Application of Response Surface Methodology in Characterization of Biolubricant with Titanium Oxide Nanoparticles

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ABSTRACT

An optimized model is often deployed to reduce trial and error in experiment approach and to obtain the multi-variant correlation. In this study, Response Surface Methodology (RSM) namely Box-Behnken design (BBD) approach has been used to investigate the characteristic of lubricant. In BBD, this approach is based on multivariate analysis whereby the effect of different parameters is considered simultaneously. The effect of three parameters namely speed, load and concentration of TiO_2 *on the coefficient of friction (CoF) was investigated in this study. By using this approach, the number of experiment has reduced to 15 from 100 experiments using optimization method. The result obtained from BBD has shown that the most influential parameters were speed and load. Analysis of variance (ANOVA) indicated that the proposed experiments from quadratic model successfully interpreted the experimental data with a coefficient of determination* $R^2 = 0.9931$*. From the contour plot of BBD, the optimization zone for interacting variables can be determined. The zone that indicates two regions of lower friction values (<0.04) were: (i) at a speed 1300 to 2000 rpm for a normal load at 10 to 16 kg and (ii) at a speed 700 to 1500 rpm for a normal load in the range of 19 to 20kg. The optimized condition shows that the minimum value of CoF (0.0159) is at the speed of 2000 rpm, load of 10 kg and* TiO_2 *concentration of 1.0 wt%.*

Keywords: *Lubricant, Nanoparticles, Box-Behnken Design, Response Surface Methodology (RSM), Coefficient Of Friction.*

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Introduction

Lubricants are often deployed on the engine components which become in contact to reduce friction and wear. Friction is the force resulting from the opposite movement of sliding surface between mechanical parts in the engine such as piston ring, cylinder or bearing. From previous research, they indicate that proportion energy output of fuel in a car engine breaks down the exhaust, cooling agent and mechanical energy system [1]. Therefore, the level of friction should be minimal as possible in order to protect the engine parts against wear as well as to enhance the efficiency of the engine. Therefore, the engine life will be prolonged.

The properties of lubricant play a prominent role to fulfill desired requirements of the engine. Lubricant have certain properties that were designed based on their operating conditions. In order to improve the lubricants performance, by adding additive it may enhance an already-existing property of the base fluid or develop a new property. In automotive applications, the conventional additives used are antioxidants and extreme pressure agents (EP) additives such as sulfur, chlorine and phosphorus [2]. These EP additives inhibit excessive wear influenced by metal-to-metal contact under extreme loads [3]. However, these additives have been restricted due to their environmental impacts.

Since the use of advancing in the technology of additive, nanoparticles additive appears as an alternative to substitute the conventional additives as they may ensure the smooth performance of engine components, more remarkable and environmental-friendly [4–7]. Lubricant displayed significant improvement in the tribological properties when added with nanoparticles such as copper oxide [8], aluminium oxide, graphite [9] and titanium oxide [10]. The addition of nanoparticles in different shape, size and concentration may affect the level of friction and wear reduction [4–6].

The main issues related to possible mechanisms due to the presence of nanoparticles in lubricant are: (i) the spherical shape of nanoparticles have the effect of rolling mechanism, (ii) mending effect mechanism makes nanoparticles deposited physical tribofilm formation [5,7,8], and (iii) nanoparticles and wear debris react chemically and creates thin protective coating between contact surfaces.

In spite of several studies on the effect of nanoparticles additive in biolubricant previously [12,13], this proposed research is feasible for understanding the effects of TiO_2 nanoparticles in biolubricant with varying speed and load operating conditions.

Response Surface Methodology

The response surface methodology (RSM) is one of the most widely used mathematical and statistical techniques. This method comprises of statistical and mathematical technique that is useful to determine optimum operating conditions based on several experimental data [9–11]. Besides that, RSM is also defined by a statistical method that deploys quantitative data from experimental work to establish and resolve multi-variable equations. The purpose of using RSM is to explore the relationships between several independent variables (factors) and various responses (output). Moreover, by using design of experiments (DOE), it may analyse the interactions between input variables that are related to output variables and identify the optimal response within the experimental region [9, 12, 13]. A mathematical model was produced using Box-Behnken design (BBD). This mathematical model has enabled statistical analysis of the relationship between input variables and the output variable namely coefficient of friction (CoF). Three parameters were regulated: speed (X_1), load(X_2) and concentration of TiO_2 (X_3).

Methodology

The response surface methodology (RSM) associated with the experimental design is applied in this study for illustrating the multiplicity of the self-determining input variables and construct mathematical models. This will contribute in inspecting an appropriate measuring relationship between input variables and the output reactions.

In the present work, mathematical models were developed in order to predict the CoF and to conduct a statistical analysis of the independent variables interactions on the response surface, by using Minitab 16.0 statistical software. Three CoF key variables (speed, load and concentration) will be modelled based on response surface methodology (RSM) with Box-Behnken experimental design technique (BBD).

Using Box-Behnken approach, a set of experimental design was generated as shown in Table 1. In the experimental design, three parameters (speed, load and concentration of TiO_2) were assigned at different configuration for each run and 15 experiments were examined. As shown in Table 2, the three parameters chosen for this study were designated as X_1, X_2 and X_3 and prescribed into three levels, coded +1, 0 and -1 for high, intermediate and low value respectively.

Table 1: The BBD matrix design.

Run order	Coded variables			Real variables		
	x_1	**x_2**	**x_3**	**X_1**	**X_2**	**X_3**
1	+1	-1	0	1100	15	0.5
2	+1	0	-1	1100	10	0.0
3	-1	0	+1	200	15	0.0
4	+1	0	+1	1100	20	1.0
5	+1	+1	0	1100	15	0.5
6	0	0	0	2000	20	0.5
7	0	0	0	2000	15	1.0
8	-1	-1	0	200	10	0.5
9	0	-1	-1	200	15	1.0
10	0	+1	+1	1100	15	0.5
11	-1	+1	0	200	20	0.5
12	0	0	0	2000	15	0.0
13	-1	-1	-1	1100	10	1.0
14	0	0	-1	2000	10	0.5
15	0	0	+1	1100	20	0.0

Table 2: Experimental level of independent variable selected.

Variables	Symbol		Coded levels		
	Uncoded	**Coded**	**-1**	**0**	**+1**
Speed (rpm)	X_1	x_1	200	1100	2000
Load (kg)	X_2	x_2	10	15	20
Concentration (wt%)	X_3	x_3	0	0.5	1

Result and Discussions

The relationship between the response variable (CoF) and the three independent variables (speed, load and concentration of TiO_2) are shown in Equation (1). The estimated regression model for CoF with uncoded variables is shown below:

$$\begin{aligned} CoF = {} & 0.235268 - 0.000164\,x_1 - 0.009209\,x_2 + 0.012886\,x_3 + \\ & 0.000064\,x_2^2 - 0.056817\,x_3^2 + 0.000005\,x_1x_2 + \\ & 0.000014\,x_1x_3 - 0.001211\,x_2x_3 \end{aligned} \quad (1)$$

The result indicates that the value of R^2 is 0.9931 at a confidence level of 0.95. Therefore, it examines that the response performed in this study is highly significant where this model yields beyond 0.8 [18]. On the other hand, the results of ANOVA and estimated regression coefficient illustrated in Table 3 and 4 respectively, clearly prove that the fit with an $R^2_{(adj)}$ value of 0.980 is satisfactory effective.

Apart of that, the results of the estimated regression coefficients as given in Table 4 shows that the *p*-values of squared terms for speed are comparatively low whereas load and concentration are comparatively high with 0.013 and 0.419 respectively. In this context, the coefficients for the squared terms, speed and concentration are shown to be very significant where these factors have a large effect on the friction coefficient. For the interaction effects; (i) interaction between speed and load and (ii) speed and concentration are shown to be significant.

Table 3: Analysis of varience (ANOVA) results for acquired model.

Source	DF	Seq SS	Adj SS	Adj MS	F-value	*p*-value
Regression	9	0.010003	0.010003	0.001111	79.66	0.000
Linear	3	0.005373	0.005250	0.001750	125.44	0.000
Square	3	0.002355	0.002355	0.000785	56.27	0.000
Interaction	3	0.002275	0.002275	0.000758	54.35	0.000
Lack-of-fit	3	0.000056	0.000056	0.000019	2.81	0.274
Pure Error	2	0.010073				

Table 4: Estimated Regression Coefficients for CoF

Source	Coef	SE Coef	T-value	*p*-value	Character-istics
Constant	0.235268	0.019670	11.960	0.000	significant
speed	-0.000164	0.000009	-19.178	0.000	significant
load	-0.009209	0.002421	-3.805	0.013	significant
concentration	0.012886	0.014623	0.881	0.419	not significant
speed*speed	0.000000	0.000000	10.131	0.000	significant
load*load	0.000064	0.000078	0.825	0.447	not significant
concent.*concent.	-0.056817	0.007775	-7.307	0.001	significant
speed*load speed	0.000005	0.000000	12.232	0.000	significant
***concentration load**	0.000014	0.000004	3.284	0.022	significant
***concentration**	0.001211	0.000747	1.621	0.166	not significant

$R^2 = 0.9931$; $Adj\text{-}R^2 = 0.980$

Likewise, the experimental and predicted friction coefficient (CoF) values are observed to display a very high concurrence among them as shown in Table 5.

Table 5: The BBD Matrix with Observed and Predicted Values of CoF.

Run order	**Coded variables**			**Real variables**			**Response**		**Error (%)**
	x_1	x_2	x_3	X_1	X_2	X_3	**Experimental**	**Predicted**	
1	+1	-1	0	1100	15	0.5	0.05521	0.05321	3.62
2	+1	0	-1	1100	10	0.0	0.05393	0.05450	1.06
3	-1	0	+1	200	15	0.0	0.09216	0.09497	3.05
4	+1	0	+1	1100	20	1.0	0.03335	0.03278	1.72
5	+1	+1	0	1100	15	0.5	0.05414	0.05321	1.71
6	0	0	0	2000	20	0.5	0.06374	0.06713	5.31
7	0	0	0	2000	15	1.0	0.03751	0.03470	7.50
8	-1	-1	0	200	10	0.5	0.13097	0.12758	8.26
9	0	-1	-1	200	15	1.0	0.06982	0.07193	3.03
10	0	+1	+1	1100	15	0.5	0.05029	0.05321	5.81
11	-1	+1	0	200	20	0.5	0.07248	0.07094	2.12
12	0	0	0	2000	15	0.0	0.03532	0.03321	5.99
13	-1	-1	-1	1100	10	1.0	0.03640	0.03767	3.50
14	0	0	-1	2000	10	0.5	0.03085	0.03239	4.99
15	0	0	+1	1100	20	0.0	0.03877	0.03750	3.29

Model Accuracy Check

Figure 4 demonstrates the linear relationship between the predicted and experimental CoF values using Equation (1). The results indicated that the CoF is distributed relatively near the straight line, and sufficient correlation exits between these values. Furthermore, a normal probability plot of residuals was also obtained to evaluate the assumptions of populations being sampled whether they are normally distributed or not. Figure 5 shows the relationship between normal probability and residuals. From the graph, they are likely clustered around blue line indication, which supported the claim that the residuals are normally distributed. Thus, our assumption of normality is valid.

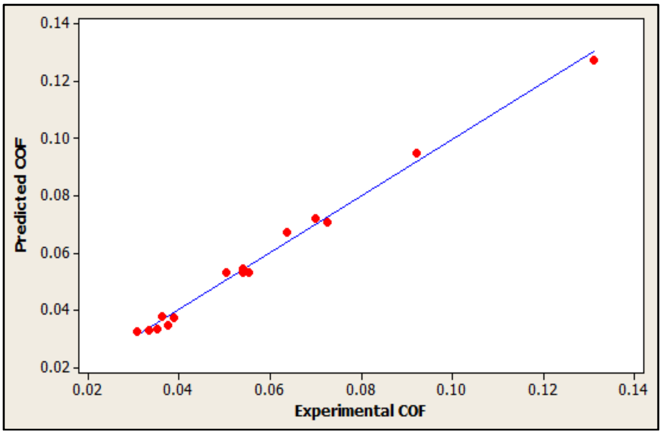

Figure 4: Comparison of predicted and experimental CoF of bio-lubricant oil.

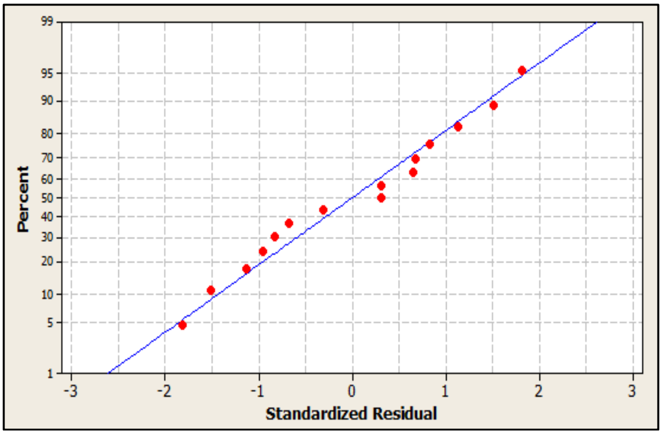

Figure 5: Normal plot residuals showing the relationship between normal probability and residuals.

Response Surface Analysis

The relationships between the CoF and these parameters are shown in Figure 6. Each plot represents the effects of two variables within their studied ranges, with the other variable is fixed to zero level. The response surface visualizes the tendency of each factor that influences the CoF. The shape of the contour plot indicates the natures and extents of interactions between factors. From the ANOVA, speed and load show the most significance of interaction variables.

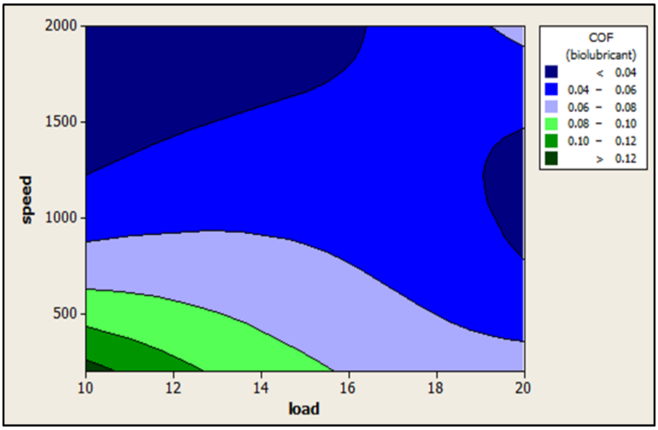

Figure 6: Contour plot of the predicted CoF on the effect of speed and load.

The zone indicates two regions of lower friction values (<0.04): (i) at speed 1300 rpm to 2000 rpm for a normal load at 10 kg to 16kg and (ii) at speed 700 rpm to 1500 rpm for a normal load in the range of 19 kg to 20 kg. From the results, it indicates that friction coefficient may increase or decrease depending on the sliding speed and load.

Optimization of CoF

Based on the model, the CoF was predicted by optimizing conditions in order to identify the minimum value of friction. The optimized condition shows that the minimum value of CoF (0.0159) is at the speed of 2000 rpm, load of 10 kg and TiO_2 concentration of 1.0 wt%. Further analysis was performed at optimum conditions in order to verify the predicted CoF. The CoF of the experimental value indicated an error between the observed and predicted values as shown in Table 6.

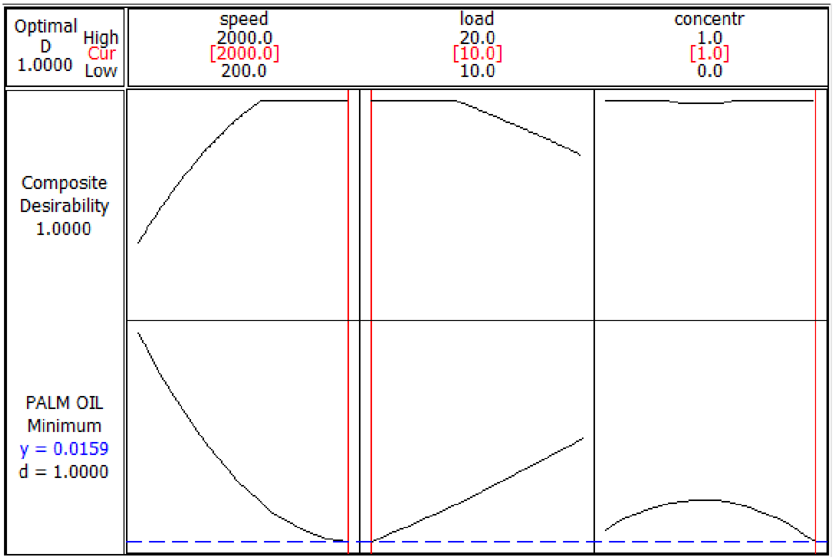

Figure 7: Response Optimization values of COF

Table 6: Optimum conditions, predicted and experimental value of COF

Optimum Condition			**CoF value**		**Error**
***x*$_1$ (rpm)**	***x*$_2$ (kg)**	***x*$_3$ (wt %)**	**Predicted**	**Observed**	**(%)**
2000	10	1.0	0.0159	0.0163	2.52

Conclusion

i. The ANOVA analysis reveals that speed and load are the main parameters which have greater influence than concentration. The interaction of input variables indicates speed and load and speed and concentration have a significant effect on friction.

ii. The zone indicates two regions of lower friction values (<0.04):
(a) at a speed of 1300 rpm to 2000 rpm for a normal load at 10 kg to 16kg and (b) at a speed of 700 rpm to 1500 rpm for a normal load in the range of 19 kg to 20 kg.

iii. The optimized condition shows that the minimum value of CoF (0.0159) is at the speed of 2000 rpm, load of 10 kg and TiO_2 concentration of 1.0 wt%.

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