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Statistical approach for prediction of thermal properties of CNC and CNC-CuO nanolubricant using Response Surface Methodology (RSM)

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Statistical approach for prediction of thermal properties of CNC and CNC-CuO nanolubricant using Response Surface Methodology (RSM)

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Abstract. In the present work, response surface methodology (RSM) using the miscellaneous design model was performed to optimize thermal properties of Cellulose nanocrystal (CNC) and hybrid of cellulose nanocrystal-copper (II) oxide (CNC-CuO) nanolubricant. Influence of temperature, concentration and type of nanolubricant is used to develop empirical mathematical model by using Response Surface Methodology (RSM) based on Central Composite Design (CCD) with aid of Minitab 18 statistical analysis software. The significance of the developed empirical mathematical model is validated by using Analysis of variance (ANOVA). In order to produce second-order polynomial equations for target outputs including thermal conductivity and viscosity, 26 experiments were performed. According to the results, the predicted values were in sensible agreement with the experimental data. In other words, more than 80% of thermal conductivity and specific heat capacity variations of the nanolubricant could be predicted by the models, which shows the applied model is precise. The predicted optimized value shown in the optimization plot is 0.1463 for thermal conductivity and 1.6311 for specific heat capacity. The relevant parameters such as concentration, temperature and type of nanolubricant are 81.51°C, 0.1, and the categorical factor is CNC-CuO. The composite shown in the plot is 0.6531. The validation result wit experimental as shown in indicate that the model can predict the optimal experimental conditions well.

Keywords. Specific heat capacity; Thermal conductivity, Cellulose nanocrystal, Hybrid of cellulose nanocrystal-copper (II) oxide, Nanolubricant

1. Introduction

Currently, optimizing the energy consumption in an engine is an issue which has attracted researchers. On the other hand, storage of requirement energies and increasing their efficiency are among the most fundamental of the industry. In internal combustion engine, engine oil is a crucial fluid to prevent corrosion of parts and reduce the abrasion wear caused by constant contact. Furthermore, engine oil is important to reduce corrosion and friction, cooling, dissipation of heat from engine, and sealing of



parts. Hence it can be assumed that appropriate use of engine oils improves engine efficiency, durability and also fuel consumption [1-3]. Various study have been conducted for better thermal properties of lubricants. One of the current trend to improve thermal properties is by adding nanomaterials to the base fluid to improve heat transfer process in systems [4, 5]. In 1873, Maxwell was the pioneer that succeeded in improving the heat transfer rate of liquids by addition of solid particle into them [6]. Unfortunately, there is still much drawbacks involved in this method such as pressure drop, precipitation, corrosion and impurities. However, as the development of the technology, many drawbacks was already overcome by various researchers [7-11].

Nanofluid are a category of fluid with a high heat transfer potential. They are prepared by the dispersion and suspension of metal (inorganic) and nonmetal (organic) nanoparticles of smaller than 100nm in a base fluid such as water, oil and ethylene glycol [12-18]. There are various techniques for heat transfer enhancement. These techniques consist of disturbing boundary layers, and improving thermophysical properties of fluids such as the thermal conductivity increase by adding solid particles to a common liquid. Mentioned fluids were called nanofluids for the first time. Among all the physical properties of nanofluids, thermal conductivity is an important feature which should be examined. Thus, many researchers have conducted different experimental and numerical studies on estimating thermal conductivity and viscosity of nanofluids [10, 19-27].

Hemmat Esfe et al. [28] experimentally investigated the thermal conductivity of MgO/EG nanofluid. They proposed a thermal conductivity model in terms of temperature, solid concentration, and particle size using ANN model. Moreover, they presented two new correlations for viscosity of the nanofluid based on solid concentration, and temperature [29]. Hemmat esfe et al. also conducted an experimental study to determine thermal conductivity and viscosity of ethylene glycol based nanofluids containing Mg (OH)₂ nanoparticles [30]. Hemmat Esfe et al. studied the thermal conductivity of nanofluids including Al₂O₃ nanoparticles with average diameter of 5nm which were suspended in water, experimentally. Thermal conductivity of Al₂O₃/water was measured in a temperature range between 26 and 55 °C. The results indicated that thermal conductivity of nanofluids was enhanced significantly by increasing temperature at any concentration [29]. Putra et al. [31] experimentally studied the thermal conductivity of Al₂O₃/water nanofluid whose nanoparticles have an average size of 131 nm. A steady-state parallel plates approach was utilized to measure the thermal conductivity. The results showed by increasing the nanofluid concentration to 4%, the thermal conductivity of the nanofluid. Etefaghi et al. [32] studied the thermal conductivity of the MWCNT-oil in a limited range of solid concentrations and at the temperature of 20 C. Their results indicated that the thermal conductivity of the nanofluid increases as the solid concentration increased. They reported 20% enhancement in thermal conductivity of the nanofluid. In another experimental investigation, which is recently published, Asadi et al. [12] studied the rheological behavior of MWCNT/MgO-SAE50 hybrid nano-lubricant. They studied the effect of temperature and solid volume fraction on dynamic viscosity of the nano-lubricant and found that the dynamic viscosity increased by increasing the solid volume fraction in all the temperatures. Toghraie et al. [33] examined the effect of temperature and particle concentration on the thermal conductivity of hybrid nanofluids of ZnO–TiO₂/EG by varying the temperature and nanoparticles volume fraction in the rage of 25–50 °C and 0–3.5 vol% respectively. They found that the thermal conductivity of the prepared hybrid nanofluid improved by increasing both the variables (temperatures and particle volume fraction).

Notable, the most important include thermal conductivity viscosity and specific heat capacity. It has been observed that majority of these studies are based on thermal conductivity while roughly 5% of the literature covers the specific heat capacity. Lubricants with a larger specific heat value lead to a smaller temperature rise for a given amount of heat energy absorption. The heat transfer properties of lubricants for low heat rejection (LHR) diesel engines are particularly important. Uncooled LHR engines do not have oil cooler and radiator, being the engine oil the only heat transfer fluid [34]. However, experimental heat capacity values are still not available for most ionic liquids and, in some cases, the uncertainty of literature data is too high, with deviations among the published values up to 18% [35, 36]. RSM, which is a collection of mathematical and statistical techniques, is used to

optimize the processes that their answers are affected by a number of variables. Mathematical model of the graphical schema provides a common definition of the response surface. By using this statistical design, the number of tests reduces. Moreover, all coefficients of the quadratic regression model and interaction factors become estimable. In this paper, In order to study the effect of main factors (temperature, concentration and type of nanolubricant) and interaction factors, the response surface model (RSM) has been applied to identify the correlation of thermal conductivity and specific heat capacity

2. Methodology

2.1. Nanolubricant preparation and thermal properties measurement

CNC and hybrid CNC-CuO nanoparticles were used in the present experimental study. CNC were procured from Blue Goose Biorefineries Inc with weight concentration at 7.4 wt%. CNC is extracted from the acetate grade dissolving pulp that is the Western Hemlock is to be in the white to slightly off white cream form. The CNC then was synthesis into the powder form. Removing water from CNC suspension was important in order to maintain nano-scale dimension of the nanofibrils and because of their hydrophilic nature, CNC neither melts at high temperature nor dissolve-es in common aqueous solvent, a major processing challenge to properly dry CNC from aqueous suspension [37]. Yuan et al suggested spray drying is appropriate process to dry the nanocellulose suspension [38]. Spray drying was conducted using a laboratory-scale blower and was performed in a condition room with relative temperature was 31°C. The suspension are quickly evaporated with the hot air flow through the orifice of the blower nozzle resulting into a stable CNC flakes form. The CNC flakes were grind manually into powder form using porcelain mortar for 30 minutes to make sure the CNC are evenly grind. Table 1 shows the specification of CNC and table 2 shows the properties of CuO used in this experiment.

70% of CNC flakes than was dry mixing with 30% CuO in order to form CNC/CuO nanoparticles. Both CNC and CNC-CuO nanolubricant were prepared with 0.1, 0.5, and 0.9% volume fraction in 200 ml of base oil SAE 40. The initial mixing process CNC nanoparticles with SAE 40 engine oil using hotplate magnetic stirrer at low stirring rate continuously at room temperature 31°C for 1 hour. Then, the prepared solution is left in ultrasonic bath (Fisherbrand model number-FB1505) for 1 hour for each concentration to enhance the homogenous and stability of the nanolubricant. The amount of the nanoparticles essential for preparation of nanolubricants is calculated using below equation.

$$\phi = \frac{\left[\frac{W_p}{\rho_p} \right]}{\left[\frac{W_p}{\rho_p} + \frac{W_{bf}}{\rho_{bf}} \right]} \quad (1)$$

W_p denotes the weight of the nanoparticles in grams, ρ_p denotes the density of the nanoparticles in g/cm^3 , W_{bf} denotes the weight of the base fluid in grams and ρ_{bf} denotes the density of the base fluid in g/cm^3 .

2.2. Measurement of thermal conductivity and specific heat capacity

A KD2 Pro thermal property analyzer from Decagon Devices Inc., USA has been employed to measure the thermal conductivity of studied nanolubricant. This device has been widely used by researcher in thermal conductivity measurement [22, 23, 27, 39]. The device works based on transient hot wire method. The device has been calibrated by the glycerin with the measured value was 0.286 W/m K with accuracy of $\pm 0.35\%$ supplied by manufacturer. A water bath was used during the measurement to maintain a constant sample temperature. In order to set the temperature of the samples uniformly, a hot water bath temperature has been employed. The measurement was repeated several

times and the average value from five sets of reading was considered. The present thermal conductivity measurement is considered at a minimum of 15 min interval time before the next reading for each data set at different temperatures and mixture ratios. This is important to minimize the error during measurement by free convection due to the temperature variation along the sensor which is in direct contact with the liquid sample. Furthermore, in order to minimize the errors resulted from the free convection, the probe (needle) of the device must be kept vertically into the fluid during the measurements.

A differential scanning calorimeter (DSC) was used in order to determine specific heat capacity. The nanolubricants samples were placed in aluminium crucibles and heated from 30°C up to 90°C at a heating rate of 5°C min⁻¹ under a dynamic atmosphere of nitrogen. The nanolubricant samples were heated from room temperature at heating rate of 5°C- 1 up to 30°C.

2.3. Design of experiment (DOE)

An experimental design was carried out by using Minitab 18, a statistical analysing software by referring factors with various levels as shown in table 1. This experiment is designed by considering 2 continuous factor and 1 categorical non-value factor. Hence, central composite design (CCD) method is approached which is suitable to develop models with 2 continuous factors and 1 categorical factor. There are classified into three levels, low value (-1), high value (+1) and centre value (0). The continuous factors are temperature (T) and volume concentration (Ø) while the categorical factors are type of nanolubricant. It means the type of nanolubricant will be included in the development of the empirical model but it won't appear in the empirical formula. A total of 26 experiments were designed to study the influence of temperature, volume concentration and type of nanolubricants on the thermal conductivity and specific heat capacity. Design layout and experimental result are summarized in table 2.

Table 1. Factors at various level.

Factors	-1	0	+1
Temperature (continuous)	30	60	90
Concentration (continuous)	0.1	0.5	0.9
Type of nanolubricant (categorical)	CNC		CNC-CuO

3. Result and analysis

3.1. Anova analysis

The analysis of variance analysis was used to test the response model, statistically. Minitab 18 software was used to perform the variance analysis. In order to study the appropriateness and significance of the model, parameters consisting of Fisher's test (F-test), probability (P-value), and coefficient of variation (R-square) are used in the variance analysis. Tables 3 and 4 show the results of ANOVA analysis for thermal conductivity and specific heat capacity. A model having a good ability to fit the data has the highest value of R-squared (R²) and adjusted R-squared (R²-adj). R², the coefficient of determination, is the ratio of the changes described by model to the whole changes. Therefore, whatever the value of R² is closer to one, the power of the fitted model describing the response changes as a function of the independent variables is greater [40]. In general, for a model with a good fitting, R² should be at least 80% [41]. In this study, the determination factors of the model for viscosity and specific heat capacity are 88.22 and 87.97%, respectively. Hence, for both parameters, the model correlates the experimental data, accurately.

Table 2. Design of experiment with experimental result.

Std Order	Run Order	Temperature	Concentration	Type of nanolubricant	Thermal conductivity (k)	Specific heat capacity (Cp)
9	1	60	0.5	CNC	0.13441	1.76148
19	2	90	0.5	CNC-CuO	0.1415	1.65383
23	3	60	0.5	CNC-CuO	0.14365	0.72787
25	4	60	0.5	CNC-CuO	0.14365	0.72787
26	5	60	0.5	CNC-CuO	0.14365	0.72787
15	6	90	0.1	CNC-CuO	0.1438	1.47654
2	7	90	0.1	CNC	0.1305	2.77698
7	8	60	0.1	CNC	0.1338	2.99186
8	9	60	0.9	CNC	0.14217	1.34358
14	10	30	0.1	CNC-CuO	0.1486	1.00852
1	11	30	0.1	CNC	0.1361	2.19904
3	12	30	0.9	CNC	0.1461	0.76851
13	13	60	0.5	CNC	0.13441	1.76148
12	14	60	0.5	CNC	0.13441	1.76148
16	15	30	0.9	CNC-CuO	0.1324	0.3923
21	16	60	0.9	CNC-CuO	0.13259	0.9104
24	17	60	0.5	CNC-CuO	0.1338	0.72787
10	18	60	0.5	CNC	0.13441	1.76148
4	19	90	0.9	CNC	0.1368	1.11952
18	20	30	0.5	CNC-CuO	0.1428	1.11124
20	21	60	0.1	CNC-CuO	0.14365	1.69873
22	22	60	0.5	CNC-CuO	0.14365	0.72787
11	23	60	0.5	CNC	0.13441	1.76148
5	24	30	0.5	CNC	0.1381	1.13875
17	25	90	0.9	CNC-CuO	0.1338	0.72787
6	26	90	0.5	CNC	0.1268	1.66326

3.2. The impact of independent variables on response

For this current work, the impact of temperature, volume concentration and type of nanolubricant as three independent variables on the thermal properties of nanolubricant for thermal conductivity and specific heat capacity has been investigated. P-value is used to determine an effective term of the model. According to Myers and Montgomery [42] When the P-value is less than 0.05, it indicates that this is an effective factor in a response. As shown in table 3, for thermal conductivity, temperature (T), type of nanolubricant (β) and the interaction between concentration and type of nanolubricant ($\emptyset\beta$) are the effective factor in thermal conductivity of the nanolubricant, In contrast from thermal conductivity, as shown in table 4 concentration (\emptyset), type of nanolubricant (β) and 2 way interaction between concentration and type of nanolubricant ($\emptyset\beta$) are the effective parameters for specific heat capacity. According to table 3 and table 4, the interaction factor is not important, so it can be omitted from the model. In otherwords, the insignificant interaction between the independent variables has no effect on the specific heat capacity response.

3.3. Development of empirical model

Equation 2 and 3 represent the empirical relationship between thermal conductivity at CNC and CNC-CuO while equation 4 and 5 indicate the empirical model for specific heat capacity at CNC and CNC-CuO accordingly.

$$k(\text{CNC}) = 0.14002 - 0.000137T + 0.0050\emptyset - 0.000000T^2 + 0.00368\emptyset^2 + 0.000026T\emptyset \quad (2)$$

$$k(\text{CNC-CuO}) = 0.15076 - 0.000017T - 0.0208\emptyset + 0.00368\emptyset^2 + 0.000026T\emptyset \quad (3)$$

$$C_p(\text{CNC}) = 1.919 + 0.0273T - 3.087\emptyset - 0.000145T^2 + 1.338\emptyset^2 - 0.00374T\emptyset \quad (4)$$

$$C_p(\text{CNC-CuO}) = 0.633 + 0.0267T - 2.011\emptyset - 0.000145T^2 + 1.338\emptyset^2 - 0.00374T\emptyset \quad (5)$$

It is apparent from the equation 1 that temperature has a negative impact while concentration has a positive impact on thermal conductivity of CNC nanolubricant. Moreover, the the factor coefficient for \emptyset is larger than T as a fixed coefficient factor which means that changing factor \emptyset has more effect on the thermal conductivity for CNC nanolubricant than factor T. As for equation 2, both T and \emptyset factors shows negative impact on the thermal conductivity for CNC-CuO nanolubricant. Furthermore, \emptyset is more effective towards CNC-CuO nanolubricant than T as the factor coefficient for \emptyset is larger than T. According to equation 3, for specific heat capacity of CNC nanolubricant, factor T give the positive impact while factor \emptyset give negative impact on the specific heat capacity of CNC nanolubricant. Similarly, for CNC-CuO nanolubricant, factor \emptyset also give negative impact and factor T give positive impact to the specific heat capacity of CNC-CuO nanolubricant. Both equations also shows that factor \emptyset is more effective towards both nanolubricant as the factor coefficient \emptyset is larger than T.

Table 3. Anova result for thermal conductivity.

Source	DF	Adj SS	Adj MS	FValue	PValue
Model	8	0.000617	0.000077	7.63	0.000
Linear	3	0.000256	0.000085	8.44	0.001
T	1	0.000080	0.000080	7.88	0.012
\emptyset	1	0.000013	0.000013	1.31	0.269
β	1	0.000163	0.000163	16.14	0.001
Square	2	0.000002	0.000001	0.10	0.909
T ²	1	0.000000	0.000000	0.01	0.905
\emptyset^2	1	0.000002	0.000002	0.19	0.669
2-Way Interaction	3	0.000359	0.000120	11.84	0.000
TC	1	0.000001	0.000001	0.08	0.784
T β	1	0.000039	0.000039	3.81	0.068
$\emptyset\beta$	1	0.000320	0.000320	31.63	0.000
Error	17	0.000172	0.000010		
Lack-of-Fit	9	0.000094	0.000010	1.08	0.463
Pure Error	8	0.000078	0.000010		
Total	25	0.000788			

Table 4. Anova result for specific heat capacity.

Source	DF	Adj SS	Adj MS	FValue	PValue
Model	8	9.4440	1.18050	16.43	0.000
Linear	3	8.6024	2.86746	39.90	0.000
T	1	0.6532	0.65317	9.09	0.008
\emptyset	1	3.9554	3.95542	55.04	0.000
β	1	3.9938	3.99379	55.58	0.000
Square	2	0.2685	0.13426	1.87	0.001
T^2	1	0.0938	0.09377	1.30	0.269
\emptyset^2	1	0.2532	0.25317	3.52	0.078
2-Way Interaction	3	0.5731	0.19104	2.66	0.081
TC	1	0.0161	0.01614	0.22	0.642
$T\beta$	1	0.0010	0.00096	0.01	0.909
$\emptyset\beta$	1	0.5560	0.55601	7.74	0.013
Error	17	1.2216	0.07186		
Lack-of-Fit	9	1.2216	0.13573	2.01	0.364
Pure Error	8	0.0000	0.00000		
Total	8	9.4440	1.18050	16.43	0.000

Table 5. Model summary of thermal conductivity.

S	R-sq	R-sq(adj)	R-sq(pred)
0.0031786	88.22%	87.97%	70.07%

Table 6. Model summary of specific heat capacity.

S	R-sq	R-sq(adj)	R-sq(pred)
0.268065	88.55%	83.16%	70.67%

Figure 1 shows the comparison of the experimental results of thermal conductivity with the data predicted by the model. As it can be seen, there is a good agreement between the model and experimental data. Therefore, there is a good correlation between the experimental results and predicted results using the statistical method. Similarly, the experimental data of specific heat capacity is compared to the data predicted by the model. As seen in figure 2, the model predicts the experimental data well. The ineffective terms which have been removed from the model are predicted well, and eliminating them has no adverse effect on the accuracy of the model.

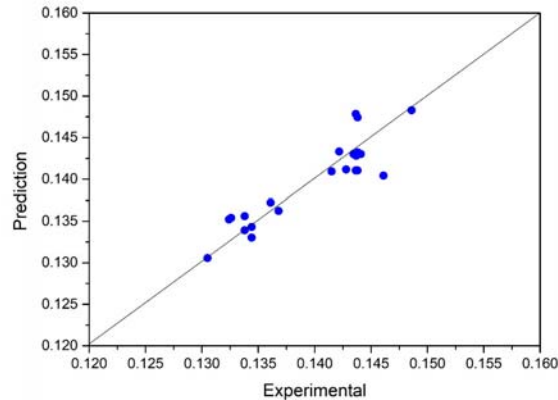


Figure 1. Comparison of experimental and predicted model for thermal conductivity.

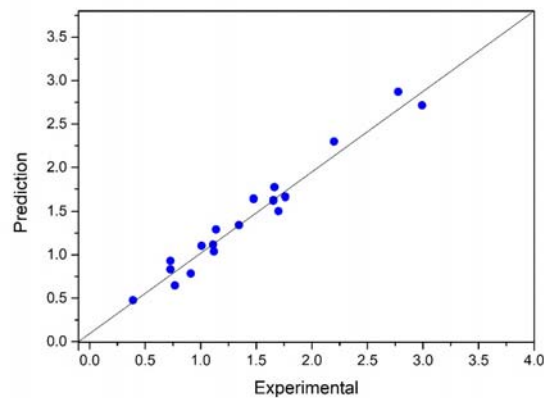


Figure 2. Comparison of experimental and predicted model of specific heat capacity.

Figure 3 and 5 show the effects of temperature and concentration on thermal conductivity of the nanolubricant in three-dimensional graphs and contour, respectively. Due to the significant effects of linear model and linear concentration the curves and contour can be expected. This indicates concentration has a significant impact on thermal conductivity of the nanolubricant. As can be seen, by increasing concentration, thermal conductivity increases. By contrast, by increasing temperature at a constant concentration, thermal conductivity increases initially and then remains almost constant in the process. Therefore, further increase in temperature has no significant effect on this property. Also there are too many theories about reasons of thermal conductivity increase by temperature increment. Brownian motions are one of the main reasons of presented increase. Adding more nanoparticles to the base fluid will cause increase in thermal conductivity of nanofluid because of higher thermal conductivity of nanoparticles in comparison to the base fluid [41]. Figs. 4 and 6 show the effects of temperature and concentration on specific heat capacity in three-dimensional graphs and contour, respectively. Similarly as thermal conductivity, as it can be seen, at a constant concentration, specific heat capacity increase significantly by increasing temperature. However, at a constant temperature, specific heat capacity decreases by increasing concentration. Since both linear and quadratic effects of temperature and concentration are the effective factors in thermal conductivity, it has a curvature form. Hence, specific heat capacity has an optimized value.

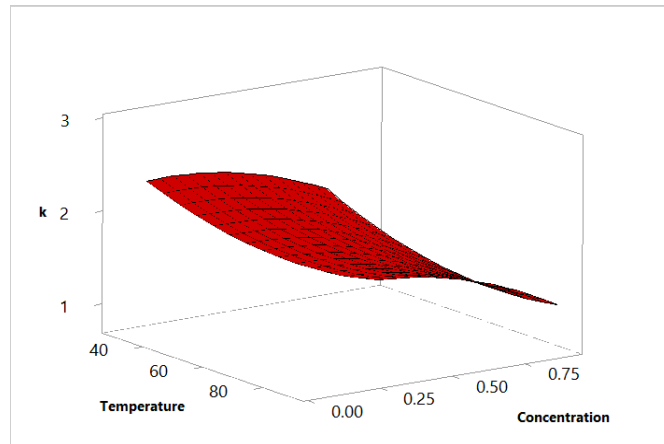


Figure 3. Surface plot for thermal conductivity.

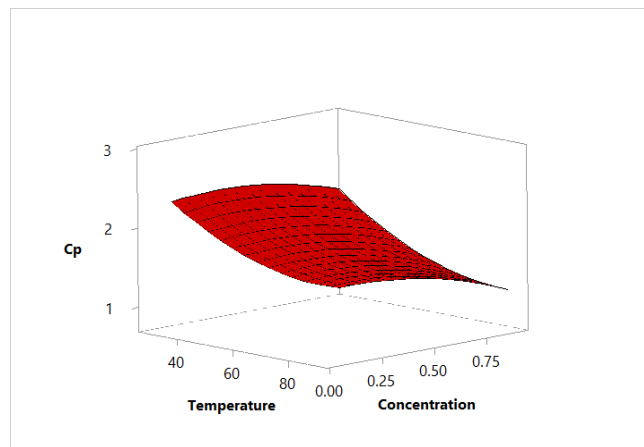


Figure 4. Surface plot for specific heat capacity.

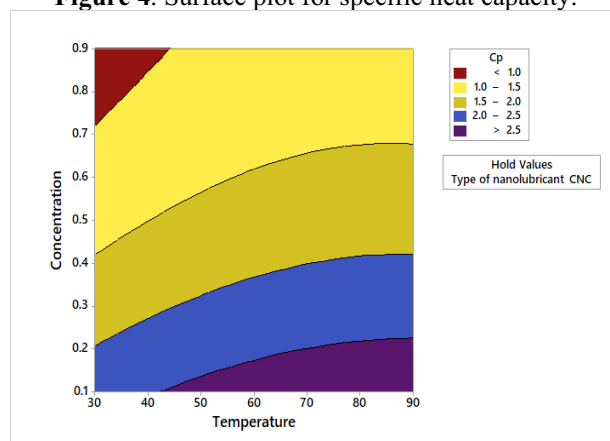


Figure 5. Contour plot for thermal conductivity.

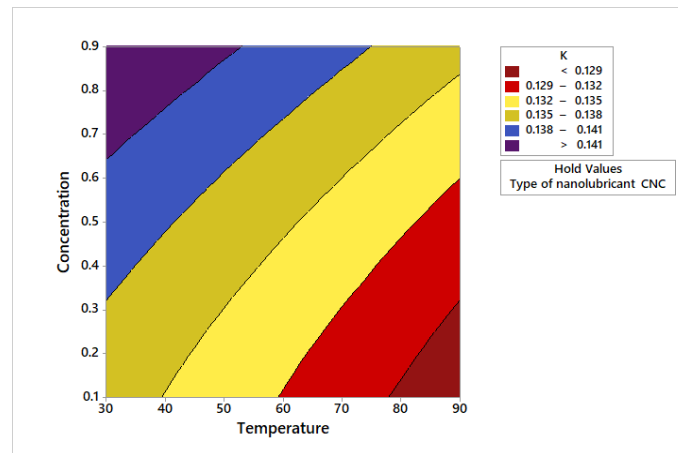


Figure 6. Contour plot for specific heat capacity.

3.4. Multi-objective optimization

The main advantage of using response surface methodology (RSM) is that the response can be optimized by controlling the input parameters [43]. According to the previous section, it is shown that by increasing concentration, thermal conductivity increases and increasing temperature at a constant concentration, thermal conductivity increases initially and then remains almost constant in the processes. Similarly as thermal conductivity, at a constant concentration, specific heat capacity increase significantly by increasing temperature. However, at a constant temperature, specific heat capacity decreases by increasing concentration. The goal of this research is to achieve the highest thermal conductivity and highest specific heat capacity.

Table 7. Optimal condition values.

Optimum results	Temperature	Concentration	Type of nanolubricant	Experimental value	Predicted value	ARE%
k	81.512	0.1	CNC/CuO	0.14422	0.1463	1.4422%
Cp	81.512	0.1	CNC/CuO	1.52589	1.6311	6.8949%

Figure 7 exhibits the optimization plot for both thermal conductivity and specific heat capacity responses. The optimum value shown in the plot is 0.1463 for thermal conductivity and 1.6311 for specific heat capacity. The relevant parameters such as concentration, temperature and type of nanolubricant are 81.51°C, 0.1, and the categorical factor is CNC-CuO. The composite shown in the plot is 0.6531.

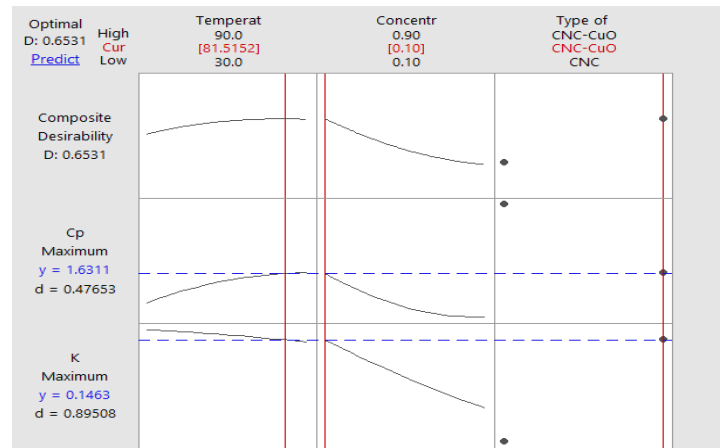


Figure 7. Optimization plot.

In order to validate the predicted optimization results, an experiment to measure thermal conductivity and specific heat capacity was run using the optimized factors. In the optimal conditions, fluid properties were measured and compared with the data predicted by the model. The results as shown in the table 7 indicate that the model can predict the optimal experimental conditions well.

4. Conclusion

The following conclusion can be derived based on the results obtained:

- There is a good correlation between the experimental results and predicted results for thermal conductivity and specific heat capacity using the statistical method as R^2 result for both responses are above 80%
- The predicted optimized value shown in the optimization plot is 0.1463 for thermal conductivity and 1.6311 for specific heat capacity. The relevant parameters such as concentration, temperature and type of nanolubricant are 81.51°C, 0.1, and the categorical factor is CNC-CuO. The composite shown in the plot is 0.6531. The validation result with experimental as shown in indicate that the model can predict the optimal experimental conditions well.

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