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# Wear analysis of CNT-AL Nanocomposites using surface response method

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## ABSTRACT

*Carbon nanotube (CNT) reinforced aluminium (Al) nano-composites were produced using powder metallurgy route with different weight percent of CNT in to the Al matrix. The wear behaviour of CNT-Al nano-composite was studied using a pin-on-disc tribometer against AISI4340 steel disc. Experiments were conducted using different sliding velocities of 0.5, 0.65 and 0.8 m/s and a normal load of 5, 7.5 and 10 N. Design Expert (DOE) version 6.0.8 was used to optimize the process variable for wear and friction test of the developed CNT-Al nano-composite using face centered central composite design based on response surface methodology and confirmed that 1.5 wt% was the optimum CNT-Al nano-composite formulations. The result showed that higher hardness value of the material showed a lesser wear rate and better wear resistance. The result showed that the wear rate of the developed nano-composite decreased with the increasing of CNT content but up to 1.5 wt% CNT into the nano-composite. The wear rate values varied from 2-0.6 x$10^{-3}$ $mm^3$/m (2, 0.2, 0.17. 0.3 and 0.6 $mm^3$/m x$10^{-3}$ for 0 wt%, 1 wt%, 1.5 wt%, 2 wt% and 2.5 wt% CNT respectively). These results also showed that the rate of wear decreases with the increase in normal load and sliding speed. DOE confirm the accuracy of the experimental result for the evaluation of the developed CNT-Al nano-composite.*

**Keywords:** Wear testing, Sliding wear, Wear modelling, Metal-matrix composite and CNT-Al nano-composite

## 1. INTRODUCTION

Aluminium in its pure form is very soft and cannot withstand excessive wear and friction during operation. Recently, there is a high demand to improve its mechanical properties, wear and frictional resistance due to the high demand of lightweight, energy servings and efficient material. The uses of aluminium alloys in some industries such as automotive have been limited by their substandard strength, rigidity and wear resistance, compared aluminium based composite especially nano-composite materials [1-3]. Aluminium nano-composites, however, offer reduced mass, high stiffness and strength, and improved wear and frictional resistance. From the wear map it is observed that, during dry sliding wear an increase of any of the operating condition such as normal applied load, sliding velocity, or duration of rubbing leads at some stage to a sudden change in the wear rate (weight loss per sliding distance). The simplest categories of the types of wear exhibiting these different wear rates is mild wear and severe wear. Mild wear marks a smooth surface that often is smoother than the original surface, with minimum plastic deformation and oxide wear debris [4, 5]. Severe wear results in a rough surface that is typically rougher than the original surface, with large plastic deformation and flake-like metallic wear debris [6, 7]. Today many researchers had introduced advance materials as a substitute to the traditionally used materials as these modified materials are light weight and excellent in wear and friction resistance, as well as improved life span [8, 9]. Scientific ideology and calculating methods of creating new materials/composites and estimation of its wear resistance of friction nodes as well as physical simulation of friction and wear processes on a small-sized laboratory test machine need to be carried out experimentally before a future material/composite is commercially introduced in the market. Before the advent of the proposed composite material, a suitable type of wear test equipment should be used together with a suitable parameters selected which reflect the real time application of the composite. Examples of this parameters are testing techniques, type of counterface used against the test samples, sliding velocities, sliding distances, applied loads, contact conditions and orientations of the test specimen with respect to the sliding direction of the counterface. Based on this idea the present authors proposed CNT-Al nano-composite material to be used for brake disc application and to be tested using a suitable technique before introduce in to the commercial sector. Wear and frictional behaviour of the developed CNT-Al nano-composite was analyzed using Design-Expert (DOE) as a suitable technique to design experiment for nano-composite material development using powder metallurgy route. The main aim of this study is to study and analyzed the wear analysis of the newly developed CNT-Al nano-composite using DOE and also to confirm the accuracy of the experimental results.

## 2. EXPERIMENTAL DETAILS

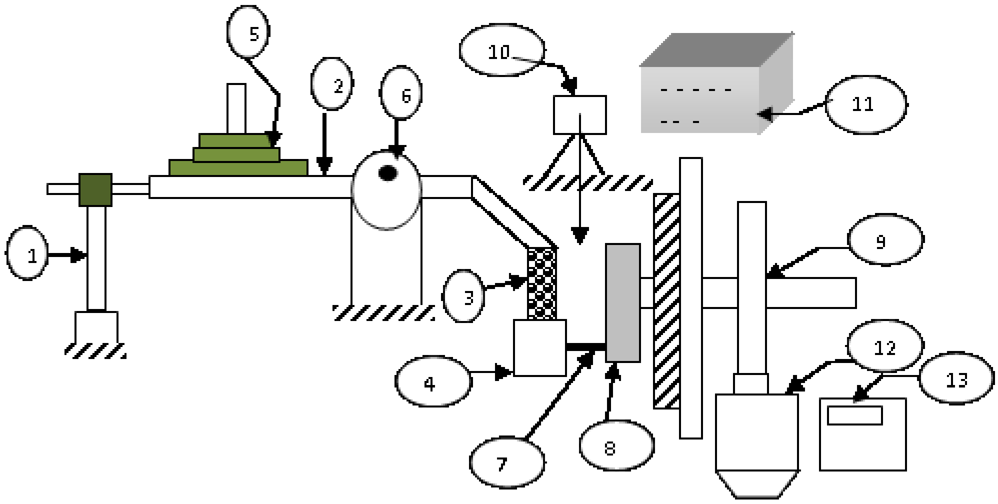
### 2.1 Materials and methods

The composite were fabricated using pure aluminium (Al) (99.7%), with particle size of 78 µm which has nearly spherical shape with some satellite sub-particles obtained from Innovative Pultrusion Sdn Bhd, a local supplier for the aluminium powder was used as a matrix material. The multi walled carbon nano tubes (MWCNTs) with a nominal diameter of 10 nm, length of 5-15 µm, and surface area of 230-280 $m^2$g-1 was also obtained from the same supplier in Malaysia and used as a reinforcement. Ethanol was used as a process control agent (PCA) during the ball milling of CNT and Al powders. The wear test was conducted using pin-on-disc machine according to ASTM G99 standard. The test was performed at room temperature and varying operating conditions for all the samples. A pin of size 9 x 9 x 20 mm (W x H x L) made of CNT-Al nano-composite with AISI 4340 steel disc as counterpart material was used. The detail experimental conditions are presented in Table 1. The experiment was conducted at different loads, speeds and at a certain time. Figure 1 shows a block diagram of a pin-on-disc tribometer which comprises of a pin (in this case CNT-Al nano-composite) that slides on a rotating vertical disc. A square shape sample (pin) was fixed in to the sample holder using a pin and a bolt, and this assembly was attached to the circular rotating disc (in this case AISI 4340 steel) as a counterpart having a long vertical shaft welded from the bottom surface of the rotating disc. The rigidity of the main structure of the pin-on-disc machine setup was gained from the three supporting square plates along with a base plate that are fixed with four vertical square bars. The base plate was fastened with the foundation and a half-horsepower motor was mounted vertically on a separate table with a dumper to rotate the shaft, the separation is to minimize the effect of vibration of the motor during the test. Weights are loaded to provide the required contact pressure, and the rotational speed was set to start the experiment.

Table 1: Experimental conditions for wear and friction test

| **Operating conditions** | **Values** |
|---|---|
| Duration of rubbing (min) | 12 |
| Wheel speed (rpm) | 42, 54 and 66 |
| Sliding speed (m/s) | 0.50, 0.65 and 0.8 |
| Frequency (Hz) | 0.7, 0.9 and 1.1 |
| Normal load (N) | 5.0, 7.5 and 10 |

| | |
|---|---|
| Materials:<br>(i) Pin<br>(ii) Disc | <br>Pure aluminium and CNT-Al nano-composite<br>AISI 4340 hardened steel (Hardness value, 650 Brinel hardness) |




1. Load arm holder
2. Load arm
3. Load cell
4. Sample holder
5. Weights
6. Pivot
7. CNT-Al nano-composite sample
8. Counterpart
9. Main shaft
10. Thermocouple
11. Load cell indicator
12. Motor
13. Speed control unit

Fig 1: Schematic illustrations of pin-on-disc tribo-meter

### 2.2 Experimental design

The main objective of the experimental design is to study the relationship between the response as the dependent variable and the various parameter levels in order to achieve the optimum level. For this study optimization of the process variable for wear and friction test of the developed CNT-Al nano-composites was accomplished through Design Expert version 6.0.8 using face centered central composite design based on response surface methodology with three factors such as duration of test (time), normal applied load and sliding speed, and the two response were coefficient of friction (COF) and wear rate (WR). Table 2 shows the coded factors and the levels, whilst, Table 3 shows the design matrix for wear and friction test with three factors.

Table 2

Experimental input factors selected for wear and friction properties

| Input factors | Units | Level | | |
|---|---|---|---|---|
| | | -1 | 0 | +1 |
| A | min | 1 | 6 | 12 |
| B | N | 5 | 7.5 | 10 |
| C | m/s | 0.5 | 0.65 | 0.8 |

-1 is the lower coded factor
0 is the center factor always generated by the software
+1 is the upper coded factor

Table 3

Experimental design matrix for wear and friction test

| Run | Experimental Block no. | Factor A: Time (min) | Factor B: Load (N) | Factor C: Sliding speed (m/s) |
|---|---|---|---|---|
| 1 | 1 | 6 | 7.5 | 0.65 |
| 2 | 1 | 6 | 7.5 | 0.65 |
| 3 | 1 | 12 | 5 | 0.8 |
| 4 | 1 | 1 | 10 | 0.8 |
| 5 | 1 | 1 | 5 | 0.5 |
| 6 | 1 | 12 | 10 | 0.5 |
| 7 | 2 | 12 | 10 | 0.8 |
| 8 | 2 | 12 | 5 | 0.5 |
| 9 | 2 | 6 | 7.5 | 0.65 |
| 10 | 2 | 1 | 5 | 0.8 |
| 11 | 2 | 1 | 10 | 0.5 |
| 12 | 2 | 6 | 7.5 | 0.65 |
| 13 | 3 | 6 | 7.5 | 0.8 |
| 14 | 3 | 6 | 7.5 | 0.65 |
| 15 | 3 | 1 | 7.5 | 0.65 |
| 16 | 3 | 6 | 10 | 0.65 |
| 17 | 3 | 6 | 7.5 | 0.65 |
| 18 | 3 | 12 | 7.5 | 0.65 |
| 19 | 3 | 6 | 7.5 | 0.5 |
| 20 | 3 | 6 | 5 | 0.65 |

### 2.3 Statistical model

In order to forecast the optimal range of values for the sliding speed, load and rubbing time, two responses of wear rate and coefficient of friction with the experimental conditions (factors of time (A), load (B) and sliding speed (C) were considered and a second order polynomial model was developed using the experimental results. To optimise the response at all the levels of the selected experimental condition, the response of the input variable can be expressed as a function of A and B using the following equation:

$y = f(A, B, C) + \varepsilon$

(1)

where y is the response, ε is the noise or error observed in the response

The choice of the model equation was to allow the opportunity for both the second order (curvature) and the interaction effects among the variables. According to Montgomery (2009), the second order response of input variables equation can be presented as:

$y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{11} A^2 + \beta_{22} B^2 + \beta 33 C^2 + \beta_{12} AB$ ………..

(2)

where y = measured value of the response;

$\beta_0, \beta_1, \beta_2 \ldots\ldots\ldots\ldots \beta_{12}$ are the model coefficients to be estimated using experimental data;

A, B and C are the factors;

AB is the possible interaction effect;

$A^2$, $B^2$ and $C^2$ are the quadratic effects of the variables

In order to develop a statistical model equation in terms of coded factors (viz. lower (-1), upper (+1) and the center factor (0)) and the actual input factors (such as factor A, B and C for the two different output COF and WR) the equations were derived from the experimental data which expressed the relationship between the responses and the input factors.

Model equations in terms of coded factors:

$(COF)^I = [(COF-4.00)/(19.5-COF)] = 0.17+1.12A - 0.36B - 0.09C - 1.0A^2 + 0.63B^2 + 0.39C^2 - 0.18AB - 0.088BC$

$WR = 0.073+6.200E\text{-}003A+5.430E\text{-}003B+2.060E\text{-}003C-6.818E\text{-}003A^2-2.988E\text{-}003B^2-2.200E\text{-}00AC-2.200E\text{-}003BC$

Final equations in terms of actual factors are:

$(COF)^I$ = [(COF-4.00)/(19.5-COF)]= 3.192*(Time)-0.031*(Load)-10.991*(Speed)-0.033*$(Time)^2$ + 0.100*$(Load)^2$ + 17.454*$(Speed)^2$ - 0.013*(Time)*(Load) - 2.359*(Load)*(Speed)

WR = -0.040+5.245E-003*(Time) + 0.013*(Load) + 0.0992*(Speed) - 2.254E-004*$(Time)^2$ - 4.748E-004*$(Load)^2$ - 2.667E - 003*(Time)*(Speed) - 5.867E -003*(Load)*(Speed)

## 2.4 Model adequacy test

The competence of the model was tested by coefficient of determination R-squared and analysis of variance based on statistical test. For model selection attention was made and focus on the model that maximized the values for adjusted R-squared and the predicted R-squared, and the system suggested always to neglect the model that is aliased. It suggested two models for this experiment that is two factorial input (2FI) and quadratic model and based on focusing on the model with higher value of R- squared approaching '1', the quadratic model was chosen for this experiment as shown in Table 2 and 3. Table 4 and 5 present the reduced quadratic models and there terms for COF and WR respectively.

Table 2: Model summary statistics for COF

| **Source** | **Std. Dev.** | **R-Squared** | **Adjusted R-squared** | **Predicted R-squared** | **PRESS** | |
|---|---|---|---|---|---|---|
| Linear | 0.840257 | 0.68889 | 0.622224 | 0.098653 | 28.63721 | |
| 2FI | 0.551995 | 0.894507 | 0.836965 | 0.762154 | 7.556757 | |
| Quadratic | 0.183794 | 0.991494 | 0.981925 | 0.854498 | 4.622835 | Suggested |
| Cubic | 0.073673 | 0.999317 | 0.997096 | -1.33573 | 74.20992 | Aliased |

Table 3: Model summary statistics for WR

| **Source** | **Std. Dev.** | **R-squared** | **Adjusted R-squared** | **Predicted R-squared** | **PRESS** | |
|---|---|---|---|---|---|---|
| Linear | 0.011372 | 0.613104 | 0.530197 | 0.112917 | 0.004152 | |
| 2FI | 0.011589 | 0.6843 | 0.5121 | -2.64274 | 0.017048 | |
| Quadratic | 0.004311 | 0.968229 | 0.932487 | 0.375378 | 0.002923 | Suggested |
| Cubic | 0.001213 | 0.998742 | 0.994653 | -3.30061 | 0.020127 | Aliased |

Table 4: Analysis of variance for COF reduced quadratic model

| Source | Sum of squares | DF | Mean square | F Value | Prob > F |
|---|---|---|---|---|---|
| Block | 0.06371 | 2 | 0.031855 | | |
| Model | 31.50134 | 9 | 3.500149 | 103.6156 | < 0.0001** |
| A | 12.51976 | 1 | 12.51976 | 370.6252 | < 0.0001** |
| B | 1.283308 | 1 | 1.283308 | 37.99003 | 0.0003** |
| C | 8.084063 | 1 | 8.084063 | 239.3142 | < 0.0001** |
| $A^2$ | 2.661028 | 1 | 2.661028 | 78.77496 | < 0.0001** |
| $B^2$ | 1.058787 | 1 | 1.058787 | 31.34349 | 0.0005** |
| $C^2$ | 0.413966 | 1 | 0.413966 | 12.25472 | 0.0081** |
| AB | 0.267586 | 1 | 0.267586 | 7.921397 | 0.0227* |
| BC | 6.260871 | 1 | 6.260871 | 185.3419 | < 0.0001** |

Table 5:Analysis of variance for WR reduced quadratic model

| Source | Sum of squares | DF | Mean square | F- Value | Prob > F |
|---|---|---|---|---|---|
| Block | 7.88E-05 | 2 | 3.94E-05 | | |
| Model | 0.001144 | 9 | 0.000127 | 26.98968 | < 0.0001** |
| A | 0.000384 | 1 | 0.000384 | 81.64885 | < 0.0001** |
| B | 0.000295 | 1 | 0.000295 | 62.62769 | < 0.0001** |
| C | 4.24E-05 | 1 | 4.24E-05 | 9.01366 | 0.0170** |
| $A^2$ | 0.000125 | 1 | 0.000125 | 26.50039 | 0.0009** |
| $B^2$ | 2.36E-05 | 1 | 2.36E-05 | 5.021203 | 0.0554* |
| AC | 3.87E-05 | 1 | 3.87E-05 | 8.224359 | 0.0209* |
| BC | 3.87E-05 | 1 | 3.87E-05 | 8.224359 | 0.0209* |

## 3. RESULTS AND DISCUSSION

### 3.1 Observation

From the experimental results it was observe that, 1.5 wt% CNT-Al nano-composite showed lower wear rate the detailed wear morphology and wear surface characteristics were done on this nano-composite and explained in the following sub-sections.

### 3.2 Wear rate

The wear behaviour of the newly developed CNT-Al nano-composite was studied and analysed. Figure 2 shows the effect of CNT content on the wear rate of CNT-Al nano-composite. The result showed that the wear rate of the developed nano-composite decreased with the increasing of CNT content but up to 1.5 wt% CNT into the nano-composite. This result was consistent with the changes in the morphologies of the wear worn surface in terms of the cracks, debris and ploughing for all formulations of CNT-Al nano-composite. On the other hand the nano-composite with higher percentage of CNT exhibited higher wear.

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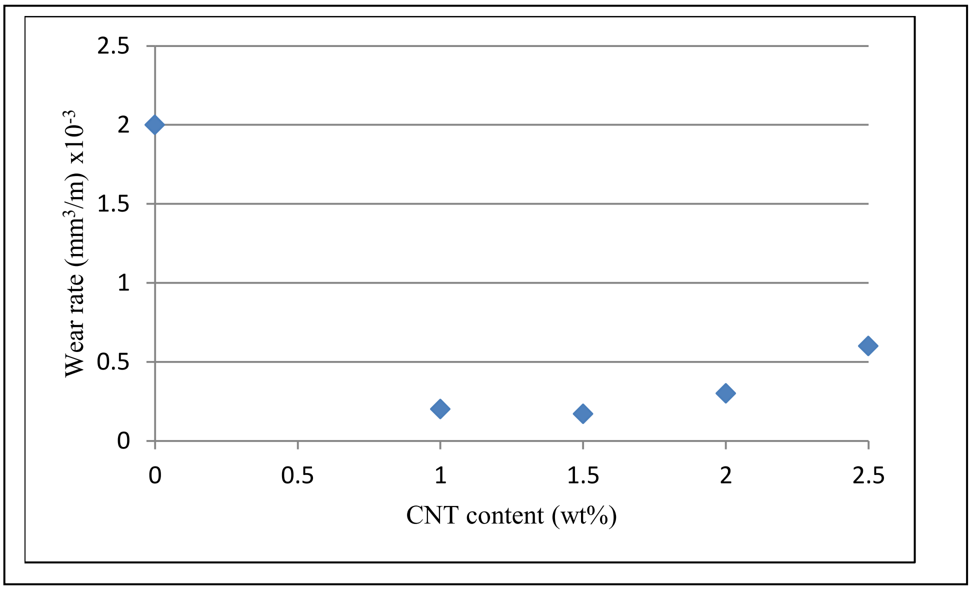


Fig 2: Effect of CNT content on wear rate of CNT-Al nano-composite

The results on the wear rate of CNT-Al nano-composite with respective to applied normal load is presented in Figure 3. The curve was plotted mainly to observe the difference of wear rate at varying loads and constant sliding speed of 0.5 m/s.

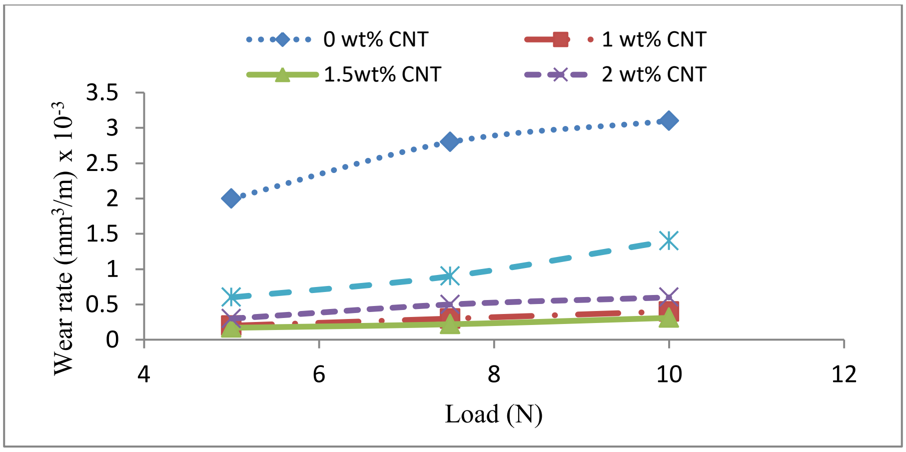


Fig 3: Effect of normal load on the wear rate of CNT-Al nano-composite

From Figure 3 it was found that, 0 wt% CNT (pure Al) has the highest wear rate and 1.5 wt% CNT nano-composite has the lowest wear rate, followed by 1, 2 and 2.5 wt% CNT content in CNT-Al nano-composite. Among the four formulations 2.5 wt% CNT-Al nano-composites showed the highest wear rate that is 3 times higher than that of 1, 1.5 wt% and 2 times higher than 2 wt% CNT formulation. The wear rate of 0 wt% CNT is 15 time higher than that of 1.5 wt5 that is the optimum value among the four formulations. This can be attributed to the brittle nature of the 2.5 wt% CNT-Al nano-composites on this. Another postulation is that formation of surface crack and induced void during rubbing action under applied load caused the plastic deformation with removal of material from the surface, hence, higher wear rate of the 2.5 wt% CNT-Al nano-composite. The counterpart material was hardened steel whose wear rate was considered negligible and hence only wear rate of CNT-Al nano-composite is presented. The wear rate values varied from 2-0.6 $x10^{-3}$ $mm^3/m$ (2, 0.2, 0.17. 0.3 and 0.6 $mm^3/m$ $x10^{-3}$ for 0 wt%, 1 wt%, 1.5 wt%, 2 wt% and 2.5 wt% CNT respectively). These results also showed that the rate of wear decreases with the increase in normal load. Effects of sintering also affect the wear resistance of the material. Higher CNT into CNT-Al nano-composite thus resulted in a higher wear rate of the material

### 3.3 DOE analysis

The modelling of coefficient of friction (COF) and wear rate (WR) during wear and friction test indicated that their variation was significantly influence by time, load and sliding speed as the factors considered for this experimental design. The reduced quadratic model via ANOVA as shown in Tables 4 and 5 indicated the strength of interaction between independent variables and their individual effects which was indicated by the coefficient and determined by the F and P-values. The lower the P-value the higher the significant of the corresponding coefficient. Based on ANOVA result presented before all the vaiables exhibited a highly significant effect in linear term for the responses of time and load and the interaction between load and speed as highly significant with regards to the COF.

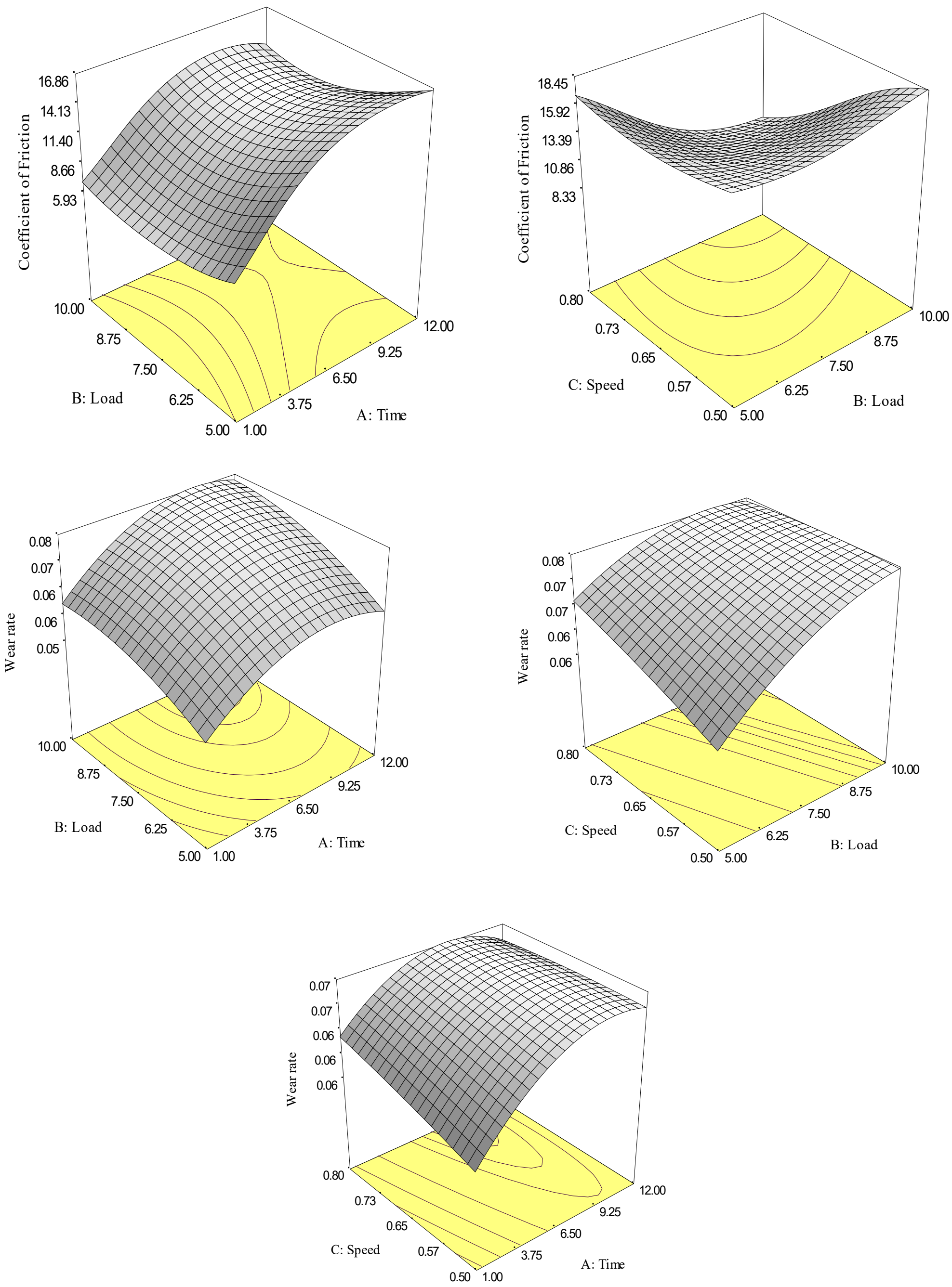


Fig 5: 3D response surface plot for wear behaviour of CNT-Al nano-composite for: (a) Load and time with fixed sliding speed, (b) Speed and load with fixed time, (c) Load and time with fixed speed, (d) Speed and load with fixed time and (e) Speed and time with fixed load.

Time and speed also shows a significant effect in squared for the first response. For the second response all the three variables shows a highly significant effect in linear and squared with only interaction between speed and load as significant model term. This indicated that all the parameters could act as a limiting parameter and a slight variation in it would noticeably affaect the wear rate and coefficient of friction during the wear and friction test. The observation was found by other researchers and in their study of dry sliding friction and wear behavior of aluminum-alumina composites using Taguchi technique [10]. Dvivedi et al., (2012), also claimed the same finding when studying the tribological characteristics of Al 6063–SiCp metal–matrix composite under reciprocating and wet conditions [11]. In friction and wear, normal applied load and sliding velosity (speed) plays a significant role with the duration of rubbing (time) at a certain point. A study was conducted using Taguchi and ANOVA on the tribological behaviour of Al-2014 alloy-10 wt% SiC composite and determine the significant factors when testing with Al 2219 SiC and Al 2219 SiC graphite material and concluded that the sliding distance, sliding velocity and load exhibit a significant effect on wear behaviour[12]. Fig. 5 is the response surface plot that shows the interaction oftwo factors at the same time with the other factor fixed. The plot was confirmed by the ANOVA and is a 3D graphical representation of the regression equation used to identify the optimum levels and the interaction among variables that were investigated for the two response considered in the wear and friction test of CNT-Al nano-composite and the graphical presented results gave an ideal interaction between the two independent variables.

## CONCLUSIONS

CNT-Al nano-composite shows lower wear rate than pure Al and wear rate of the tested materials increases with increase in normal applied load. The results also showed that wear rate decreased with the increasing of CNT content in CNT-Al nano-composite. The developed statistical models for the evaluation of CNT-Al nano-composite for wear properties adequately and significantly fitted to justify the optimization of 1.5 wt % CNT among other formulations. Interaction of two factors at a time via surface plots revealed that rubbing time, normal applied load and sliding speed showed a significant effect on the wear properties of CNT-Al nano-composite.

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