

Tribological Behaviour Improvement of Lubricant Using Copper (II) Oxide Nanoparticles as Additive

M. A. Hassan, M. H. Sakinah, K. Kadirgama, D. Ramasamy, M. M. Noor, M. M. Rahman

Abstract—Tribological properties that include nanoparticles are an alternative to improve the tribological behaviour of lubricating oil, which has been investigated by many researchers for the past few decades. Various nanostructures can be used as additives for tribological improvement. However, this also depends on the characteristics of the nanoparticles. In this study, tribological investigation was performed to examine the effect of CuO nanoparticles on the tribological behaviour of *Syntium* 800 SL 10W–30. Three parameters used in the analysis using the wear tester (piston ring) were load, revolutions per minute (rpm), and concentration. The specifications of the nanoparticles, such as size, concentration, hardness, and shape, can affect the tribological behaviour of the lubricant. The friction and wear experiment was conducted using a tribo-tester and the Response Surface Methodology method was used to analyse any improvement of the performance. Therefore, two concentrations of 40 nm nanoparticles were used to conduct the experiments, namely, 0.005 wt % and 0.01 wt % and compared with base oil 0 wt % (control). A water bath sonicator was used to disperse the nanoparticles in base oil, while a tribo-tester was used to measure the coefficient of friction and wear rate. In addition, the thermal properties of the nanolubricant were also measured. The results have shown that the thermal conductivity of the nanolubricant was increased when compared with the base oil. Therefore, the results indicated that CuO nanoparticles had improved the tribological behaviour as well as the thermal properties of the nanolubricant oil.

Keywords—Concentration, improvement, tribological, Copper (II) oxide, nanolubricant.

I. INTRODUCTION

SEVERAL research papers have reported that the addition of nanoparticles to lubricant is effective for improving tribological behaviour in the mechanical system [1]-[4]. Our technology-driven civilization expands year by year and the conservation of materials and energy is important. Nanoparticles are being hailed as one of revolutionary technology of the 21st century. According to [4]-[9], several characteristics must be considered when applying nanoparticles as an additive in lubricant, which include size, shape, concentration and hardness. The friction-reduction and anti-wear behaviour depend on these characteristics. However, each characteristic has different influences on tribological behaviour. The average size of nanoparticles used is between 2 and 120 nm [8], [9]. The tribological behaviour of a piston

ring contact has been recognized as an important influence on the performance of internal combustion engine in several aspects, such as harmful exhaust emissions, blow-by, power lost, fuel consumption, and oil consumption [10]. A lubricant with solid nanoparticles can reduce the friction coefficient while simultaneously increase the load carrying capacity of the lubricant fluid [11]. Nowadays, normal lubricants, such as petroleum, coals and natural gases are in high demand. However, these natural resources have their limitations and will be depleted due to high fuel consumptions around the world [12]. According to Idris, Vezir [13], during the movement of the piston ring within the dead-centre of the cylinder, the maximum friction coefficient occurred, where the lubricant is minimal and the piston ring was at a higher speed, while the minimum friction coefficient occurred during the mid-stroke. Ettefaghi, Ahmadi [14] have stated that the natural wear and friction coefficient had improved at the same time when the viscosity of the base oil had increased with increasing concentration of Cu nanoparticles.

In recent years, many studies have studied the application of nanoparticles in the field of lubrication. It has been reported that the concentration of nanoparticles in the base-oils is an important parameter while formulating a nanolubricant. According to Koshy, Rejendrakumar [6], one significant observation is that improvements in the desired property of the base-oils occurred with low concentrations of nanoparticles.

A. Design of Experiment (DOE)

Successful experimentation requires knowledge of the important factors that can influence the output. The design of experiment and the statistical analysis of data are two basic aspects in scientific experimentation. Response surface methodology or RSM is a mathematical and statistical technique that is useful for analysing and modelling a problem that may be influenced by several variables. The objective of using these techniques is to optimize responses. The RSM uses the Box-Behnken design, developed to require only three levels, coded as -1, 0 and +1. This design may be used to optimize one or more responses. According to Alpaslan Atmanlı, Bedri Yüksel [15], the general second-order polynomial response surface mathematical model (full quadratic model) for the experimental design is as shown in (1):

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i < j}^n \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

where Y is the response and X_i is the values of the factors.

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The terms β_0 , β_i , β_{ii} and β_{ij} are the regression coefficients for determining the regression equation and they also represent the residual matrix. The experimental results based on the RSM method have been established with 95 % confidence. The optimum values of the selected variables were obtained by solving the regression equation using the Analysis of Variance (ANOVA). ANOVA represents the relationship between the parameters with the overall process performance. The ANOVA is a simple idea of introducing the no-way (or no-factor) analysis of variance that builds up to one-way (or one-factor) analysis of variance and eventually to a multi-way (or multi-factor) analysis of variance. The Minitab 15 software was used to generate the response surface plots and contour plots as well as optimize the factors used to reduce the tribological behaviour [16].

TABLE I
 FACTOR AND LEVEL FOR EXPERIMENT

Factor	Parameters (units)	Level		
		-1	0	1
A	Load (N)	20	55	90
B	Speed (rpm)	200	250	300
C	Concentration (wt%)	0	0.005	0.01

B. Wear Rate

Wear test involves making a linear movement similar to a cylinder-piston ring pair operating under real condition [13]. Wear is an interaction between two surface, specifically the removal and deformation of the material of the specimen on a surface. This study involved the adhesive wear found between surfaces during frictional contact which normally referring to unwanted displacement and attachment of wear debris. According to Agarwal, Patnaik [17], the wear rate can be calculated using (2):

$$\Delta W = (W_2 - W_1) \quad (2)$$

where Δw = weight loss of specimen, W_1 = weight of the specimen before the test and w_2 = weight of the specimen after the test.

ΔV = volume loss of the specimen is computed as:

$$\Delta V = \frac{W_2 - W_1}{\rho} \times 1000 \quad (3)$$

where ρ = density of the specimen. Therefore, wear rate can be calculated as:

$$w_s = \frac{\Delta V}{F_n - S_s} \quad (4)$$

where F_n = normal load (N) and S_s = sliding distance (m).

C. Coefficient of Friction (COF)

Friction occurs when one solid surface slides over the other surface. Various forces are present during friction, such as static frictional force, a force that is parallel to resistance force [18]. The equation for the coefficient of friction is as:

$$COF = \frac{\text{Friction force of opposing motion (F)}}{\text{Right angle load to the surface (N)}} \quad (5)$$

or $F = \mu.N$, where μ is the coefficient of friction.

The coefficient of friction is one of the important aspects that can influence the tribological behaviour of the lubricant. The friction coefficient will represent the energy loss caused by friction [11]. Therefore, the addition of nanoparticles, especially copper (II) oxide, can reduce the energy in the mechanical loss. The lower coefficient of friction will give the maximum friction reduction and better wear using the lubricant added with the additive. According to [7], higher friction coefficient will exhibit lower friction reduction in a contact. It will also give lower wear when using copper nanoparticles at higher concentration, therefore it improves the tribological behaviour of the suspension.

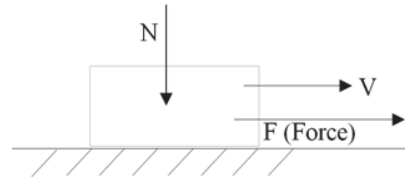


Fig. 1 Relationship between friction force, load and coefficient of friction [18]

II. EXPERIMENTAL PROCEDURE

A. Nanoparticles and Lubricant

Spherical nanoparticles of 40 nm in size were dispersed in the *Syntium* 800SL 10W-30 with concentrations of 0 wt% (control), 0.005 wt% and 0.01 wt% in separate containers using a water bath sonicator for 1 hour. The nanoparticles as an additive could improve the anti-wear, load-carrying and friction-reduction performance of base oil. Additionally, nanolubricant could be used to avoid sedimentation and agglomeration. The properties test for the nanolubricant was conducted before the experiments were conducted in order to determine the properties of the lubricant, with and without the additive.

B. Materials and Method

The material used as a specimen was aluminium 6061 with a dimension of 5 cm (L) x 0.6 cm (H) x 2.5 cm (W) and a grey cast iron as a tool. All components were cleaned using ethanol before and after use and then dried. The RSM was designed using the Minitab15 software. Experiments were conducted using a tribo-tester according to the table of experiment (see Table II) with the response results for COF and wear.

The time for each load step was 5-10 minutes. There were 15 experimental runs, with loads that range from 20N to 90N for a total time of 75-150 min. The sliding distance was 0.19 mm. The friction recorded by the Arduino software will be used to find the coefficient of friction as shown in (5). The wear surface on the specimen was characterized using SEM while XRD was used to detect the elements that were present on the specimen.

III. RESULTS AND DISCUSSION

A. Empirical Model and Regression Analysis on Tribological Properties

The design matrix and the corresponding responses for the experimental studies were carried out using ANOVA. Fig. 2 shows the residual plots of the COF of the CuO nanoparticles in base oil as a function of the independent variable and based on the line in the normal probability plot and the even distribution of data in the versus fits, it can be assumed that the residuals of the model for the COF were normally distributed.

Tables III and IV present the estimated regression coefficients and analysis of variance (ANOVA) for the coefficient of friction and wear rate of the nanolubricant. From these results, the probability values (p-value) for the COF and for wear rate were 0.002 and 0.031, respectively. Since both probability values were lower than 0.05, this means the terms in the model had a significant influence on the COF and wear rate. Meanwhile, the lack-of-fit p-value for the COF and wear rate was non-significant as they were higher than 0.05. The "R-squared value" was satisfactory as it was higher than 95 % for the COF model. However, "R-squared value" for wear rate was less than 95%. It can be summarized that the effect of the individual parameters and their interactions fit as a second-

order quadratic model. Therefore, the generated models were significant. Equations (6) and (7) are the empirical equations for the COF and the wear rate average for the lubricant in the nanolubricant model as a function of the independent variables of load (L), speed (S) and concentration (C) in coded units.

TABLE II
 EXPERIMENT RUNS

No	Load	Speed	Concentration	Coefficient of Friction (COF)	Wear Rate (x E03) mm ³ /Nm
1	2.0	250	0.000	23.40126004	0.314657568
2	9.0	250	0.000	7.085381512	0.076025215
3	5.5	250	0.005	0.354564546	0.095592
4	5.5	200	0.010	5.708489192	0.12534764
5	2.0	200	0.005	13.74824027	0.454798581
6	2.0	250	0.010	30.81165905	0.317011418
7	5.5	250	0.005	5.921227919	0.125121275
8	9.0	300	0.005	6.565353511	0.07422928
9	9.0	200	0.005	0.606699334	0.06125
10	5.5	300	0.000	4.183861644	0.126530674
11	9.0	250	0.010	7.58374168	0.076139837
12	5.5	250	0.005	0.248195182	0.09865
13	5.5	300	0.010	0.673672638	0.093777
14	2.0	300	0.005	33.34679556	0.365237165
15	5.5	200	0.000	3.261993824	0.124685879

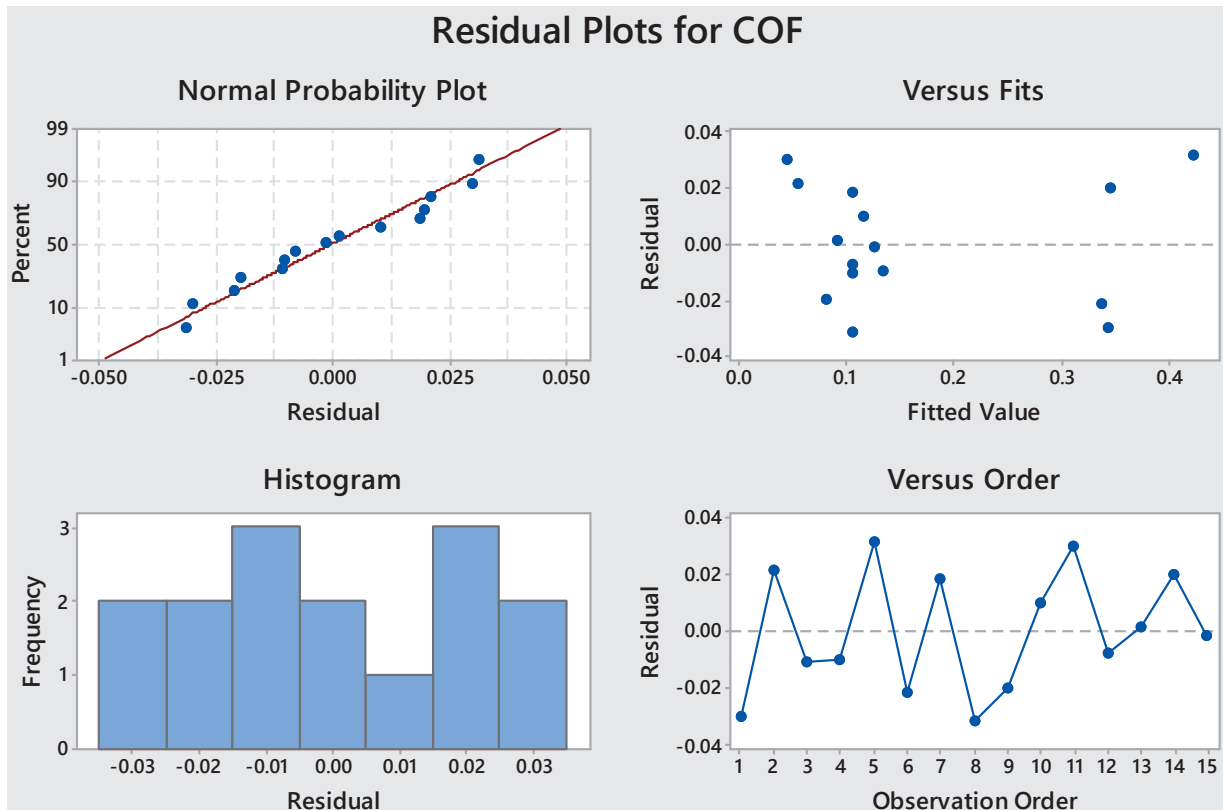


Fig. 2 Residual plot of data obtained for COF.

$$\text{COF}; Y_1 = 0.1065 - 0.1455 L - 0.0133 S - 0.0037 C + 0.1054 L^2 + 0.0270 S^2 - 0.0159 C^2 + 0.0.256 LS - 0.0006 LC - 0.00084 SC \quad (6)$$

$$\text{Wear Rate}; Y_2 = 2.17 - 9.93 L + 2.68 S + 0.86 C + 12.58 L^2 - 1.19 S^2 + 2.47 C^2 - 3.41 LS - 1.73 LC - 1.49 SC \quad (7)$$

TABLE III
 ESTIMATED REGRESSION COEFFICIENT AND ANALYSIS OF VARIANCE FOR
 COF OF SPECIMEN

Term	Effect	Coef.	SE Coef.	t-value	p-value	VIF
Constant		0.1065	0.0203	5.23	0.003	
Load	-0.2910	-0.1455	0.0125	-11.68	0.000	1.00
Speed	-0.0266	-0.0133	0.0125	-1.07	0.335	1.00
Concentration	-0.0074	-0.0037	0.0125	-0.30	0.778	1.00
Load*Load	0.2108	0.1054	0.0183	5.75	0.002	1.01
Speed*Speed	0.0541	0.0270	0.0183	1.47	0.201	1.01
Concentration*Concentration	-0.0318	-0.0159	0.0183	-0.87	0.426	1.01
Load*Speed	0.0513	0.0256	0.0176	1.46	0.205	1.00
Load*Concentration	-0.0011	-0.0006	0.0176	-0.03	0.976	1.00
Speed*Concentration	-0.0167	-0.0084	0.0176	-0.47	0.655	1.00
Source	df	Adj. SS	Adj. MS	F-Value	p-value	
Model	9	0.218558	0.024284	19.56	0.002	
Linear	3	0.170902	0.056967	45.89	0.000	
Load	1	0.169380	0.169380	136.44	0.000	
Speed	1	0.001413	0.001413	1.14	0.335	
Concentration	1	0.000110	0.000110	0.09	0.778	
Square	3	0.044747	0.014916	12.02	0.010	
Load*Load	1	0.041017	0.041017	33.04	0.002	
Speed*Speed	1	0.002697	0.002697	2.17	0.201	
Concentration*Concentration	1	0.000933	0.000933	0.75	0.426	
Interaction	3	0.002909	0.000970	0.78	0.553	
Load*Speed	1	0.002629	0.002629	2.12	0.205	
Load*Concentration	1	0.000001	0.000001	0.00	0.976	
Speed*Concentration	1	0.000279	0.000279	0.22	0.655	
Residual Error	5	0.006207	0.001241			
Lack-of-Fit	3	0.005680	0.001893	7.18	0.125	
Pure Error	2	0.000527	0.000264			
Total	14	0.224764				

S= 0.0352333, R²= 97.24%; R²(adj)= 92.27%; R²(pred)= 59.04%; df= degree of freedom; Adj SS: adjusted sum of squares; Adj MS: adjusted mean squares.

The t-value and P-value in the estimated regression coefficient of the COF in Table III and the wear rate in Table IV denote the significant influence of each input variable in the models. The smaller numerical values of "p" and the larger values of "t" signify that the related regression coefficients are highly significant [16]. The p-value in the ANOVA analysis helps to determine which effect, whether factor or interaction, is statistically significant. When the p-value is smaller, the probability to make a mistake is also smaller. Based on the COF models, the highest significant level was shown by the linear load, followed by the quadratic load and lastly, the quadratic speed. Meanwhile, the quadratic concentration, the interaction between load and concentration as well as the interaction between speed and concentration were less significant. Highly significant impacts to the wear rate model were displayed by the linear load, which was followed by the linear speed and the quadratic load for the estimated regression coefficient of the wear rate. Meanwhile, less significant impacts were shown by the linear concentration, quadratic speed and the load-concentration interaction.

B. Response of Wear Rate and Average Control Parameters

The three-dimensional surface plots and contour plots of the collective impacts of the control parameters (load, speed and concentration) on the COF and wear rate of the nanolubricant are shown in Figs. 3 and 4. The load was more significant than the speed, as shown in Fig. 3 (a). This figure shows the plots as a function of load and speed on COF at a mean concentration of 0.005 wt%. It is clear that the COF was reduced based on the load (20–90 N) and speed (200–300 rpm) that were chosen. A similar trend is shown in Fig. 3 (c); the load-concentration plots for both parameters have shown their effects on the COF of the tribology in the nanolubricant. The speed and concentration plots in Fig. 3 (b) show that as the speed increases, the COF dropped slightly. Meanwhile, the COF was reduced to the lowest value at a certain concentration. Then, it rose when the concentration was increased even at lower speed. The lowest COF of the nanolubricant was noted in the contour plot at a speed of 300 rpm and a concentration between 0.004-0.006 wt%. The COF was reduced at a certain speed and rose again when the speed was increased at higher load-concentration condition.

TABLE IV
 ESTIMATED REGRESSION COEFFICIENTS AND ANALYSIS OF VARIANCE FOR
 WEAR RATE OF NANOLUBRICANT

Term	Effect	Coef.	SE Coef.	t-value	p-value	VIF
Constant		2.17	3.07	0.71	0.510	
Load	-19.87	-9.93	1.88	-5.29	0.003	1.00
Speed	5.36	2.68	1.88	1.43	0.213	1.00
Concentration	1.71	0.86	1.88	0.46	0.668	1.00
Load*Load	25.16	12.58	2.77	4.55	0.006	1.01
Speed*Speed	-2.37	-1.19	2.77	-0.43	0.686	1.01
Concentration*Concentration	4.94	2.47	2.77	0.89	0.413	1.01
Load*Speed	-6.82	-3.41	2.66	-1.28	0.256	1.00
Load*Concentration	-3.46	-1.73	2.66	-0.65	0.544	1.00
Speed*Concentration	-2.98	-1.49	2.66	-0.56	0.599	1.00
Source	DF	Adj SS	Adj MS	F-Value	p-value	
Regression	9	1531.60	170.178	6.03	0.031	
Linear	3	852.71	284.237	10.07	0.015	
Load	1	789.37	789.371	27.96	0.003	
Speed	1	57.48	57.482	2.04	0.213	
Concentration	1	5.86	5.857	0.21	0.668	
Square	3	611.57	203.855	7.22	0.029	
Load*Load	1	584.13	584.128	20.69	0.006	
Speed*Speed	1	5.19	5.191	0.18	0.686	
Concentration*Concentration	1	22.49	22.491	0.80	0.413	
Interaction	3	67.33	22.442	0.79	0.547	
Load*Speed	1	46.51	46.512	1.65	0.256	
Load*Concentration	1	11.94	11.944	0.42	0.544	
Speed*Concentration	1	8.87	8.871	0.31	0.599	
Residual Error	5	141.18	28.236			
Lack-of-Fit	3	120.12	40.040	3.80	0.215	
Pure Error	2	21.06	10.530			
Total	14	1672.78				

S= 5.31379, R²= 91.56%, R²(adj)= 76.37 %R²(pred)= 0.00%, df= degree of freedom, Adj SS: adjusted sum of squares, Adj MS: adjusted mean squares

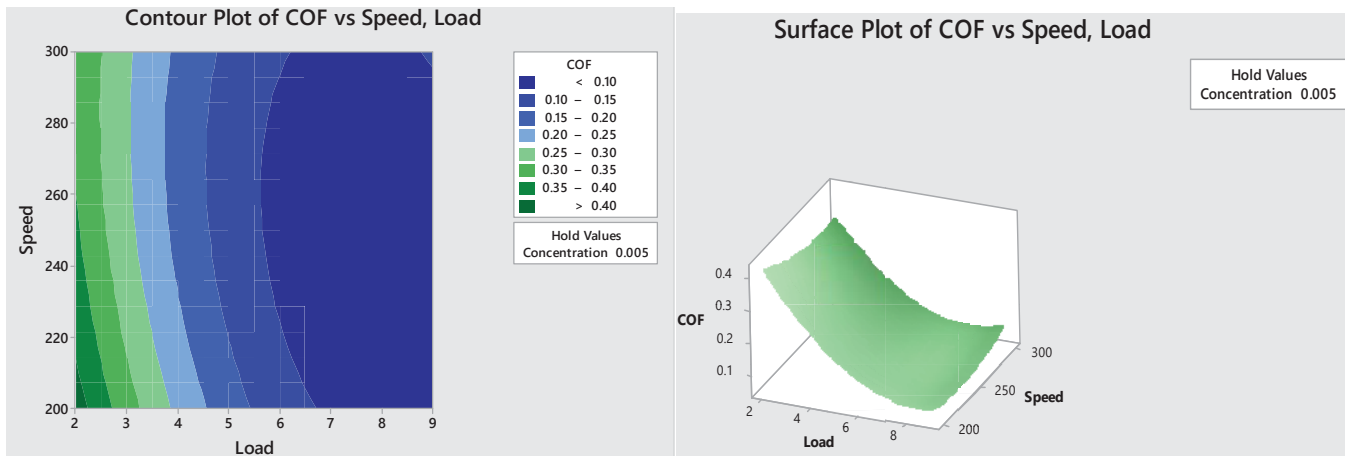
During the sliding, the hard asperities on the counterface had extricated the contacting material on the specimen's surface as wear debris. It happened due to the deposit of small fragments of wear debris between the counterface of the abrasive asperities that had broken off from the softer material. According to [7], the nanoparticles can penetrate into the contact area of the specimen and deposit on it because of the smaller or similar size of the lubricant's film thickness. However, nanoparticles may also have a deleterious effect that can increase friction or wear in some cases. The COF and wear rate of the lubricant containing additive were decreased when the loads were increased. The protection layer was also formed when the load was increased.

The response surface plots and contour plots presented in Fig. 4 show the effects of different control parameter combinations on the wear rate of the nanolubricant. The load interaction in Fig. 4 (a) has revealed that the wear rate was

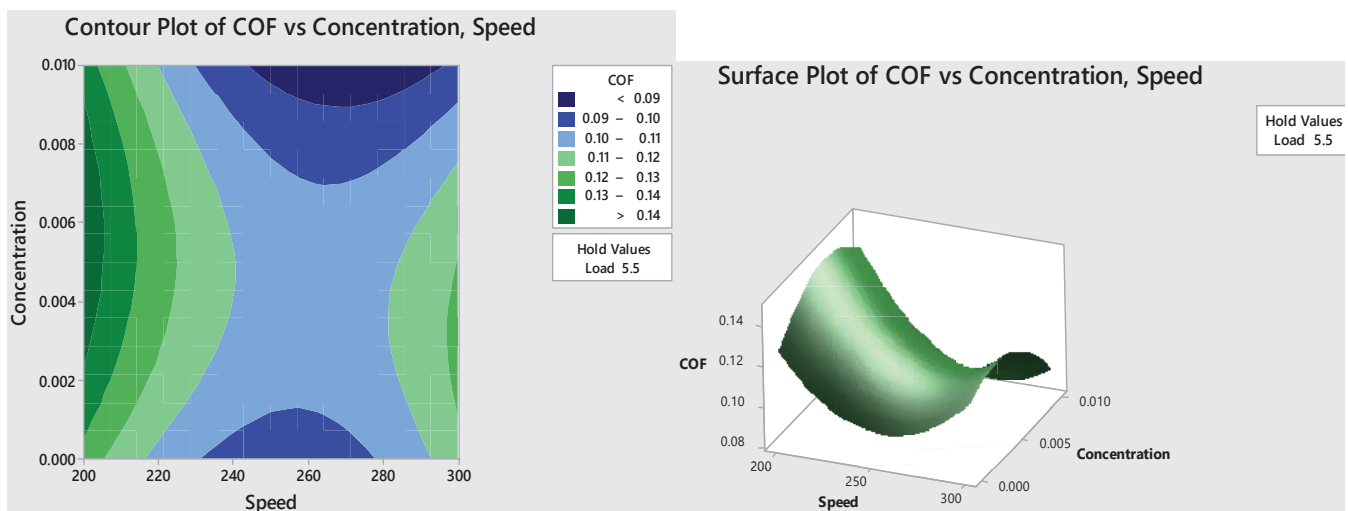
decreased when the load was increased between 10 to 20N. Meanwhile, the increase in wear rate was observed following the addition of applied load. This could be due to the increased friction between the contact surface of the specimen and the tool. The minimum wear rate had occurred at a load of 20 N and speed of 300 rpm. The load-concentration plots in Fig. 4 (b) have shown that the wear rate had slightly decreased when the load was increased, while the wear rate had reduced to the lowest value at a certain speed, and then, went up when the speed was increased, even under low load conditions.

In Fig. 4 (c), the wear rate increased when the speed was increased, while the concentration of nanoparticles had less significant impact on the wear rate. This minor impact on the tribological behaviour of the specimen may be due to some trapped debris. The protection layer was not formed during the friction, which may have mitigated the roughness of the contacting surface.

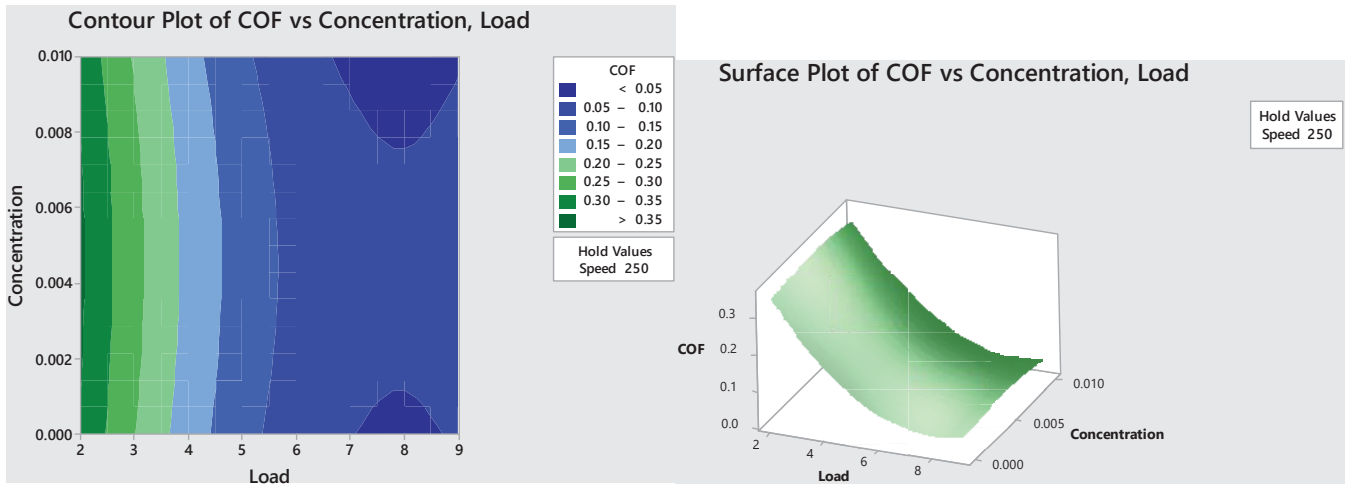
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(a)

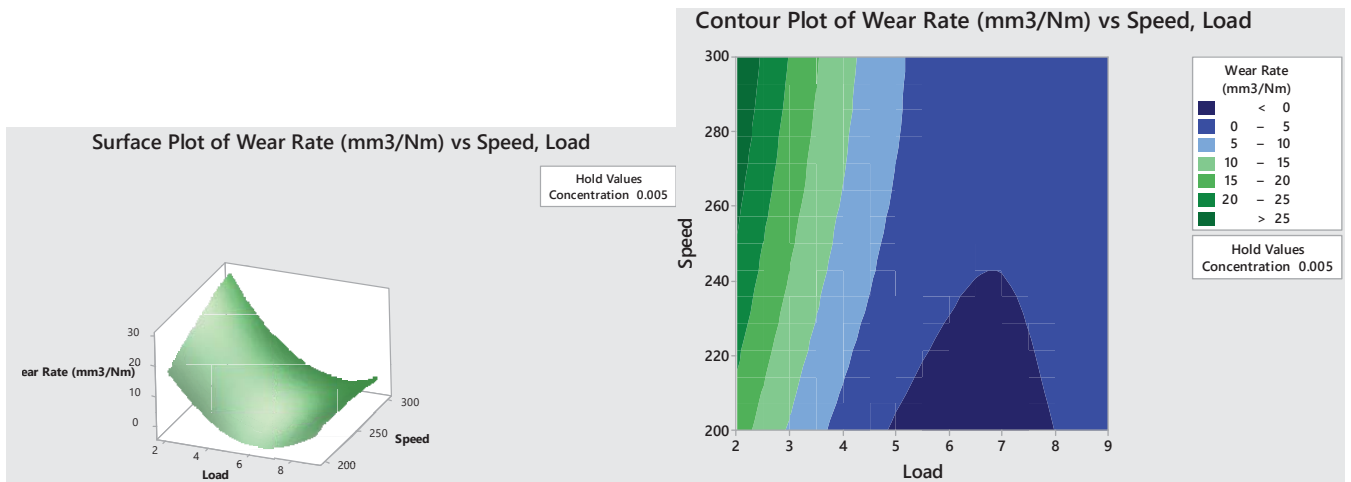


(b)

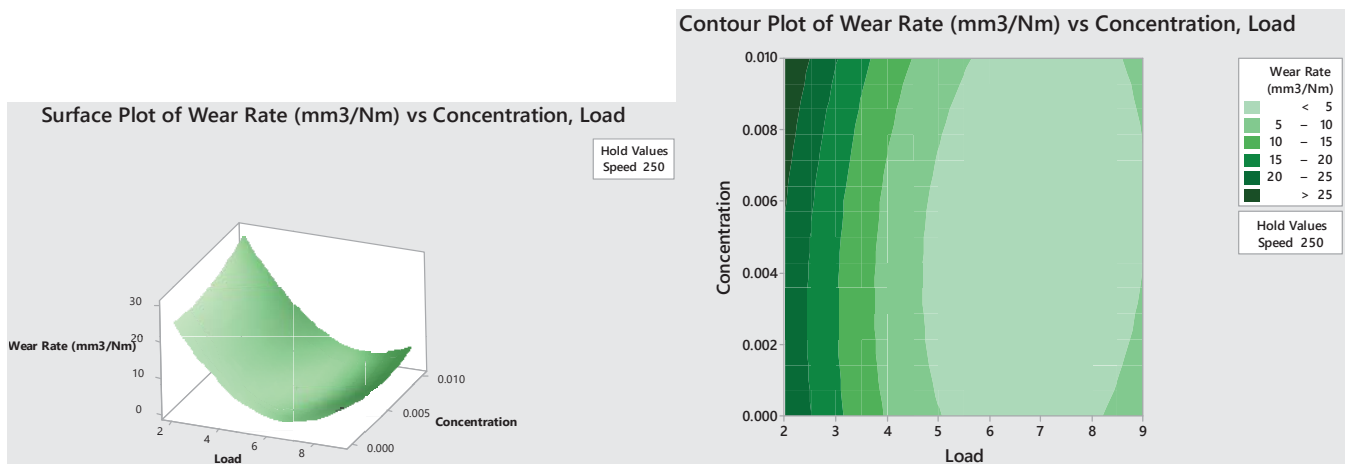


(c)

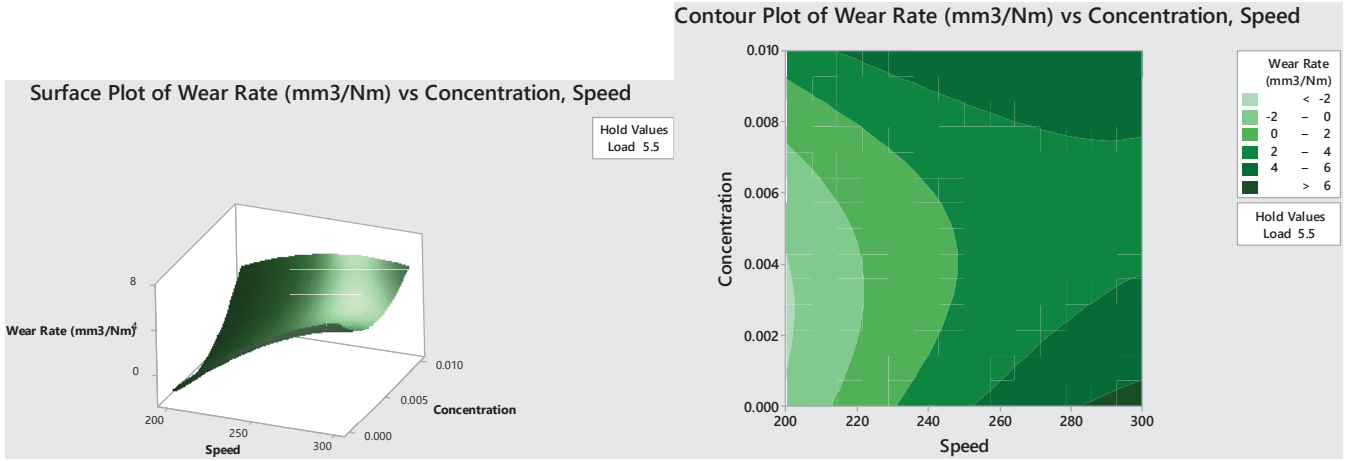
Fig. 3 Surface plots and contour plots of the combined effect of the input variables of COF; (a) load-speed, (b) speed-concentration and (c) load-concentration



(a)



(b)



(c)

Fig. 4 Surface plots and contour plots of the combined effect of the input variables of wear rate; (a) load-speed, (b) load-concentration and (c) speed-concentration

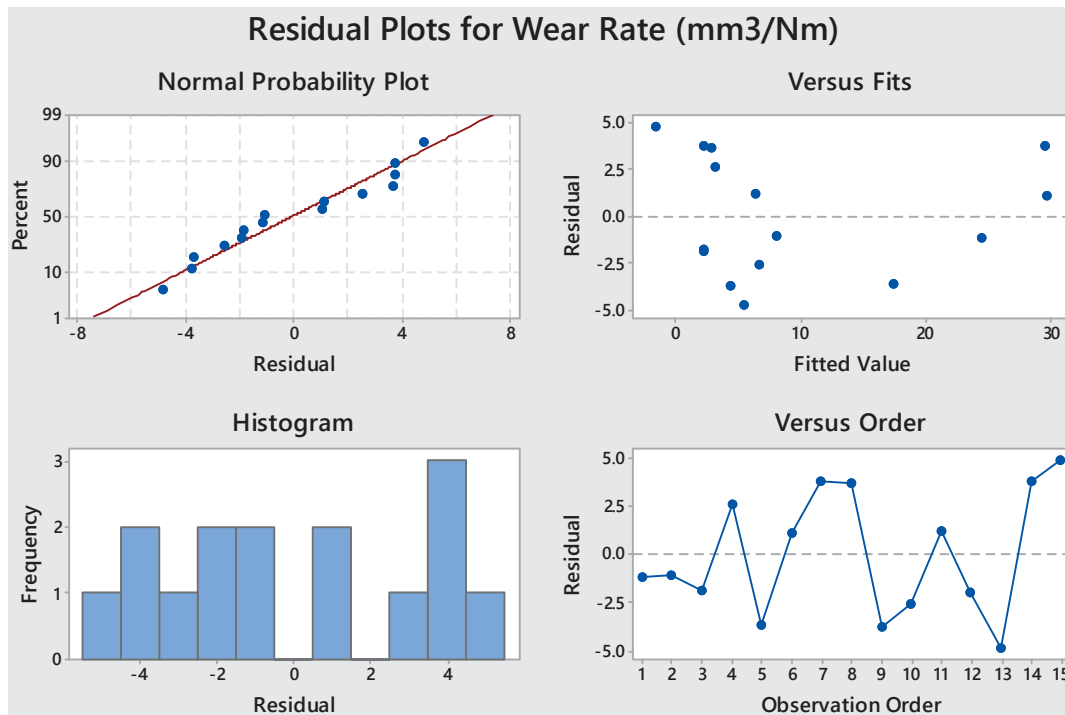


Fig. 5 Residual Plots of data obtained for wear rate.

C. Optimization of Tribological Behaviour

The main advantage of using the response surface methodology is that the responses can be optimized by controlling the parameters. The tribological performance of a material does not only depend on the characteristics of the CuO nanoparticles but also on the load, sliding distance, speed and other factors. In the majority of cases, the development of a nanolubricant with lower wear rate and COF is preferred because it can provide longer service life. In the present study, the optimization process was carried out with the aim of reducing friction and wear of the lubricant using the studied

variables. The optimization of the two responses was conducted but one response was compromised since it was impossible to reduce both simultaneously with the same variables.

TABLE V
 TARGET VALUE AND UPPER VALUE OF RESPONSES OF NANOLUBRICANT

Responses	Target Value	Upper Value
COF	0.061250	0.4548
Wear Rate	0.248195	33.3468

Table V presents the target values and upper values for both wear rate and COF of the nanolubricant while Fig. 6 shows the optimization of the responses of the nanolubricant. The predicted operating conditions required for the input variables to achieve minimum wear and COF for the nanolubricant were; a load of 7.5152 N, speed of 291.3360 rpm and concentration of 0.0086 wt% with a composite desirability of 1.0.

IV. CONCLUSION

This study has examined the effects of control parameters, namely, load, concentration and speed, on the COF and wear rate of a lubricant added with CuO nanoparticles. The following conclusions can be drawn from the findings of this study:

- i. The predicted optimized parameters required to produce lower responses of COF and wear rate when using nanoparticles as an additive to improve the lubricant were a load of 7.5152 N, speed of 291.3360 rpm and at 0.0086 wt% concentration. These optimized parameters produced better tribological behaviour compared to that of the parameters without nanoparticles as additive.
- ii. The relationships between the control parameters (load, speed, and concentration) and responses (wear rate and average COF) with CuO nanoparticles as an additive in the lubricant gave a better performance through the use of RSM. The parameters had significant effects on the tribological behaviour of the lubricant.

According to these results, it can be concluded that the CuO nanoparticles can be used as an additive in a lubricant to reduce wear and friction.

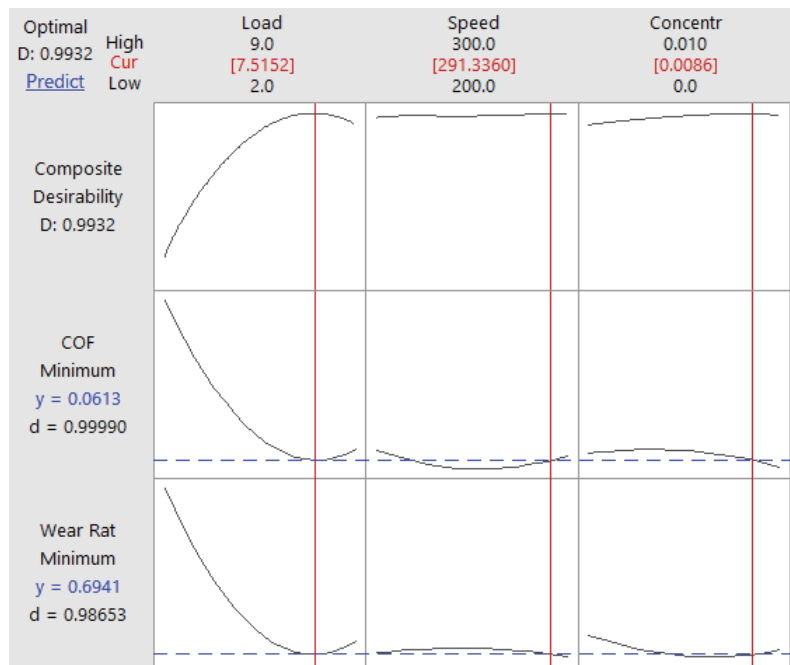


Fig. 6 Optimal conditions for the control variables on the wear rate and friction responses of the nanolubricant.

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