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Tribological investigations of carbon nanotube-reinforced polymer (UHMWPE) nanocomposites using Taguchi methodology

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ABSTRACT: Taguchi design techniques have been applied to investigate the significant influence of various operating and design parameters, such as contact load, rotational sliding speed, and carbon nanotubes (CNTs) concentration on the tribological properties of ultra-high molecular weight polyethylene nanocomposites. Analysis of variance was conducted to discuss the significance of each of the parameters. Simple regression models were developed for wear rate as well as for the coefficient of friction (COF) of the nanocomposite. Applied normal force was found to be the dominant factor controlling the wear rate and friction coefficient. The significance of CNTs concentration on both COF and wear rate closely follow that of applied load. Rotational sliding speed has the least influence on the tribological properties of the nanocomposite. The developed model for predicting wear rate and the COF was found to give very good predictions against the experimental data. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 44018.

KEYWORDS: carbon nanotubes; composites; friction; mechanical properties; wear

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INTRODUCTION

Polymer composites are finding their way very rapidly in demanding tribological applications such as bearings, cams, gears, seals, etc., because of their high strength to weight ratio, excellent resistance to corrosive environments, and ease of fabrication.[1–3] Various high performance polymers such as polyether ether ketone (PEEK), ultra-high molecular weight polyethylene (UHMWPE), polytetrafluoro ethylene (PTFE), and polyimide (PI) have been extensively used to reinforce with different micron and nano-sized reinforcements to improve their tribological properties.[3] However, among them UHMWPE is an exceptional polymer which combines highest sliding abrasion resistance and highest notched impact strength combined with low coefficient of friction (COF). UHMWPE and its composites in both bulk and in the form of coatings showed excellent wear resistance.[4–9]

Different fillers have been used to reinforce UHMWPE to improve its performance such as carbon nanotubes (CNTs), layered silicates, and metal oxide particles. CNTs is among the most explored nanofillers to be used for almost all polymers and as well as for UHMWPE.[10–13] CNTs is known to exhibit a good mechanical strength, excellent chemical resistance, and extraordinary thermal and electrical properties.[14,15] Zoo *et al.*[11] made one of the initial efforts to investigate the effect of CNTs on tribological properties of UHMWPE matrix. Results revealed, almost 85% improvement in wear resistance of UHMWPE with COF increasing slightly. Dangsheng *et al.*[16] studied the tribological properties of HDPE/UHMWPE blend reinforced with multiwalled carbon nanotubes (MWCNTs) and concluded reduction in specific wear rate for polymer blend with addition in the range of 0.2–2 wt %. Similarly, one of studies on CNTs reinforced HDPE depicted that 5 wt % of CNTs resulted in 53% reduction in specific wear rate as well as 12% drop in COF.[17] Galtez *et al.*[18] used nano-carbon fiber as reinforcement to UHMWPE for improved mechanical and tribological properties. Carbon nano-fiber resulted in reducing the specific wear rate of UHMWPE as well as improving the mechanical properties.

Even though quite a number of studies have been conducted on evaluating the performance of CNTs reinforced UHMWPE composites, no study has revealed which of the operating and design parameters such as normal load, sliding speed, and the concentration of CNTs has the most significant effect on the tribological properties of the CNTs reinforced UHMWPE composites. The resultant wear rate and COF of UHMWPE/CNTS polymer matrix is a combined effect of more than one interacting parameter. A statistical design of experiment (DOE) can be effectively utilized to determine the significant level of each operating and design parameter affecting the performance of CNTs reinforced UHMWPE composites. Hence, the focus of the present study is to apply the Taguchi methodology to design the experiments and to evaluate the effect of three input parameters namely, normal load, rotational sliding speed, and the wt

% of CNTs on two tribological properties namely, specific wear rate and COF of the UHMWPE nanocomposites reinforced with CNTs. For this study, the smaller is the best characteristic is used. That is, smaller the wear rate and lower the COF is the desired best result.

DESIGN OF EXPERIMENTS (DOE)

In the past, the conventional optimization techniques for a system have been mainly single variable dependent, with other variables fixed. This kind of optimization approach demands huge amount of experimental runs. Additionally, data needed to correlate the object functions and decision variables can hardly reflect the interaction of the multivariable dependent process. This method is time consuming and inefficient, especially for complicated problems of multivariables with a large value domain that requires high accuracy.[19] Due to the aforementioned setbacks, statistical optimization approaches known as DOE, which includes Taguchi technique, response surface methodology (RSM), and factorial design are now widely used in place of one-factor-at-a-time experimental approach.

Taguchi methodology employs fractional factorial design matrix, and has been widely used to optimize design parameters. The technique is used to investigate how different parameters affect the mean and variance of a system performance.[20] Taguchi optimization techniques can significantly lower the total experimental time and cost.[21] The method has shown that there is no need to run full factorial experiments if one can carefully select the experimental runs. Compared with other DOE techniques, Taguchi method allows obtaining experimental results using fewer experimental runs and offers a simple and systematic approach to optimize the performance and quality.[22,23] This technique has been applied to solve some confusing problems in manufacturing, especially to observe the influence degree of the control factors and to determine optimal set of conditions.[24] One limitation of this technique is, unlike RSM which provide three-dimensional (3D) plots that helps in better visualization and understanding of the effect of operating parameters on response, the Taguchi technique can only provides the mean effect plots of response at given level of parameters. Taguchi methodology also has the possibility of not given the best optimum product, since it is a fractional factorial design matrix.

The Orthogonal array (OA) and the signal to noise ratio (*S/N* ratio) are the two major tools used in Taguchi design. The OA is a matrix of numbers arranged in rows and columns, selected from all possible combinations of the controllable factors.[25] The signal to noise ratio (*S/N*) is the ratio of sensitivity to variability. By minimizing the effect of noise factor, we are actually maximizing *S/N* ratio, thereby improving the product quality characteristic. Taguchi uses signal-to-noise ratio as the quality characteristic of choice. *S/N* ratios compare the level of a desired signal (such as music) to the level of background noise. The higher the ratio, the less obtrusive the background noise is. In other terms, *S/N* ratio can be referred to as the ratio of useful information to false or irrelevant data. Noise factors are uncontrollable in nature. The main purpose of noise factors is to cause performance of a system to deviate from its target value. In this work, the present of noise is expected since each experiment was repeated three times at the same conditions.

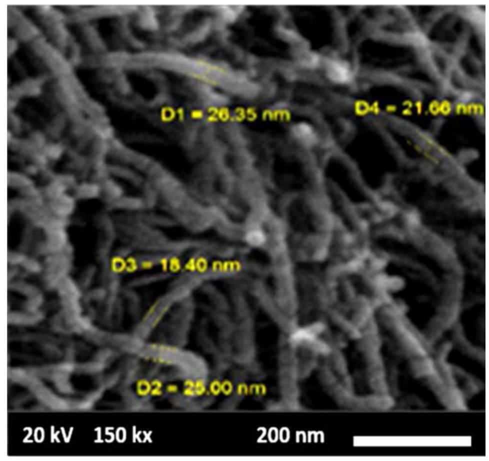

Figure 1. FE-SEM image showing CNTs used in current study. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Depending on the objective function, *S/N* ratio characteristics may be classified as; smaller is the best, nominal is the best, and larger is the best respectively. The three *S/N* ratio characteristics are given by eqs. (1–3), when the characteristic is continuous.[26,27]

For nominal is the best characteristic,

$$\frac{S}{N}=10\log\frac{\bar{y}}{S_y^2} \tag{1}$$

For smaller is the best characteristic,

$$\frac{S}{N}=-10\log\frac{1}{n}\left(\sum y^2\right) \tag{2}$$

For larger is the best characteristic,

$$\frac{S}{N}=-10\log\frac{1}{n}\left(\sum \frac{1}{y^2}\right) \tag{3}$$

where $\bar{y}$, is the average of observed data, S_y^2 is the variation of y, n is the number of observations, and y the observed data. For our case, the smaller is the best characteristic was adopted.

EXPERIMENTAL

Materials

UHMWPE in powder form with an average particle size of 80–90 µm, as supplied by Goodfellow Cambridge Ltd, the United Kingdom was used. MWCNTs with an average diameter of 25–26 nm as shown in Figure 1 have been used as the reinforcements.

Sample Preparation

UHMWPE and the required quantity of CNTs was ball milled with a ball to powder ratio of 10:1 using a high energy ball mill. A milling cycle of 20 min at a speed of 200 rpm was repeated six times, resulting in a total milling time of 2 h. The milled powders were then hot pressed. The mixture was initially compacted in the molder using a pressure of 6 MPa at room temperature. Then the temperature was raised to 170 °C and a

Table I. Parameters and Their Levels

Factors	Level 1	Level 2	Level 3
CNTs concentration (wt %)	0.5	1.5	3.0
Load (N)	30	50	70
Speed (RPM)	100	200	300

pressure of 25 MPa was applied for 15 minutes. After that, the mold was cooled to 50 °C and the pressure was released. The final samples were of a cylindrical geometry with a diameter of 30 mm and a thickness of 6 mm. The average surface roughness (R_a) value of approximately was approximately 0.928 ± 0.05 µm. Three different weight percentages of 0.5, 1.5, and 3 wt % of CNTs loadings were used to fabricate UHMWPE nanocomposites in this study.

Wear Testing

Wear tests on the prepared samples were conducted using ball on disc tribometer (UMT-3, Bruker, The United States) to simulate the contact conditions in most of the tribological applications. A 440C stainless steel ball of hardness RC 62 (as specified by the manufacturer) having a diameter of 6.3 mm was used as a counterface. Wear testing was performed according to the Taguchi methodology DOE presented in Tables I and II. After each wear test, optical images of the counterface ball along with the corresponding wear track images of the sample were recorded to investigate the wear mechanisms. Three wear tests were conducted for each sample type and the average value of the COF and the specific wear rates (WR) are reported. All the wear tests were carried out at a room temperature of 25 ± 2 °C and a relative humidity of $55 \pm 5\%$.

RESULTS AND DATA ANALYSIS

Effect of the Input Parameters on the Specific Wear Rate

The objective of the experiments is to find the significant factors and their combinations influencing the tribological properties of CNTs/UHMWPE composites. The three levels of input parameter combinations are tabulated in Table I. The L_9 (3^3) Taguchi design OA, the measured specific wear rate is presented in Table II. A total number of nine experiments were conducted, and each experiment is repeated three times to observe the effects of uncontrollable factors (*S/N* ratio) on the process.

The main effect plots for mean specific wear rate and *S/N* ratio are as depicted in Figures 2 and 3, respectively. The plots are based on average values of each experimental run, and it is effectively used to investigate the trends and influence of each factor on the performance of a process. It is obvious from the figures that 1.5 wt % of CNTs concentration gives the lowest specific wear rate. This can be attributed to the fact that the addition of CNTs to the UHMWPE composites increases the hardness of the composite and subsequently increases its wear resistance as shown in Figure 4. CNTs have superior mechanical properties in terms of strength, modulus, hardness, and stiffness,[28] which results in increased resistance to indentation. Variation of microstructure and load-carrying capacity may be considered as the other possible explanations for increase in hardness and decrease in wear loss. However, this is not the case at 3 wt % CNTs concentration which recorded larger volume of material removal as compared with that of 1.5 wt % CNTs concentration. The observed abnormality of lower wear resistance at 3 wt % CNTs concentration may be due to the non-homogeneous dispersion of CNTs into the UHMWPE polymer matrix, which results in the agglomeration of CNTs.

The impact of normal load on the wear rate is quite pronounced. It is very clear from the main effect plots that wear rate increases with increasing normal load. With increased load, the plastic deformation is expected to be more effective. Because of the increased plastic deformation, more tearing, fracturing and fragmentation of the material will take place, leading to increased material removal. Moreover, it can also be seen that the COF increases as a result of an increase in the normal load. This could be attributed to an increase in the adhesive component of the COF due to the softening of the polymer as a result of the increased localized temperatures with increasing load. The influence of sliding velocity on wear resistance is obviously of no significance within the specified speed range. This is

Table II. Experimental Results of Average Specific Wear Rate and S/N Ratio

				Responses (specific wear rate, WR) (mm^3/Nm)				
Runs	CNTs concentration (wt %)	Load (N)	Speed (RPM)	Trial 1 (mm^3/Nm)	Trial 2 (mm^3/Nm)	Trial 3 (mm^3/Nm)	Mean WR (mm^3/Nm)	S/N ratio (dB)
1	0.5	30	100	0.000220576	0.000191444	0.00020118	0.0002044	73.7752
2	0.5	50	200	0.000226744	0.000275167	0.00024964	0.0002505	71.9963
3	0.5	70	300	0.000338693	0.000324459	0.000331186	0.0003314	69.5904
4	1.5	30	200	9.26576E-05	9.21111E-05	9.09643E-05	0.0000919	80.7324
5	1.5	50	300	0.000134549	0.000119437	0.000120263	0.0001248	78.0658
6	1.5	70	100	0.000230471	0.000209426	0.000223886	0.0002213	73.0951
7	3	30	300	0.000150292	0.000150089	0.000149889	0.0001501	76.4730
8	3	50	100	0.000247268	0.000233855	0.000242342	0.0002412	72.3518
9	3	70	200	0.000287161	0.000282933	0.000278695	0.0002829	70.9658

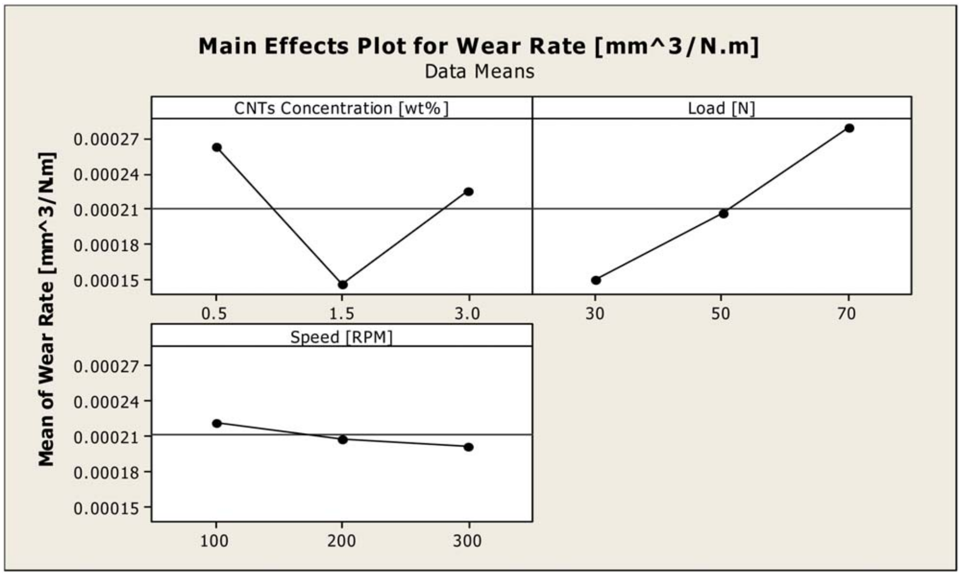

Figure 2. Main effect plot for mean wear rate. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

because wear rate is independent of the sliding velocity according to wear equations ($v = kWx/H$), where v is the volume of wear being worn away, k is a non-dimensional wear coefficient, W is the applied normal load, x is the sliding distance, and H is the hardness of the surface being worn away.

To evaluate the relative influence of each operating parameter on wear rate, analysis of variance (ANOVA) was performed in Minitab 16 program, and the results are presented in Tables III and IV. While Table III summarizes the analysis of variance for WR *S/N* ratios, Table IV provide the summary of the ANOVA for mean WR. The ANOVA analysis was conducted at 95% confidence level (using a level of significance $\alpha = 0.05$). ANOVA is used to test differences between two or more means by analyzing variance. It tests general differences among means. Null hypothesis and alternative hypothesis are tested using ANOVA.

From Table III, both contact load and CNTs concentration has a *P*-value of 0.006 each, which is less than 0.05. This is a clear indication that both the operating parameters have significant effect on the variation in wear rate. The sliding rotational speed is considered to be statistically insignificant when compared with contact load and CNTs concentration, since it's *P*-value is 0.059, which is greater than 0.05. In Table IV, the *P*-value of contact load and CNTs concentration are 0.02 and 0.024, respectively. These values are lower than 0.05. Hence, the two factors are statistically significant. The sliding rotational speed on the other hand has a *P*-value (0.448) greater than the set

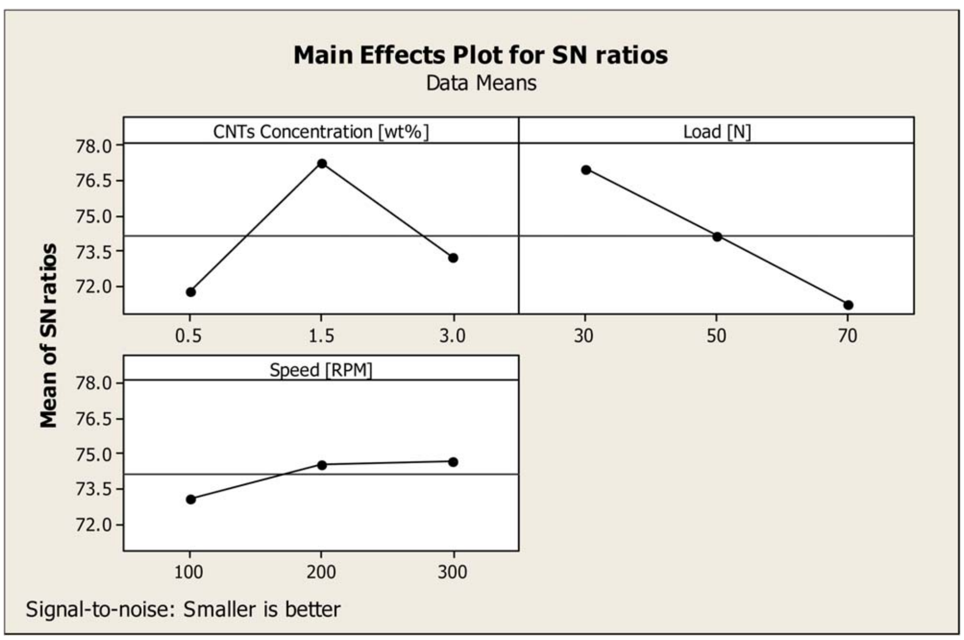

Figure 3. Main effect plot for WR signal to noise ratio. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

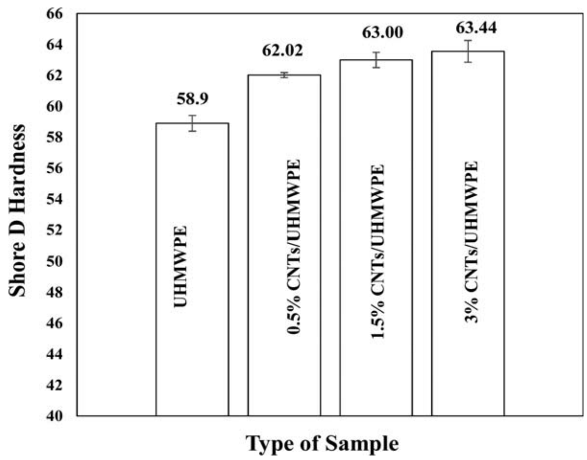

Figure 4. Shore D hardness for UHMWPE/CNTs composites.

0.05 significant level, which is an indication that this parameter is not contributing much to the variation in the measured wear rate.

P-value is used as a tool for checking the significance of the variables, and may indicate the patterns of the interactions among the variables. The smaller the *P*-value the stronger the effect of that variable on the process performance is. The *F*-value and sum of square can also be utilized to identify the importance of each interacting factor. Generally, a factor with the largest *F*-value is said to be the most influential parameter.[25] The ranking of process operating parameters were also displayed together with the ANOVA tables (Tables III and IV) for both mean signal to noise ratios and mean wear rate.

Other terms in ANOVA table includes; DF which stand for the number of degrees of freedom. DF is the number of values in the final calculation of a statistic that are free to vary. The analysis uses that information to estimate the values of unknown population parameters.[29,30] Seq SS is the sequential sums of squares. It is the reduction in the error sum of squares when one or more predictor variables are added to the regression model. Seq SS is used to test whether one slope parameter is 0, or to test whether a subset (more than two, but less than all) of the slope parameters are 0. Adj SS is the adjusted sums of squares. It is a measure of variation for different components of the model or term. Adj SS is used to calculate the *P*-value for a term. Adj MS stand for adjusted mean squares. It measure how much variation a term or a model explains.[29,30] Minitab uses the adjusted mean square to calculate the *P*-value for a term. Minitab also uses the adjusted mean squares to calculate the adjusted R^2 statistic.

Effect of the Input Parameters on the COF

Table V presents the L_9 (3^3) Taguchi design OA for the measured COF. The main effect plots for the mean friction coefficient and that of its signal to noise ratio are presented in Figures 5 and 6, respectively. It can be noticed that mean COF initially increases with an increase in CNTs concentration in UHMWPE polymer, and subsequently decreases with further increase in CNTs concentration. This abnormal behavior may be attributed to non-uniform distributions of CNTs in UHMWPE matrix at higher CNTs concentration. As discussed earlier, the composites showed an increase in COF due to the presence of the hard CNTs in the polymer matrix.[11] This can be attributed to the fact that increasing the CNTs loadings in the polymer improves its shear strength which consequently increases the COF.

It can also be seen that COF increases with increasing contact load, as a result of plastic deformation of asperities at high loads. This leads to an increase in the contact area, and eventually higher friction coefficient. At low contact load, there is little

Table III. Analysis of Variance for WR S/N Ratios

Analysis of variance for SN ratios						
Source	DF	Seq SS	Adj SS	Adj MS	*F*	*P*
CNTs concentration (wt %)	2	48.819	48.8195	24.4097	157.26	0.006
Load (N)	2	50.053	50.0528	25.0264	161.24	0.006
Speed (RPM)	2	4.919	4.9191	2.4595	15.85	0.059
Residual error	2	0.310	0.3104	0.1552		
Total	8	104.102				

Response table for signal to noise ratios Smaller is better			
Level	CNTs concentration (wt %)	Load (N)	Speed (RPM)
1	71.79	76.99	73.07
2	77.30	74.14	74.56
3	73.26	71.22	74.71
Delta	5.51	5.78	1.64
Rank	**2**	**1**	**3**

Table IV. Analysis of Variance for Means WR

Analysis of variance for means						
Source	DF	Seq SS	Adj SS	Adj MS	*F*	*P*
CNTs concentration (wt %)	2	0.000000	0.000000	0.000000	40.70	0.024
Load (N)	2	0.000000	0.000000	0.000000	48.99	0.020
Speed (RPM)	2	0.000000	0.000000	0.000000	1.23	0.448
Residual error	2	0.000000	0.000000	0.000000		
Total	8	0.000000				

Response table for means			
Level	CNTs concentration (wt %)	Load (N)	Speed (RPM)
1	0.000262	0.000149	0.000222
2	0.000146	0.000205	0.000208
3	0.000225	0.000279	0.000202
Delta	0.000116	0.000130	0.000020
Rank	**2**	**1**	**3**

or no true asperities contact. Thus, lower friction coefficient. The real/true contact area increases proportionally with load ($A_r \approx W/H$), and friction force increases proportionally to the contact load ($F_T = \mu W$). Where A_r is the true area of contact, W is the applied normal load, H is the hardness of the softer material, F_T is the frictional force, and μ is the friction coefficient.

Figure 5 also shows that COF decreases with increasing sliding velocity. This is because change in sliding velocity leads to change in the shear rate, which influences the mechanical properties of the CNTs/UHMWPE composite. At higher sliding velocity, the composite shear rate increases, which results in less real area of contact and consequently lowered the COF. High sliding velocities can also lead to high interface temperatures. This may give rise to localized surface melting, and significantly reduces shear strength of CNTs/UHMWPE composite, thereby lowering the COF value.

The ANOVA results for the friction coefficient and signal to noise ratio, along with the factors ranking are shown in Tables VI and VII, respectively. On the examination of the percentage of contribution (*P*-value) of the different parameters for COF and signal to noise ratio, it can be noticed that all the factors contribute to the variation in friction coefficient value significantly, since each parameter has a *P*-value lower than the set 0.05 confidence level. However, the most dominant parameters influencing the variation in COF and quality characteristic are the normal load and the CNTs concentration. The least factor affecting the COF is the rotational sliding velocity, with a *P*-value of 0.048.

Wear Mechanism

To provide further understanding of the earlier discussed mean effect plots presented as Figures 2–5, and 6, and the ANOVA, wear mechanism of the contacting surfaces need to be

Table V. COF Experimental Results, Average COF and S/N Ratio

				Responses (COF)				
Runs	CNTs concentration (wt %)	Load (N)	Speed (RPM)	Trial 1 COE	Trial 2 COE	Trial 3 COE	Mean (COF)	S/N ratio (dB)
1	0.5	30	100	0.103090	0.10301	0.10390	0.103333	19.7151
2	0.5	50	200	0.114503	0.11200	0.11250	0.113001	18.9380
3	0.5	70	300	0.112451	0.11100	0.11090	0.111450	19.0582
4	1.5	30	200	0.112950	0.11300	0.11205	0.112667	18.9640
5	1.5	50	300	0.115190	0.11700	0.11601	0.116067	18.7057
6	1.5	70	100	0.134806	0.13754	0.13866	0.137002	17.2649
7	3	30	300	0.101825	0.09680	0.09898	0.099202	20.0678
8	3	50	100	0.118900	0.11840	0.11870	0.118667	18.5134
9	3	70	200	0.123650	0.12425	0.12210	0.123333	18.1782

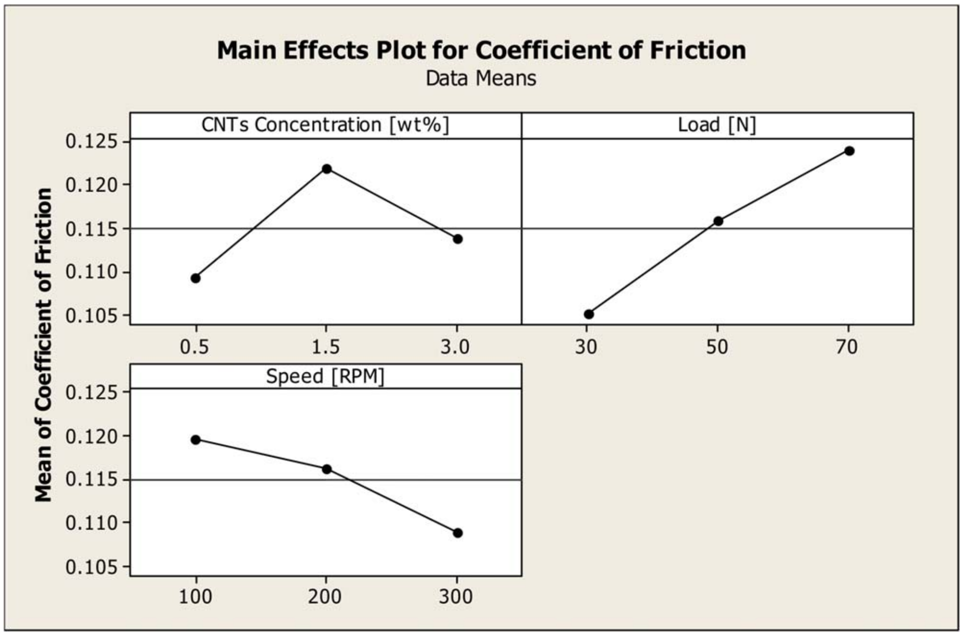

Figure 5. Main effect plot for mean COF. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

commented upon. To study the wear mechanisms of the contacting surfaces, the worn surfaces were examined and the SEM (images of the wear track surfaces are as shown in Figure 7. Abrasive wear seems to be the dominant wear mechanism with grooved and light furrows showing signs of plastic deformation for all nanocomposites. It can be seen form Figure 7 that the worn surface for 3 wt % CNTs/UHMWPE [(c), (f)] shows deep grooves and slightly more deformed surface as compared with 1.5 wt % CNTs/UHMWPE [(b), (e)]. This is reflected in slightly increased specific wear rate for 3 wt % as presented in Figure 2 of the main effect plot. The reason for this behavior can be attributed to the agglomeration of CNTs at higher loading. Figure 8 shows FE-SEM images for dispersion of CNTs in UHMWPE matrix. CNTs can be seen uniformly dispersed in 0.5 and 1.5 wt % composites. Individual CNTs are clearly visible dispersed in UHMWPE, which has resulted in improving the wear resistance and load bearing ability of the composite. While for 3 wt % CNTs/UHMWPE, agglomeration zones are seen. This is an indication for poor dispersion at higher CNTs content. Hence, CNTs were unable to support and transfer stress during plastic deformation resulting in higher specific wear rate for 3 wt % CNTs/UHMWPE.

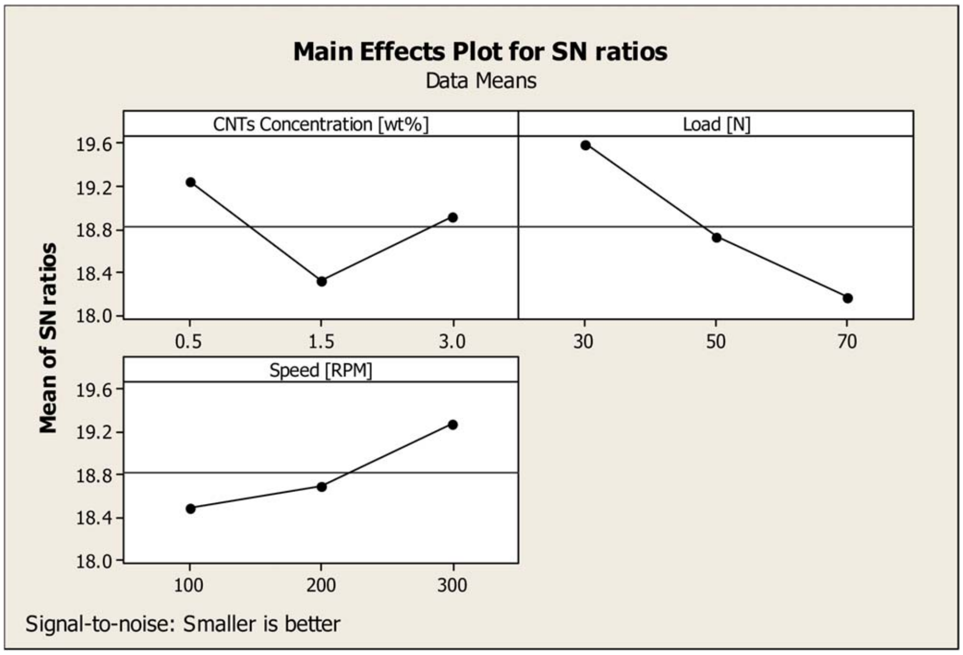

Figure 6. Main effect plot for COF signal to noise ratio. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table VI. Analysis of Variance for Means COF

Analysis of variance for means						
Source	DF	Seq SS	Adj SS	Adj MS	*F*	*P*
CNTs concentration (wt %)	2	0.000247	0.000247	0.000123	26.74	0.036
Load (N)	2	0.000538	0.000538	0.000269	58.22	0.017
Speed (RPM)	2	0.000182	0.000182	0.000091	19.72	0.048
Residual Error	2	0.000009	0.000009	0.000005		
Total	8	0.000976				

Response table for means			
Level	CNTs concentration (wt %)	Load (N)	Speed (RPM)
1	0.1093	0.1051	0.1197
2	0.1219	0.1159	0.1163
3	0.1137	0.1239	0.1089
Delta	0.0127	0.0189	0.0108
Rank	**2**	**1**	**3**

Figure 9 shows the optical images of the counter face ball. For 0.5 and 3 wt % CNTs considerable material transfer can be seen on counterface, whereas for 1.5 wt % CNTs there is no transfer film. This can be attributed to the fact that CNTs/UHMWPE composite with less amount of CNTs (0.5 wt % CNTs) will experience more polymer pullout resulting in a thicker transfer film on the counterface. However, in the case of 3 wt % CNTs, the thick transfer film formation may be because of the non-uniform dispersion of CNTs resulting in agglomerates which become ineffective in resisting the material pull out. Thus, worn surface morphologies and formed tribo-film behaviors of CNTs/UHMWPE composite account for different wear and friction characteristics of contacting surfaces.

Regression Modeling

In model development, wear rate and friction coefficient were modeled individually as dependent variables, while the normal contact load, sliding speed, and CNTs concentration in UHMWPE polymer matrix were modeled as independent variables.

Prior to model development, the actual response surface was plotted in order to have the general idea of the suitable variables function that will enable the smooth fitting of the model to

Table VII. Analysis of Variance for COF S/N Ratios

Analysis of variance for SN ratios						
Source	DF	Seq SS	Adj SS	Adj MS	*F*	*P*
CNTs concentration (wt %)	2	1.32736	1.32736	0.66368	65.60	0.015
Load (N)	2	3.05280	3.05280	1.52640	150.87	0.007
Speed (RPM)	2	0.98660	0.98660	0.49330	48.76	0.020
Residual error	2	0.02023	0.02023	0.01012		
Total	8	5.38700				

Response table for signal to noise ratios Smaller is better			
Level	CNTs concentration (wt %)	Load (N)	Speed (RPM)
1	19.24	19.58	18.50
2	18.31	18.72	18.69
3	18.92	18.17	19.28
Delta	0.93	1.42	0.78
Rank	**2**	**1**	**3**

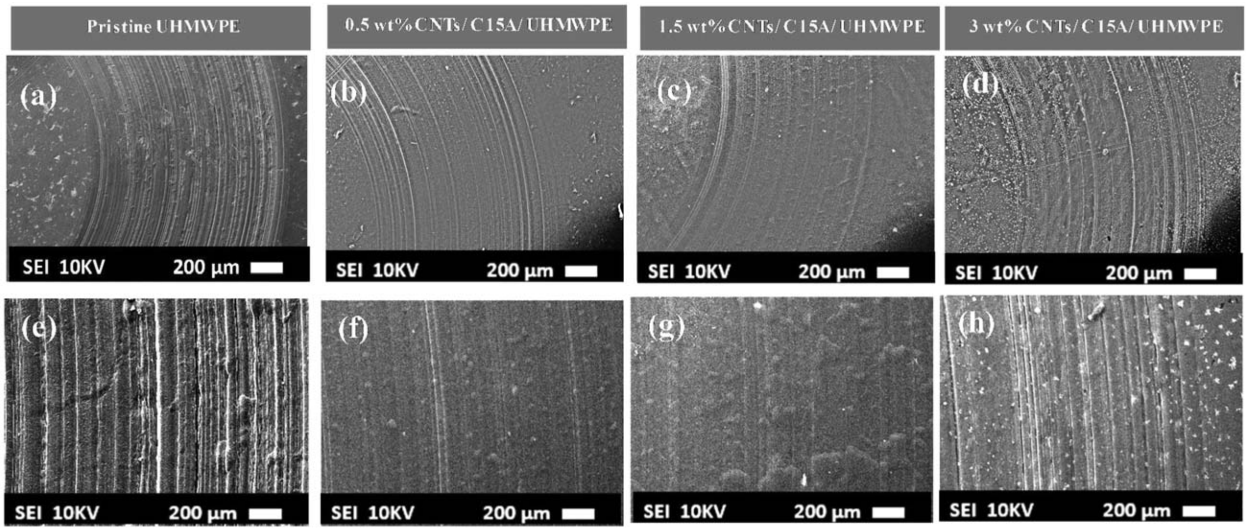

Figure 7. SEM wear track images showing wear morphology for CNTs/UHMWPE composites at low (a–c) and high magnification (d–f).

the actual response surface. Following which potential suitable models were generated, first, with normal contact load, sliding speed, and CNTs concentration as variables. Thereafter, all other possible suitable combinations were generated, including quadratic terms depending on the shape of the actual response plane. Comparisons were then made and the best model to represent the property change was selected.

Best subsets regression approach was adopted in the model development. For this approach, all possible regression equations were generated using all possible combinations of

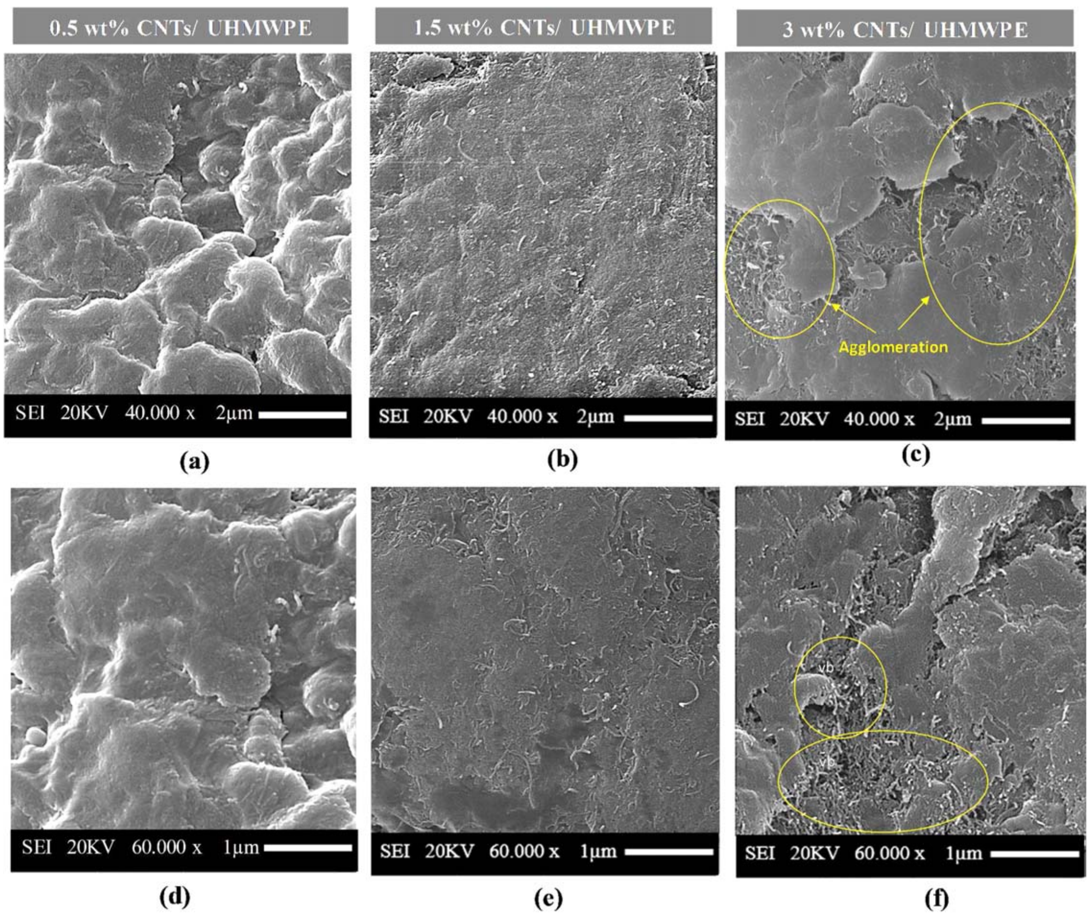

Figure 8. FE-SEM images of CNTs/UHMWPE composites showing dispersion of CNTs at low (a–c) and high magnification (d–f). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

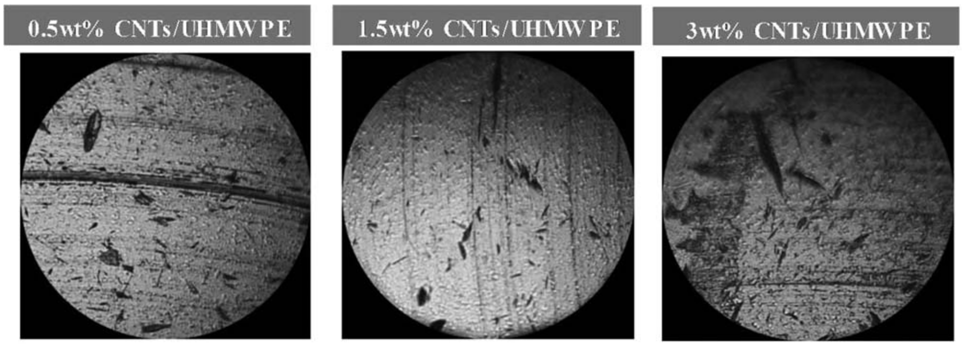

Figure 9. SEM optical images of the counter face ball.

independent parameters. Based on the highest *R*-square (*R*-Sq), adjusted *R*-square [*R*-Sq(adj)], and lowest standard error estimate (*S*), the best fit of the model was selected. The best regression model for predicting wear rate and COF were expressed in eqs. (4) and (5), respectively.

Regression equation for predicting wear rate is:

$$\text{Wear rate} = [24188.2+2039.76\text{C}-717.61\text{W} +2.7185\text{N}+32.0309\text{WC}-1130.77\text{C}^2+5.40781\text{W}^2]^{-1} \quad (4)$$

Summary of Model

$S=0.00287592 \quad R-\text{Sq}=99.56\% \quad R-\text{Sq(adj)}=98.25\%$

The regression equation for predicting COF is given as:

$$\text{COF} = [12.9526-1.94975\text{C}-0.0866633\text{W}-0.00559609\text{N} +0.523398\text{C}^2+ 0.000512153\text{W}^2+0.0000234289\text{N}^2]^{-1} \quad (5)$$

Summary of Model

$S=R-\text{Sq}=99.90\% \quad R-\text{Sq(adj)}=99.60$

where COF is the friction coefficient, C is the percentage of CNTs concentration in UHMWPE [%wt], W is the contact load [N], and N is the rotational sliding speed [RPM]. Note that Wear rate is in [mm^3/Nm].

The developed simple regression model for predicting wear rate has a *R*-Square of 99.56%. This is an indication that 99.56% of variation in wear rate is due to variation in normal contact load, sliding speed, and CNTs concentration. The model also has adjusted *R*-Sq of 98.25%. This means that 98.25% of variation in wear rate is explained by variation in normal contact load, sliding speed, and CNTs concentration, taking into account the experimental data size and number of independent variables. The standard error estimate (*S*) of the model, which is the measure of deviation of observed wear rate from the fitted line is 0.00287592. Similar terms with different coefficient appear in the regression model for predicting friction coefficient.

The appeared coefficient terms in the model eq. (4) indicates that CNTs concentration has positive linear effect on the wear rate. This indicates that increasing the CNTs concentration in UHMWPE will lead to greater material removal rate. However, it's negative quadratic term overrun this positive linear term. Thus, increasing this term will increase the wear resistance of UHMWPE composite. For the COF model given as eq. (5), the CNTs concentration has positive quadratic term, which dominate it's negative linear term. This implies that increasing the percentage of CNTs concentration in UHMWPE polymer will result in increasing COF.

The coefficient terms for the applied normal load in both eqs. (4) and (5) showed an overall (comparing quadratic term with the linear term) positive effect on the wear rate and COF, which is an indication that increasing the term will lead to a higher COF and wear rate. From model eq. (5), the sliding velocity has negative linear term whose effect on friction coefficient slightly overrun it's positive quadratic term. This showed that increasing the sliding rotational speed would slightly decrease the wear rate and COF of the CNTs/UHMWPE composite. Therefore, we concluded from the above model equations that the wear rate increases with increasing applied contact load, and decreases

Table VIII. Experimental and Predicted Specific Wear Rate and COF at 1 wt % CNTs Concentration

CNTs concentration (wt %)	Load (N)	Speed (RPM)	COF			Wear rate		
			Experiment	Predicted	% Error	Experiment	Predicted	% Error
1	60	250	0.129	0.121425764	5.8715010	0.000237356	0.000243298	−2.503626
1	40	150	0.147	0.116728372	20.592944	0.000153067	0.000148494	2.9876311
1	35	280	0.131	0.106492411	18.708083	0.00010799	0.000117818	−9.100344
1	45	180	0.129	0.118831043	7.8829127	0.000224119	0.000175862	21.531682
1	65	230	0.119	0.124855355	−4.920465	0.000243046	0.000249513	−2.660913

Table IX. Experimental and Predicted Specific Wear Rate and COF at 1.5 wt % CNTs Concentration

CNTs concentration (wt %)	Load (N)	Speed (RPM)	COF			Wear rate		
			Experiment	Predicted	% Error	Experiment	Predicted	% Error
1.5	60	250	0.14	0.12634467	9.7538073	0.000226	0.00021488	5.538189
1.5	40	150	0.15	0.121266948	19.155368	0.000156	0.000143239	8.351396
1.5	35	280	0.123	0.110257072	10.360103	0.000101	0.000115545	−14.7908
1.5	45	180	0.129	0.123537898	4.2341873	0.000228	0.000166296	27.19816
1.5	65	230	0.145	0.130062005	10.302065	0.000239	0.000214813	10.26854

Table X. Experimental and Predicted Specific Wear Rate and COF at 2.25 wt % CNTs Concentration

CNTs concentration (wt %)	Load (N)	Speed (RPM)	COF			Wear rate		
			Experiment	Predicted	% Error	Experiment	Predicted	% Error
2.25	60	250	0.128	0.126189312	1.4146001	0.000236206	0.000223791	5.255709
2.25	40	150	0.153	0.121123819	20.834104	0.000149858	0.000158937	−6.05837
2.25	35	280	0.137	0.110138741	19.606758	0.000106962	0.00012747	−19.1735
2.25	45	180	0.13	0.123389362	5.0851059	0.000236911	0.00018369	22.46467
2.25	65	230	0.124	0.129897376	−4.755948	0.000241959	0.000218998	9.489743

with increasing CNTs concentration. However, the sliding velocity has slightly decreasing/negligible effect on the wear rate. The COF values increases with increasing CNTs concentration and the applied normal load, and it slightly decreases with increasing sliding velocity.

Model Confirmation

In order to validate conclusions drawn on Taguchi's parameter design approach, additional comprehensive experimental run was conducted using a specific combination of the parameters and levels. Tables 3(VIII–X) shows the parameters combination and levels used for both friction coefficient and wear rate. The corresponding experimental results and the predicted values are equally presented in Tables (VIII–X). The percentage variation in actual and predicted data was as well presented in the tables. It can be seen that the actual experimental data are in close agreement with those obtained by the regression models eqs. (4) and (5). This confirms that the DOEs could be used effectively to model and optimize the process variables of UHMWPE/CNTs composite using the statistical DOEs concept. The model was found to be adequate in predicting both wear rate and friction coefficient. It is worth mentioning that the capability of the developed models is within the domain and environmental conditions in which the tribological test was conducted. Thus, the model predictions outside the range and the domain of the test conditions may be inaccurate.

CONCLUSIONS

The following important conclusions can be drawn from the above discussion:

- Taguchi optimization techniques has proven to be a very good approach that can be provides better understanding of the influence of different test parameters on the tribological properties of CNTs/UHMWPE composites.
- Applied contact load (P-value = 0.020) is the most dominant factor influencing the wear rate of the composite. This is closely followed by CNTs concentration (P-value = 0.024). The sliding rotational speed (P-value of 0.448) showed negligible impact on wear rate. For the COF, the P-value of contact normal load, CNTs concentration, and rotational speed are 0.017, 0.036, and 0.048 respectively.
- At higher normal contact load, both friction coefficient and rate of material removal increase.
- Increasing the rotational sliding velocity lower the friction coefficient, and has negligible impact on wear rate.
- The addition of CNTs up to 1.5 wt % to UHMWPE polymer matrix increases both wear resistance and COF of the CNTs/UHMWPE composite. Beyond 1.5 wt %CNTs, both friction coefficient and wear rate decreases, perhaps due to non-homogeneous dispersion of CNTs in the matrix of UHMWPE polymer. This indicates that the dispersion and of the CNTs and the homogenization of the mixture needs further improvement.

Thus, CNTs is a promising additive for polymer-based composites that demands very good wear resistance.

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REFERENCES

1. Zhang, S. W. *Tribol. Int.* **1998**, *31*, 49.
2. Friedrich, K.; Lu, Z.; Hager, A. *Wear* **1995**, *190*, 139.
3. Brostow, W.; Kovačevic, V.; Vrsaljko, D.; Whitworth, J. *J. Mater. Ed.* **2010**, *32*, 273.
4. Harvey, L. S; Ticona, L. L. C. Engineered Materials Handbook; ASM International: Materials Park, **1999**.
5. Samad, M. A.; Satyanarayana, N.; Sinha, S. K. *J. Adhes. Sci. Technol.* **2010**, *24*, 2557.
6. Samad, M. A.; Sinha, S. K. *Tribol. Lett.* **2010**, *38*, 301.
7. Samad, M. A.; Sinha, S. K. *Wear* **2011**, *271*, 2759.
8. Samad, M. A.; Sinha, S. K. *Wear* **2011**, *270*, 395.
9. Samad, M. A.; Sinha, S. K. *Tribol. Int.* **2011**, *44*, 1932.
10. Aldousiri, B.; Shalwan, A.; Chin, C. *Adv. Mater. Sci. Eng.* **2013**, *2013*, 1.
11. Zoo, Y. S.; An, J. W.; Lim, D. P.; Lim, D. S. *Tribol. Lett.* **2004**, *16*, 305.
12. Plumlee, K.; Schwartz, C. J. *Wear* **2009**, *267*, 710.
13. Kavita, M. B.; Tiwari, R. K. *J. Appl. Polym. Sci.* **2016**, *133*, DOI: 10.1002/app.43333.
14. Junghee, P.; Nasrin, R.; Eun, S. O.; Keungarp, R. *J. Appl. Polym. Sci.* **2016**, *133*, DOI: 10.1002/app.43307.
15. Ozcan, K.; Mustafa, K.; Demet, O. *J. Appl. Polym. Sci.* **2013**, *127*, 4557.
16. Dangsheng, X. *Mater. Lett.* **2005**, *59*, 175.
17. Johnson, B. B.; Santare, M. H.; Novotny, J. E.; Advani, S. G. *Mech. Mater.* **2009**, *41*, 1108.
18. Galetz, M. C.; Blaß, T.; Ruckdäschel, H.; Sandler, J. K. W.; Altstädt, V.; Glatzel, U. *J. Appl. Polym. Sci.* **2007**, *104*, 4173.
19. Zuo, G.; Guan, G.; Wang, R. *Desalination* **2014**, *339*, 1.
20. Khalifa, A. E.; Lawal, D. U. *Arab. J. Sci. Eng.* **2015**, *40*, 3627.
21. Pang, J. S.; Ansari, M. N. M.; Zaroog, O. S.; Ali, M. H.; Sapuan, S. M. *HBRC J.* **2014**, *10*, 138.
22. Negrete, C. C. *J. Clean. Prod.* **2013**, *53*, 195.
23. Asilturk, I.; Neseli, S. *Measurement* **2012**, *45*, 785.
24. Uysal, A.; Altan, M.; Altan, E. *Int. J. Adv. Manuf. Technol.* **2012**, *58*, 915.
25. Mavruz, S.; Ogulata, R. T. *Fibres Text. East. Eur.* **2010**, *18*, 78.
26. Asilturk, I.; Akkus, H. *Measurement* **2011**, *44*, 1697.
27. Shetty, R.; Pai, R. B.; Rao, S. S.; Nayak, R. *J. Braz. Soc. Mech. Sci. Eng.* **2009**, *31*, 12.
28. Salvetat, J. P.; Bonard, J. M.; Thomson, N. H.; Kulik, A. J.; Forro, L.; Benoit, W.; Zuppiroli, L. *Appl. Phys. A* **1999**, *69*, 255.
29. Guerrero, A. L.; Nachmias, C. F. Essentials of Social Statistics for a Diverse Society, 2nd ed.; SAGE Pub.: Chicago, **2015**; Chapter 8, p 179.
30. Montgomery, D. C. Design and Analysis of Experiments, 7th ed.; Wiley: New York, **2009**; Chapter 3, p 60.