3.67 m/sec., 60 N, 10 wt. %, 5000 m test parameter conditions, the maximum wear occurs at 3.67 m/sec., 20 N, 0 wt. %, 5000 m test parameter conditions and the nominal wear occurs at 3.67 m/sec., 20 N, 20 wt. %, 3000 m test parameter conditions as shown in the Figures 13a-c.

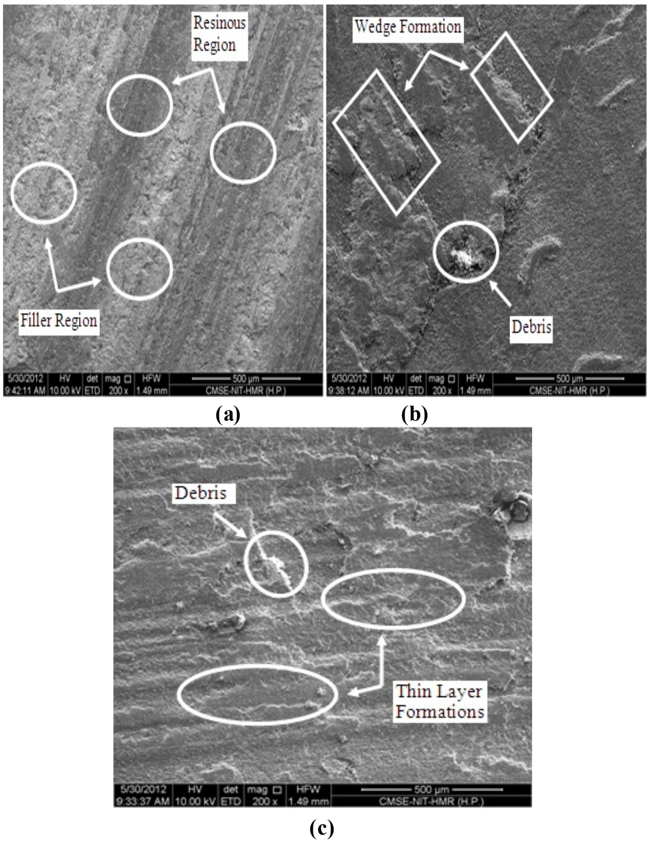

Figure 12. SEM pictures of composites at (a) 3.67 m/sec., 60N, 20 wt. %, 1000 m (b) 3.67 m/sec., 20N, 0 wt. %, 5000 m (c) 3.67 m/sec., 20N, 10 wt. %, 1000 m. (For $CaCO_3$ filled vinyl ester composites)

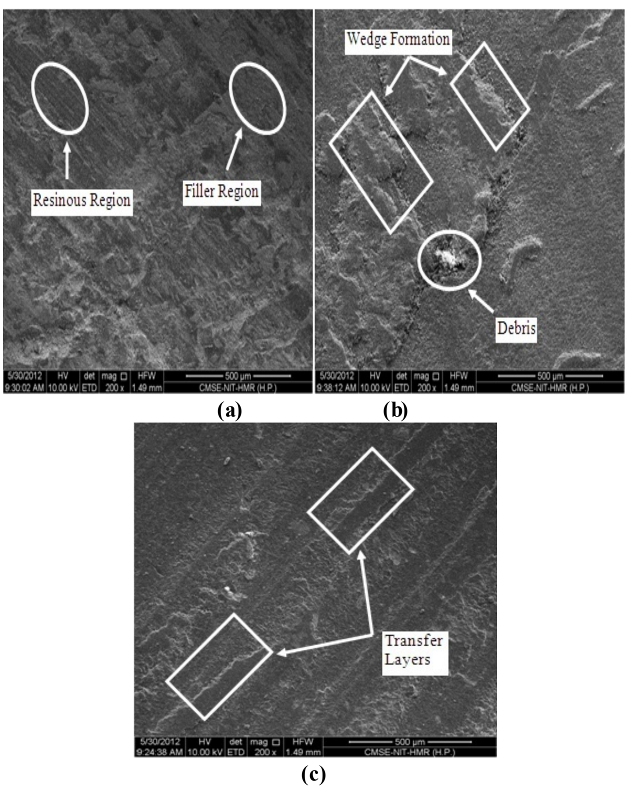

Figure 13. SEM pictures of composites at (a) 3.67 m/sec., 60N, 10 wt. %, 5000 m (b) 3.67 m/sec., 20N, 0 wt. %, 5000 m (c) 3.67 m/sec., 20N, 20 wt. %, 3000 m. (For $CaSO_4$ filled vinyl ester composites)

Figure 13a shows the resinous and filler region as in case of $CaCO_3$ which results in the lesser wear rate because the filler covers the maximum matrix region. Figure 13b found the maximum wear rate due to wedge formation and matrix debris adhered into the filler region as in the case of $CaCO_3$ which increases the wear rate. Figure 13c shows the nominal wear rate due to thin transfer layer formation.

3.6. Confirmation Experiments

The confirmation experiment is the final step in the design of experiments process. It predicts and verifies the improvements in the observed values through the use of optimal combination level of control factors. For filler $CaCO_3$, the confirmation experiment was performed by taking an arbitrary set of factor combination A_2 B_2 C_3 D_1 to predict the coefficient of friction and for specific wear rate factor setting is A_2 B_2 C_1 D_1. Now, the estimated S/N ratio for coefficient of friction can be calculated with the help of the following predictive equation.

$$\eta_{CaCO\ 3} = \overline{T} + (\overline{A}_2 - \overline{T}) + (\overline{B}_2 - \overline{T}) + (\overline{C}_3 - \overline{T}) + (\overline{D}_1 - \overline{T}) + [(\overline{A}_2\overline{C}_3 - \overline{T}] - (\overline{A}_2 - \overline{T}) - (\overline{C}_3 - \overline{T})] + [(\overline{B}_2\overline{C}_3 - \overline{T}] - (\overline{B}_2 - \overline{T}) - (\overline{C}_3 - \overline{T})] \quad (4)$$

Where, $\eta_{CaCO\ 3}$ is the predicted average of $CaCO_3$ for coefficient of friction, $\overline{T}$ is the overall experimental average $\overline{A}_2\overline{C}_3$, $\overline{B}_2\overline{C}_3$ and $\overline{D}_1$ is the mean response for factors and interactions at designed levels. By combining all the terms Eq. (4) reduces to

$$\eta_{CaCO\ 3} = \overline{A}_2\overline{C}_3 + \overline{B}_2\overline{C}_3 + \overline{D}_1 - \overline{C}_3 - \overline{T} \quad (5)$$

A new combination of factor levels A_2 B_2 C_3 D_1 are used to predict the S/N ratio of coefficient of friction through predictive Eq. (5) and is found to be $\eta_{CaCO\ 3}$= 5.6781 db. For each of performance measures an experiment is conducted for different combination of factors and results are compared with those obtained from the predictive equation as shown in the Table 11. Similarly a predictive equation is developed for estimating S/N ratio of specific wear rate as shown in Eq. (6).

$$\eta_{CaCO\ 3} = \bar{T} + (\bar{A}_2 - \bar{T}) + (\bar{B}_2 - \bar{T}) + (\bar{C}_1 - \bar{T}) + (\bar{D}_1 - \bar{T}) + [(\bar{A}_2\bar{C}_1 - \bar{T}] - (\bar{A}_2 - \bar{T}) - (\bar{C}_1 - \bar{T})] + [(\bar{B}_2\bar{C}_1 - \bar{T}] - (\bar{B}_2 - \bar{T}) - (\bar{C}_1 - \bar{T})] \quad (6)$$

Where, $\eta_{CaCO\ 3}$ is the predictive average of $CaCO_3$ for specific wear rate, $\bar{T}$ is the overall experimental average $\overline{A}_2\overline{C}_1$, $\overline{B}_2\overline{C}_1$ and $\overline{D}_1$ is the mean response for factors and interactions at designed levels. By combining all the terms Eq. (6) reduces to

$$\eta_{CaCO\ 3} = \overline{A}_2\overline{C}_1 + \overline{B}_2\overline{C}_1 + \overline{D}_1 - \overline{C}_1 - \overline{T} \quad (7)$$

A new combination of factor levels A_2 B_2 C_1 D_1 are used to predict the S/N ratio of specific wear rate through predictive Eq. (7) and is found to be $\eta_{CaCO\ 3}$= 81.1478 db. For each of performance measures an experiment is conducted for the prediction equation as shown in Table 11. The resulting equations seem to be capable of predicting the coefficient of friction and specific wear rate. An error of 6.49% for the S/N ratio of the coefficient of friction and 5.41% for the S/N ratio of the specific wear rate is observed.

Table 11. Results of the confirmation experiments for the coefficient of friction and specific wear rate. (For $CaCO_3$)

	Optimal control parameters		Error
	Prediction	Experimental	
Level	A_2 B_2 C_3 D_1	A_2 B_2 C_3 D_1	(%)
S/N ratio for coefficient of friction (db)	5.6781	6.0727	6.49
Level	A_2 B_2 C_1 D_1	A_2 B_2 C_1 D_1	(%)
S/N ratio for specific wear rate (db)	81.1478	85.7860	5.41

For filler $CaSO_4$, the confirmation experiment was performed by taking an arbitrary set of factor combination A_2 B_2 C_3 D_1 to predict the coefficient of friction and for specific wear rate factor setting is A_2 B_2 C_1 D_1 as same for filler $CaCO_3$. Now, the estimated S/N ratio for coefficient of friction can be calculated with the help of the following predictive Eq. (8).

$$\eta_{CaSO\ 4} = \bar{T} + (\bar{A}_2 - \bar{T}) + (\bar{B}_2 - \bar{T}) + (\bar{C}_3 - \bar{T}) + (\bar{D}_1 - \bar{T}) + [(\bar{A}_2\bar{B}_2 - \bar{T}] - (\bar{A}_2 - \bar{T}) - (\bar{B}_2 - \bar{T})] + [(\bar{B}_2\bar{C}_3 - \bar{T}] - (\bar{B}_2 - \bar{T}) - (\bar{C}_3 - \bar{T})] \quad (8)$$

Where, $\eta_{CaSO\ 4}$ is the predicted average of $CaSO_4$ for coefficient of friction, $\bar{T}$ is the overall experimental average $\bar{A}_2\bar{B}_2$, $\bar{B}_2\bar{C}_3$ and $\bar{D}_1$ is the mean response for factors and interactions at designed levels. By combining all the terms Eq. (8) reduces to

$$\eta_{CaSO\ 4} = \bar{A}_2\bar{B}_2 + \bar{B}_2\bar{C}_3 + \bar{D}_1 - \bar{B}_2 - \bar{T} \quad (9)$$

A new combination of factor levels A_2 B_2 C_3 D_1 are used to predict the S/N ratio of coefficient of friction through predictive Eq. (9) and is found to be $\eta_{CaSO\ 4}$= 3.2593 db. For each of performance measures an experiment is conducted for different combination of factors and results are compared with those obtained from the predictive equation as shown in the Table 12. Similarly a predictive equation is developed for estimating S/N ratio of specific wear rate as shown in Eq. (10).

$$\eta_{CaSO\ 4} = \bar{T} + (\bar{A}_2 - \bar{T}) + (\bar{B}_2 - \bar{T}) + (\bar{C}_1 - \bar{T}) + (\bar{D}_1 - \bar{T}) + [(\bar{A}_2\bar{C}_1 - \bar{T}] - (\bar{A}_2 - \bar{T}) - (\bar{C}_1 - \bar{T})] + [(\bar{B}_2\bar{C}_1 - \bar{T}] - (\bar{B}_2 - \bar{T}) - (\bar{C}_1 - \bar{T})] \quad (10)$$

Where, $\eta_{CaSO\ 4}$ is the predictive average of $CaSO_4$ for specific wear rate, $\bar{T}$ is the overall experimental average $\bar{A}_2\bar{C}_1$, $\bar{B}_2\bar{C}_1$ and $\bar{D}_1$ is the mean response for factors and interactions at designed levels. By combining all the terms Eq. (10) reduces to

$$\eta_{CaSO\ 4} = \bar{A}_2\bar{C}_1 + \bar{B}_2\bar{C}_1 + \bar{D}_1 - \bar{C}_1 - \bar{T} \quad (11)$$

A new combination of factor levels A_2 B_2 C_1 D_1 are used to predict the S/N ratio of specific wear rate through predictive Eq. (11) and is found to be $\eta_{CaSO\ 4}$= 82.2855 db. For each of performance measures an experiment is conducted for the prediction equation as shown in Table 12. The resulting equations seem to be capable of predicting the coefficient of friction and specific wear rate. An error of 9.18% for the S/N ratio of the coefficient of friction and 8.04% for the S/N ratio of the specific wear rate is observed.

Table 12. Results of the confirmation experiments for the coefficient of friction and specific wear rate. (For $CaSO_4$)

	Optimal control parameters		Error
	Prediction	Experimental	
Level	A_2 B_2 C_3 D_1	A_2 B_2 C_3 D_1	(%)
S/N ratio for coefficient of friction (db)	3.2593	3.5888	9.18
Level	A_2 B_2 C_1 D_1	A_2 B_2 C_1 D_1	(%)
S/N ratio for specific wear rate (db)	82.2855	89.4786	8.04

4. Conclusions

An experimental study has been carried out for friction and dry sliding wear of vinyl ester matrix with fillers $CaCO_3$ and $CaSO_4$ sliding against smooth stainless steel counterface using Taguchi experimental design. Taguchi's design of experiment method can be used to analyze the coefficient of friction and the dry sliding wear of polymer matrix composites as presented in this research paper. The following conclusions can be drawn from the present study:-

(1) The tensile strength for both the fillers $CaCO_3$ and $CaSO_4$ decreases with the increase of the filler content. While the tensile modulus for both the fillers increases with the increase of filler content.

(2) For filler $CaCO_3$, flexural and compressive strength increases with the increase of filler content. While, hardness is increases at 10 wt.% and then decreases at 20 wt.%.

(3) For filler $CaSO_4$, flexural and compressive strength decreases with the increase of filler content. While, the hardness is increases at 10 wt.% and then decreases at 20 wt.% as same in case of $CaCO_3$ filler content.

(4) For pure vinyl ester the coefficient of friction and specific wear rate increases with the increase of normal load, sliding velocity and sliding distance.

(5) The coefficient of friction and specific wear rate for $CaCO_3$ filler decreases with the increase of filler content. But, for filler $CaSO_4$ the coefficient of friction and specific wear rate decreases at 10 wt.% and then increases at 20 wt.%.

(6) For both the fillers $CaCO_3$ and $CaSO_4$ it is observed that the control factor filler content (C) has major impact on

the specific wear rate followed by velocity (A), normal load (B) and sliding distance (D).

(7) The predictive equations based on Taguchi approach is successfully used for the prediction of effect of four control factors and predicted results are consistent with the experimental observations.

(8) It is demonstrated that Taguchi approach based on ANOVA well reflect the effects of various factors on the friction and sliding wear loss.

The significance of this current research is to create the interest between the various young researchers towards such fillers which are good not only in the mechanical behaviour but also good in the friction and dry sliding wear behaviour.

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