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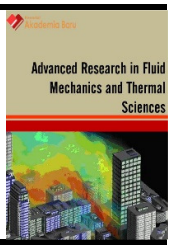

Evaluation of Specific Heat Capacity and Density for Cellulose Nanocrystal-based Nanofluid

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ABSTRACT

In this present study, the experimental results for density and specific heat capacity for CNC based nanofluid are reported. The nanofluid used in this investigation was prepared by adopting two-step preparation method. Experiment results from the work carried out testifies that the density has proportional relationship to volume concentration and an inverse relationship with temperature. Meanwhile, the specific heat capacity displays a proportional relationship with temperature and has an inverse trend to volume concentration. An empirical mathematical model has been developed for relative density and relative specific heat capacity through Response Surface Method (RSM) by using Central Composite Design (CCD). Statistical analytical software (Minitab 17) was used to compute the theoretical model and ANOVA table. The significances of the empirical mathematical model were validated through ANOVA table by considering R2 (predicted), the difference between R2-R2 (adjusted), PRESS value and p-value in lack of fit. The obtained empirical model for relative specific density and relative specific heat capacity observed to be in good term with the experimental results with a maximum error of 0.26% and 0.72% respectively. Thus, the proposed mathematical model is suitable in predicting the specific heat capacity and density for CNC dispersed in the ethylene-glycol mixture.

Keywords:
Nanocellulose, Nanofluid, Density, Specific heat capacity, Ethylene glycol-water, RSM

1. Introduction

The research on nanotechnology has been advancing intensely all over the world particularly in the area of nanofluid. Nanofluid is a branch of nanotechnology which involves the study of the nano-scale material dispersion into base fluid. Usually, this nanofluid is prepared by colloidal suspension of nanomaterial (ranged between 1-100nm) into any liquid (known as base fluid). The uniqueness of nanofluid in enhancing the thermophysical property compared to conventional fluid embarked

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investigators for implementation in heat transfer applications. As such, there has been a race to invent nanofluid with highest thermal conductivity and specific heat capacity. Besides, nanofluid also has captivated characteristics such as slowing sedimentation process, higher stability, resolve corrosion and abrasion issues on flowing channels compared to a fluid with micro-sized particles.

Thermophysical property measurement of nanofluid is composed of thermal conductivity, dynamic viscosity, density and specific heat capacity. At present, most of the researchers keen to perform experimental measurement for thermal conductivity and viscosity. Thus, most of the publication to date, reports findings specifically in this area [1]. For instance, Tawfik [2] published a review of experimental studies on thermal conductivity and Azmi, Sharma [3] publications which focus on review comprises of both thermal conductivity and viscosity, signifies the amount of work in those two properties. From the conducted various investigations, it is testified that the enhancement of dynamic viscosity in nanofluid influences the rheological behaviour of nanofluid [4]. Conversely, thermal conductivity enhancement is related to heat removal rate which is a primary concern for heat exchanger applications [5,6]. However, the high thermal conductivity of nanomaterial is believed not to be the only reason for thermal characteristic enhancement in nanofluids as highlighted by Hong, Yang [7].

Specific heat capacity is another vital thermophysical property for thermal performance evaluation [8, 9]. Ironically, there are limited experimental evaluations been carried out to determine the specific heat capacity. The data available at present are mostly computed by using the theoretical model developed by Pak and Cho [10]. However, according to Vajjha and Das [11], a theoretical model is not always suitable to precisely determine the specific heat capacity. Hence, the experimental evaluation is required to provide accurate specific heat capacity value, thermal characteristics and performance of nanofluid. As such, Zhou and Ni [12] used the differential scanning calorimetry (DSC) to determine the Al2O3 water-based nanofluid specific heat capacity and reported an inverse relation with volume concentration. In addition, O'Hanley, Buongiorno [13] used heat flux type DSC to evaluate the specific heat capacity for CuO, SiO, and Al2O3 based nanofluid at a temperature up to 55oC and at a maximum volume concentration of 0.1% and observed comparable findings. The similar outcome was also reported by Le-Ping, Bu-Xuan [14] using CuO–ethylene glycol based nanofluid.

Besides, another overlooked thermophysical property “density”, is an essential property for thermal performance evaluation. It requires the same consideration as dynamic viscosity since it influences the rheological behaviour, fluid dynamic and heat transfer performance in nanofluid [15]. Also, density affects the Reynold number, Nusselt number, pressure drop and friction factor in flowing channels [16,17]. Frequently, the density value is determined from the theoretical mathematical model proposed by Pak and Cho [10] which was developed from experimental results using TiO2 and Al2O3 nanoparticle for volume concentration of 4.5% at 25oC. As such, the predicted density value for another nanomaterial by using this numerical model would not provide an accurate result. Pastoriza-Gallego, Casanova [18] have investigated the density of Al2O3 nanoparticle dispersed in water by using various weight concentration from 0.5 to 7% and reported highest weight concentration has the highest density value. Whereas, Tomar and Tripathi [19] stated that the density has an inverse relationship with temperature from their for CuO-water based nanofluid experiment results. From the conducted literature study, it reveals that limited experimental investigations are available for density and specific heat capacity. Thus, the need for experimental studies on nanofluid density and specific heat capacity is crucial to obtain a reliable data.

Response Surface Methodology (RSM) is the preferable soft computing method for empirical model determination. This is because the influence of independence factors can be determined with high accuracy and with least number of experiments. At the same time, the interaction among the

factors is not neglected during empirical model development. Thus, in this research, the empirical model for relative density and relative specific heat capacity is developed by using Minitab 17 software through RSM method. The developed empirical model are validated statistically by using outcomes from Analysis of Variance (ANOVA) table.

The present paper is a continuation work by Ramachandran, Kadirgama [20] (author) who determined the experimental and numerical model for effective thermal conductivity and relative dynamic viscosity for Cellulose Nanocrystal (CNC) dispersed in ethylene glycol-water mixture. The stability of the nanofluid has already been proved and discussed by the author in his previously published paper. However, a brief discussion is included for supporting purposes. This research paper will be primarily focusing on the experimental determination of density and specific heat capacity. Also, the empirical model developed for relative density and relative specific heat capacity is reported. The theoretical models are produced by using statistical analysis software (Minitab 17) through RSM and validated from ANOVA table.

2. Methodology

2.1 Nanofluid Preparation and Stability Analysis

Nanofluid used in this experiment is CNC (nanomaterial) dispersed in ethylene glycol-distilled water mixture (base fluid) at a volume ratio of 40:60. This CNC is procured from Blue Goose Biorefineries Inc which has 8.0% of weight/weight suspension. Figure 1 depicts TEM image of CNC at 500nm times magnification scale. The CNC has average crystal diameter of 9-14nm, average crystal length of 100-150nm, hydrodynamic diameter of 150nm and crystallinity index of 80%. The nanofluid for density and specific heat capacity experimental measurement is prepared by adopting two-step method. Initially, the CNC in weight concentration converted to volume concentration by using Equation (1). Then, the required nanofluid concentration is prepared by dilution method through the addition of ethylene glycol-water mixture by using Equation (2). The nanofluid volume concentration prepared and examined this experiment is 0.1, 0.5 and 0.9% volume concentration. The prepared nanofluid is subjected to uniform high shear using magnetic stirrer for 30 minutes. Then, the nanofluid solution is immersed in an ultrasonic bath (Fisher brand model number-FB1505) for 2 hours to produce a homogenous solution with high stability and without agglomeration.

$$\phi = \frac{\omega \rho_{bf}}{\left(1-\frac{\omega}{100}\right)\rho_p + \frac{\omega}{100}\rho_{bf}} \tag{1}$$

$$\Delta V = (V_2 - V_1) = V_1 \left(\frac{\phi_1}{\phi_2} - 1\right) \tag{2}$$

Nanofluid stability for this research is performed through qualitative (sedimentation observation) and quantitative method (drop in absorbance evaluation). The details of this analysis and findings can be referred to our previously published paper [1]. However, a brief discussion is deliberated for completion purpose. In sedimentation observation, visual examination of any settlement at the test tube bottommost was observed for one month. From the observation, it is noticed that there is no buildup or settlement of the CNC at the bottom of test tube. This also verifies that there is no agglomeration of this particles to form microparticles. Also, there is no significant change in the appearance of the nanofluids. These have evidently shown that the prepared nanofluid has excellent stability characteristics. Thus, the preparation technique is suitable and capable of producing even CNC-based nanofluid. The similar and supporting finding was also observed through the drop in

evaluation method. Readers are suggested to refer to our previously published paper for deeper understandings.

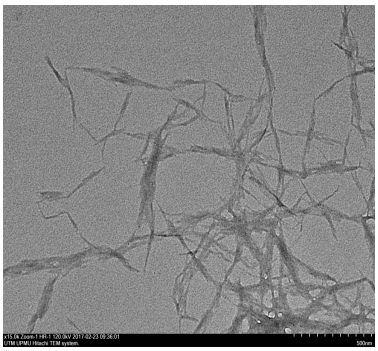

Fig. 1. TEM image of CNC under x500nm magnification scale

2.2 Density Measurement

The density value for nanofluid in this study is measured by weighing samples in a standard beaker on a high precision electronic balance [8]. A sample volume of 100ml, 200ml and 300ml is used for each volume concentration to calculate density by using formula as shown in Equation (3). The beaker used in this experiment is insulated with aluminum foil to prevent heat loss during heating and to maintain temperature. The magnetic stirring hotplate is used to heat up the sample (nanofluid) and to stir for uniform heating of the sample. Thermocouple sensor is fitted onto the magnetic stirrer to ensure and monitor the temperature. The setup for the density measurement is as depicted in Figure 2.

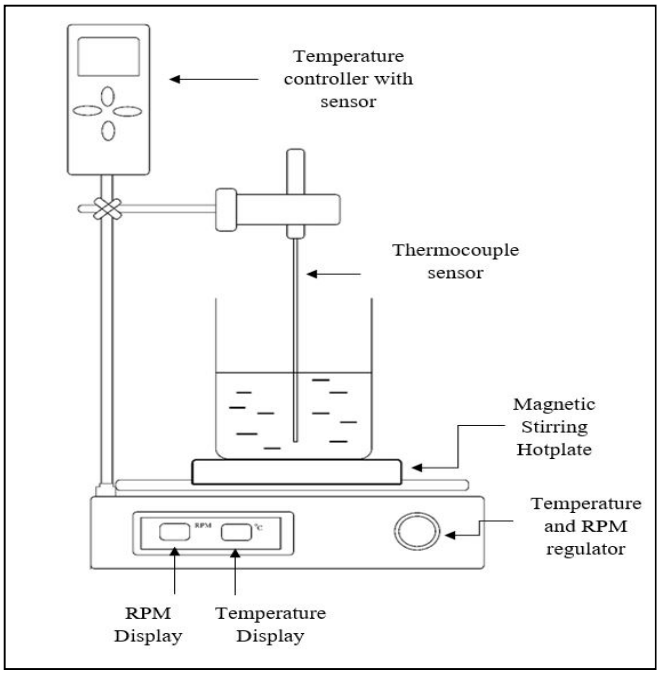

Fig. 2. Density measurement setup by using a magnetic stirring hotplate

The measurement procedure is further validated by measuring the density of the base fluid (ethylene glycol-water mixture alone) and compared with ASHRAE datasheet [21]. The highest obtained error is 1.257% which indicates the procedure capable of measuring density for the nanofluid precisely. The measurement procedure is repeated ten times and the obtained data are averaged for each sample volume (100, 200, 300ml) and nanofluid volume concentration (0.1%, 0.5%, 0.9%).

$$\rho_{nf} = \frac{W_{Total} - W_{Empty\ Beaker}}{V_{nf}} \quad (3)$$

2.3 Specific Heat Capacity Measurement

Specific heat capacity for the nanofluid in this research is determined experimentally by using Perkin Elmer (STA-8000) thermal analyzer. The simultaneous thermal analyzer (STA) is used to obtain the endothermic peak's property under various temperatures. This STA equipment has weight accuracy of 0.0001mg and temperature accuracy of 0.1°C. Also, this equipment capable of providing accurate real-time thermal analysis data as a function of weight change [22]. The samples fusion heat is measured at a heating rate of 10°C/minute from 30°C to 90°C. The lower scanning rate is chosen to eliminate the occurrence of exothermic or endothermic during oxidation [23]. Benzophenone and caffeine were used for temperature calibration meanwhile heat flow calibration is performed by using indium. The specific heat capacity was then measured for nanofluid at different volume concentration and operating temperature.

2.4 Design of Experiment

Design of experiment (DOE) for the present work is tabulated by using Minitab 17 statistical analytical tool by considering input variables as listed in Table 1. The variables are temperature, A (30-90°C) and volume concentration, B (less than 1%). Meanwhile, the factorial input variable level is divided into three; high value (+1), center value (0) and low value (-1).

Table 1
Factors at the various level used in this experiment

Factors	Level		
	(-1) Low	0 (Center point)	(+1) High
A - Temperature (oC)	30	60	90
B - Volume Concentration (ϕ)	0.1	0.5	0.9

The DOE combination and the findings obtained are shown in Table 2. For RSM, the Central Composite Design (CCD) with temperature and volume concentration as a continuous factor is used to design the experiment order. A total of 13 experiments was carried out to investigate the effect of temperature and volume concentration on relative density and relative specific heat capacity.

Table 2
Design of experiment and experimental result for relative density and relative specific heat capacity

Standard Order	Experiment Order	Factorial Input Variable		Experimental Result	
		A Temperature	B Volume Concentration	$\frac{\rho_{nf}}{\rho_{bf}}$	$\frac{C_{p_{nf}}}{C_{p_{bf}}}$
3	1	30	0.9	1.0025	1.0002
2	2	90	0.1	1.0018	1.0692
4	3	90	0.9	1.0061	1.0092
8	4	60	0.9	1.0042	1.0064
9	5	60	0.5	1.0024	1.0353
11	6	60	0.5	1.0023	1.0345
13	7	60	0.5	1.0022	1.0341
1	8	30	0.1	1.0002	1.0242
10	9	60	0.5	1.0023	1.0335
5	10	30	0.5	1.0013	1.0094
6	11	90	0.5	1.0039	1.0471
12	12	60	0.5	1.0023	1.0335
7	13	60	0.1	1.0006	1.0565

3. Results

3.1 Experimental Evaluation of Density

The experimental findings for density against temperature for varying volume concentration are depicted in Figure 3. From the scatter plot, maximum density recorded are 1061.74 kg/m^3 at a temperature of 30°C and volume concentration of 0.9%. Meanwhile, the lowest density recorded are 1034.31 kg/m^3 at a temperature of 70oC and volume concentration of 0.1%. From the plot, it is apparent that density has an inverse relation with temperature and proportional relation to volume concentration. Density measurement reported by Said, Kamyar [24] using TiO_2, SiO_2 and $TiSiO_4$ nanoparticles also showed to have a similar trend as in this research. Moreover, Mostafizur, Saidur [25] have also reported the same trend with the use of Al_2O_3 nanoparticle in their study.

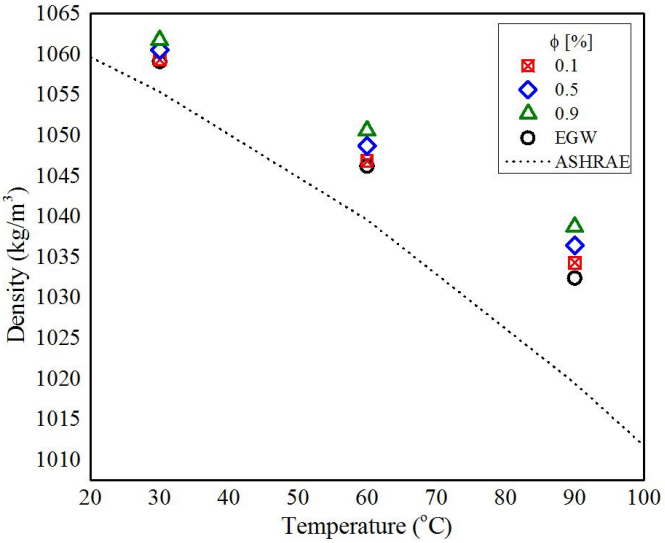

Fig. 3. Experimental results against temperature at varying volume concentration for Relative Density

The enhancement of density at higher volume concentration is alleged to be related to the colloidal suspension of nanomaterial that spontaneously fills the gaps between CNC surrounded by ethylene glycol-water mixture, which in turn, increases the overall mass of the nanofluid [8]. Meanwhile, the decline in density value as the temperature increase is believed to be attributed to thermal expansion (change of volume) in liquids at higher temperatures [26]. Also, during the heating, the kinetic energy weakens the intermolecular adhesion force which results in the decay of density value. Since density is an essential thermophysical property, characteristics such as Reynolds number and friction factor can be influenced by density. Correspondingly, the enhancement in density value would increase the pressure drop of the cooling system and eventually reduces overall performance factor [27]. As such, nanofluid with minimum density value is preferred for an efficient thermal transport [26].

3.2 Empirical Model Development of Relative Density

A theoretical model for relative density is developed by using CCD function in Minitab 17 statistical analytical tool. The empirical model is developed by considering functional variables in this experiment (temperature and volume concentration). Equation (4) shows the developed empirical model for relative density for CNC based nanofluid. This numerical model is anticipated suitable for predicting relative density at a temperature between 30-70°C and volume concentration less than 1%.

$$\frac{\rho_{nf}}{\rho_{bf}} = 1.00002 - 0.000015\,(T) + 0.001522\,(\phi) + 0.00022\,(\phi^2) + 0.000042\,(T.\phi) \quad (4)$$

The comparison between experimental and fitted data for relative density are shown in Figure 4. The fitted value for relative density is obtained from the statistical software based on the proposed model as shown in Equation (4). The estimated error is 0.05%, which indicates that the proposed model is in excellent agreement with the experimental data.

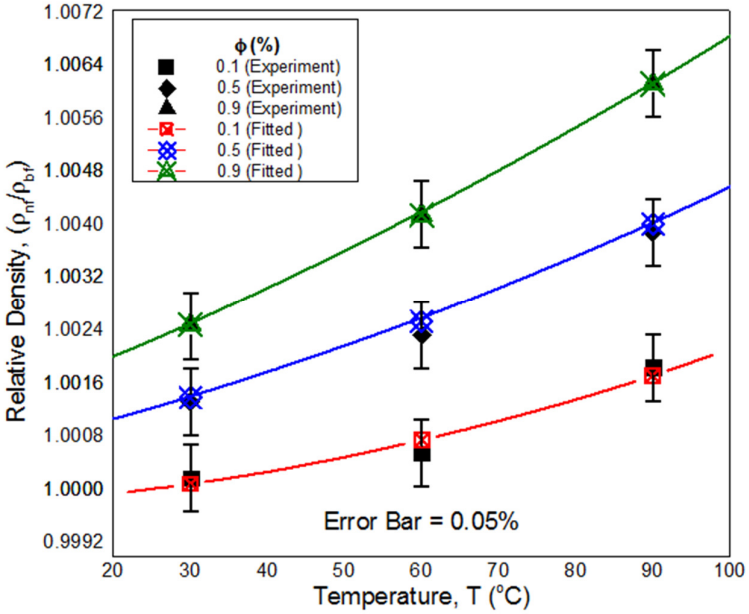

Fig. 4. Comparison of experimental data and fitted value of relative density with an error bar of 0.05%

Meanwhile, Figure 5(a) and (b) shows the residual plot with a ±0.0001 residual error for all the proposed fitted value using RSM. Whereas, Figure 5(c) depicts the normal probability plot for the residual with 45º random scatter line. Most of the fitted value seen to have a very small deviation which proves high accuracy of the quadratic model in predicting relative density for CNC. The relative density value for the proposed model is calculated manually by using Equation (4). The recorded average error was 0.13% with a maximum error of 0.26% as shown in Figure 5(d). Thus, the proposed empirical model capable of predicting relative density for CNC based nanofluid at volume concentration less than 1% accurately.

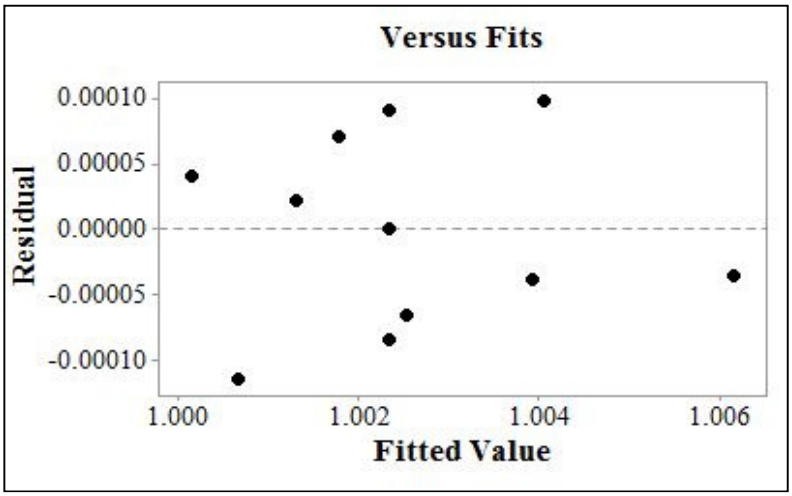

Fig. 5(a). Residual plot for proposed relative density. (Residual against fitted value)

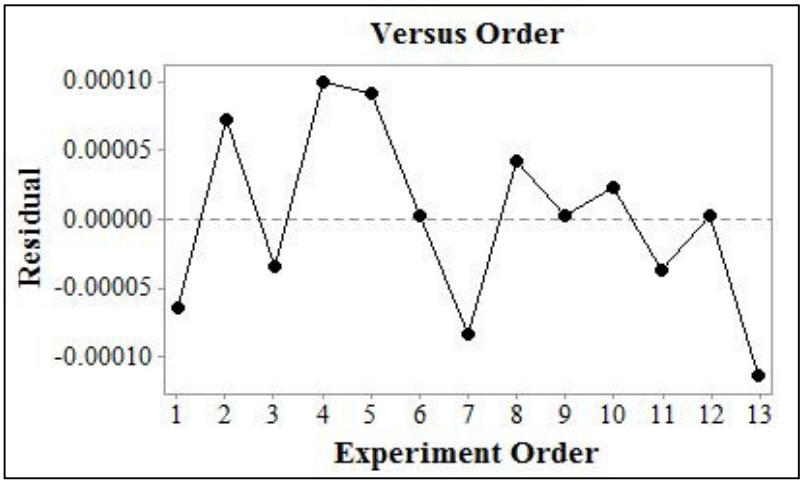

Fig. 5(b). Residual plot for proposed relative density. (Residual against experiment order)

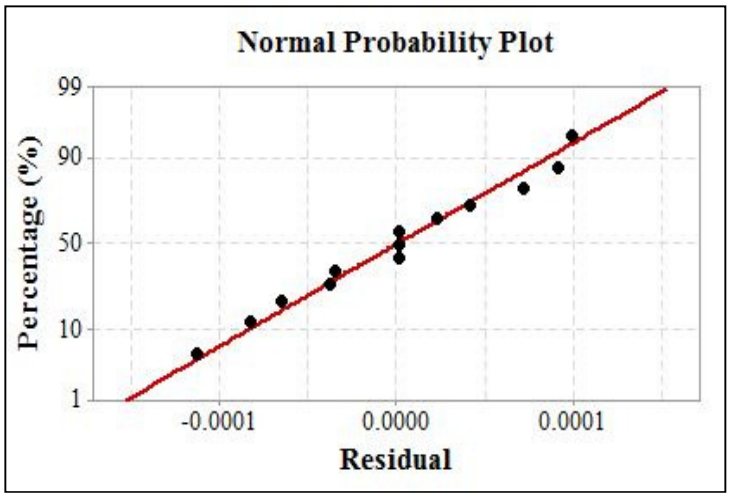

Fig. 5(c). Residual plot for proposed relative density. (Percentage against residual for relative density)

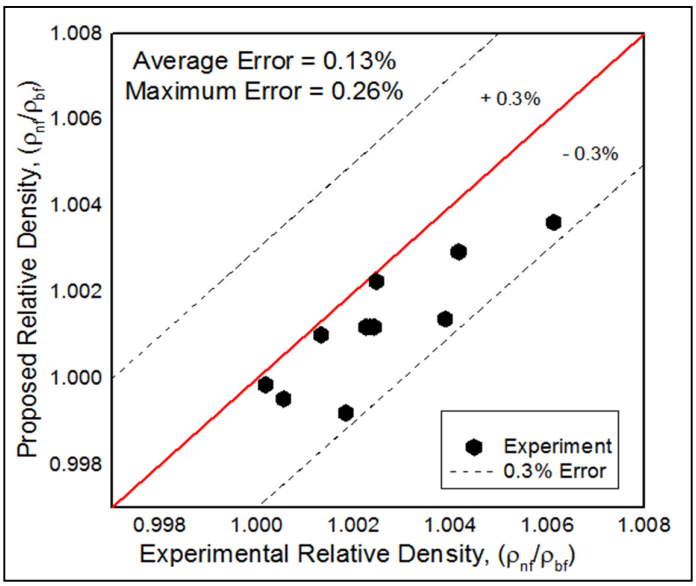

Fig. 5(d). Residual plot for proposed relative density. (Proposed relative density against experimental data in excellent)

Analysis of Variance (ANOVA) method is further approached to validate the developed empirical model for relative density, as shown in Table 3. The empirical model is substantiated by considering Predicted Residual Error of Sum Square (PRESS), R2(predicted) value, the difference in R2 - R2(adjusted) and lack of fit. According to literature, the lowest PRESS value indicates that the fitting model has smallest residual in predicting relative density using numerical model [20]. The PRESS value obtained from the statistical analysis is 0.0000004, which indicates the proposed model has a very small residual.

Meanwhile, the R2 predicted value was 98.63% which resembles high accuracy of the model in predicting the relative density value for CNC based nanofluid. Also, the variation obtained to be less than 0.2 for R2 - R2 adjusted which indicates the good fit of the proposed model with a minimal error. According to Iranmanesh, Mehrali [28], p-value obtained in lack of fit should not be more than 0.5.

The value higher than 0.5 indicates the model is not significant and does not influence the error. The computed p-value for lack of fit from ANOVA analysis for this work is 0.145. Whereas, the highest f-value is achieved for volume concentration, and followed by temperature for relative density, as shown in Table 3.

Table 3
Summary of ANOVA analysis for relative density

Source of variation	(Degree of Freedom)	(Sum of Square)	(Mean Square)	F-value	P-value
Model	5	0.000029	0.000006	1851.84	0.000
A - Temperature	1	0.000010	0.000010	1387.65	0.000
B - Volume Concentration	1	0.000017	0.000017	2316.03	0.000
AA	1	0.000000	0.000000	29.83	0.001
BB	1	0.000000	0.000000	0.46	0.521
AB	1	0.000001	0.000001	134.40	0.000
Lack of fit	3	0.000000	0.000000	3.21	0.145
Pure error	4	0.000000	0.000000		
Total	12				

Model Summary			
R-square	99.82% @ 0.9982	R-square (Predicted)	98.63% @ 0.9863
R-square (Adjusted)	99.69% @ 0.9969	PRESS	0.0000004
Mean	1.0025	Standard Deviation	0.00156
Coefficient of Variance	0.16	Variance	0.000002

The relationship between volume concentration and temperature on relative density is shown in contour plot Figure 9(a) and surface plot in Figure 9(b). Relative density observed to have proportional relation to temperature and volume concentration as shown in Figure 4. As an overall, the proposed relative density model satisfies all the validation criteria from ANOVA analysis.

3.3 Experimental Evaluation of Relative Specific Heat Capacity

The experimental specific heat capacity finding against temperature for varying volume concentration is illustrated in Figure 6. The recorded maximum specific heat capacity is 3972J/kg.°C at a temperature of 90°C and volume concentration of 0.1%. Meanwhile, the measured lowest specific heat capacity is 3522 J/kg.°C at a temperature of 30°C and volume concentration 0.9%. From the scatter plot, it is evident that the specific heat capacity has proportional relation to temperature and an inverse relation to volume concentration.

The similar finding has also been reported by Zhou and Ni [12] from their research for specific heat capacity. Volume concentration observed to have great influence than the temperature towards heat capacity enhancement [12]. Since there are limited studies been carried out in determining the specific heat capacity, is it quite difficult to explain the anomaly behind this observation. Nevertheless, it is believed that the interaction between the bulk liquid and nanomaterial alter crystallization structure in colloidal suspension contributes to the observed findings [29-35].

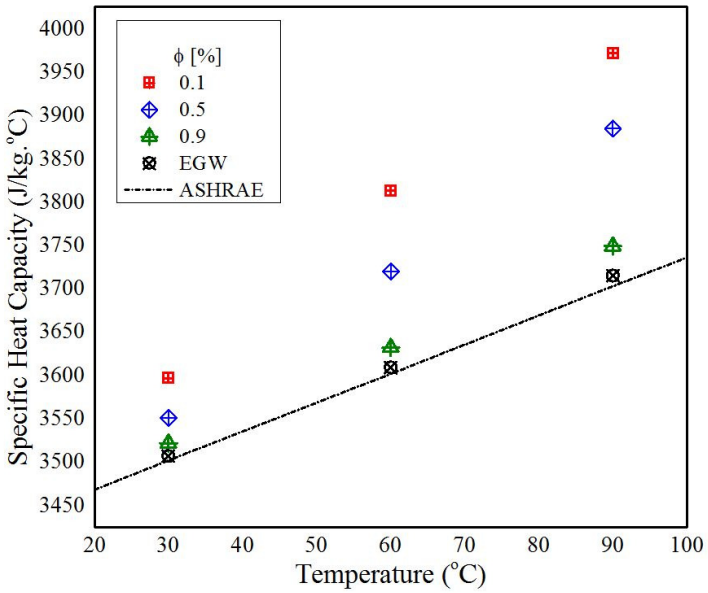

Fig. 6. Experimental results against temperature at varying volume concentration for specific heat capacity

3.4 Empirical Model Development of Relative Specific Heat Capacity

The empirical model for relative specific heat capacity is determined by using the same procedure as previous. Equation (5) is computed for relative specific heat capacity by using Minitab 17, statistical analytical tool.

$$\frac{Cp_{nf}}{Cp_{bf}} = 0.98154 + 0.001664\ (T) + \ 0.0057\ (\phi) - 0.000007\ \ (T^2) - 0.0166 - 0.000749\ (T.\phi) \qquad (5)$$

Figure 7 shows the fitted value for relative specific heat capacity obtained from the statistical analysis. The fitted value is compared with experimental value to show good the predictability of the empirical model. From the scatter plot, it can be observed that relative specific heat capacity has proportional relation with temperature and inverse relation to volume concentration.

Whereas, Figure 8(a) and Figure 8(b) shows the residual plot for fitted value and experimental value respectively. The residual value obtained to be ± 0.004 which shows the good fit of the proposed model. Random scatter 45oC line as illustrated in Figure 8(c) and Figure 8(d), further verifies the fitted value has good agreement with minimal residual. Equation (5) is used to calculate the specific heat capacity which highlights that all the predicted value is within a maximum error of 0.72%.

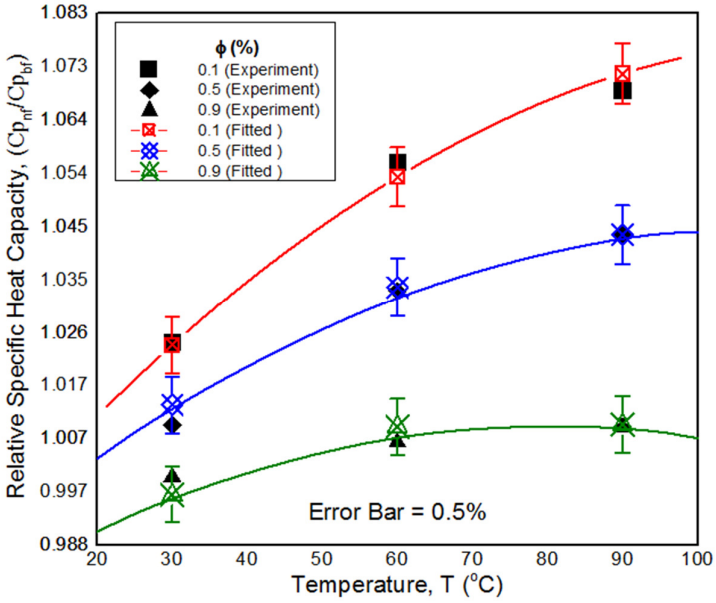

Fig. 7. Comparison of experimental data and fitted value of relative specific heat capacity with an error bar of 0.5%

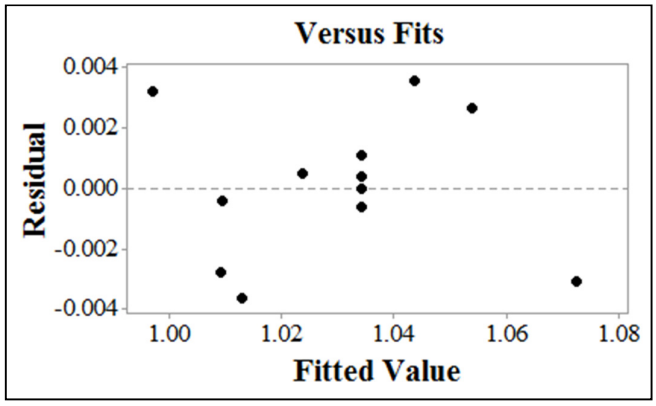

Fig. 8(a). Residual plot for relative specific heat capacity. (Residual against fitted value)

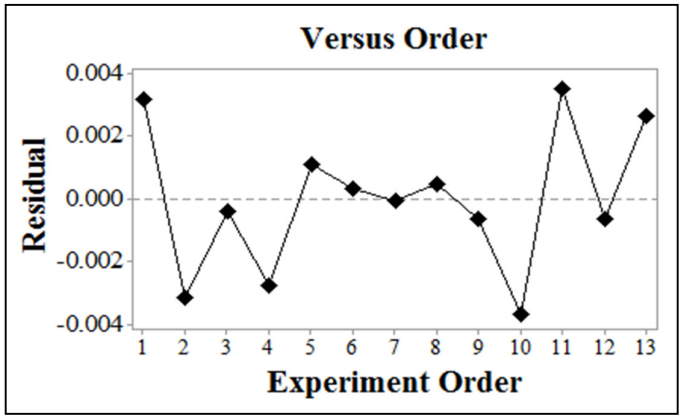

Fig. 8(b). Residual plot for relative specific heat capacity. (Residual against experiment order)

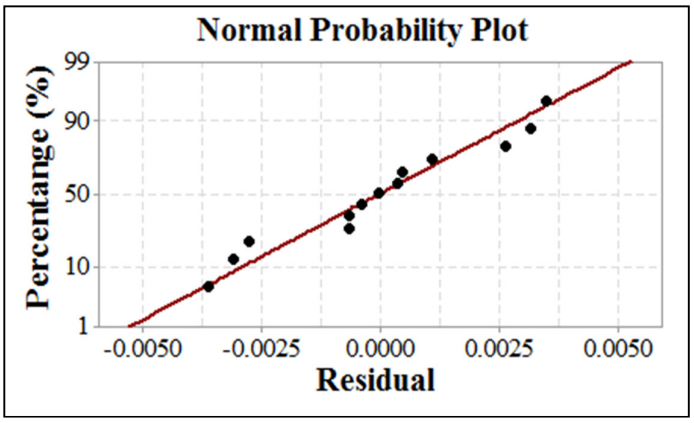

Fig. 8(c). Residual plot for relative specific heat capacity. (Percentage against residual for relative specific heat capacity)

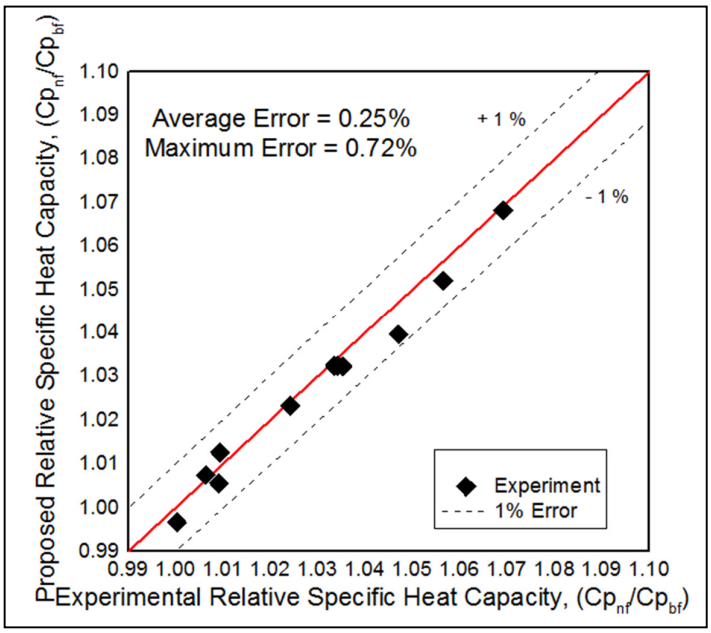

Fig. 8(d). Residual plot for relative specific heat capacity. (Predicted relative specific heat capacity against experimental data in excellent fit)

The developed empirical model is further validated by using the same approach in the previous section. The ANOVA table for relative specific heat capacity is as tabulated in Table 4. The PRESS value obtained for the mathematical model is 0.0000865 which shows the model in a satisfactory level. R2 predicted value for this empirical model is 98.53% which indicate the accuracy of the model in predicting the relative specific heat capacity. The difference between R2 and R2 (adjusted) is 0.0012 which is less than 0.2. Finally, the p-value for the lack of fit is 0.076 which agrees with the requirement (<0.5). Whereas, highest f-value obtained on volume concentration indicates that it has greater effect on relative volume concentration compared to temperature. The relationship between volume concentration and temperature on relative specific heat capacity is as shown in contour plot Figure 9(c) and surface plot in Figure 9(d). Relative specific heat capacity observed to have a proportional relation with temperature and an inverse relation with volume concentration as shown in Figure 4.

Table 4
Summary of ANOVA analysis for relative specific heat capacity

Source of variation	(Degree of Freedom)	(Sum of Square)	(Mean Square)	F-value	P-value
Model	5	0.005884	0.001177	793.91	0.000
A - Temperature	1	0.001802	0.001802	1215.70	0.000
B - Volume Concentration	1	0.003582	0.003582	2416.72	0.000
AA	1	0.000093	0.000093	62.63	0.000
BB	1	0.000019	0.000019	12.66	0.009
AB	1	0.000332	0.000332	223.96	0.000
Lack of fit	3	0.000008	0.000003	5.05	0.076
Pure error	4	0.000002	0.000001		
Total	12				

Model Summary			
R-square	99.82% @ 0.9982	R-square (Predicted)	98.53% @ 0.9853
R-square (Adjusted)	99.7% @ 0.997	PRESS	0.0000865
Mean	1.0303	Standard Deviation	0.0222
Coefficient of Variance	2.15	Variance	0.00049

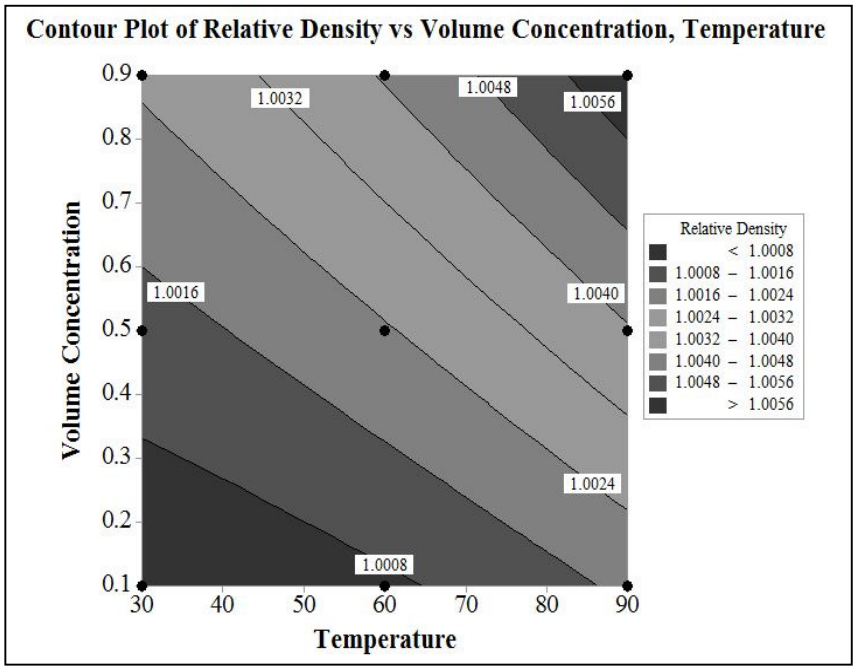

Fig. 9(a). Interaction of temperature and volume concentration on Relative density - Contour plot

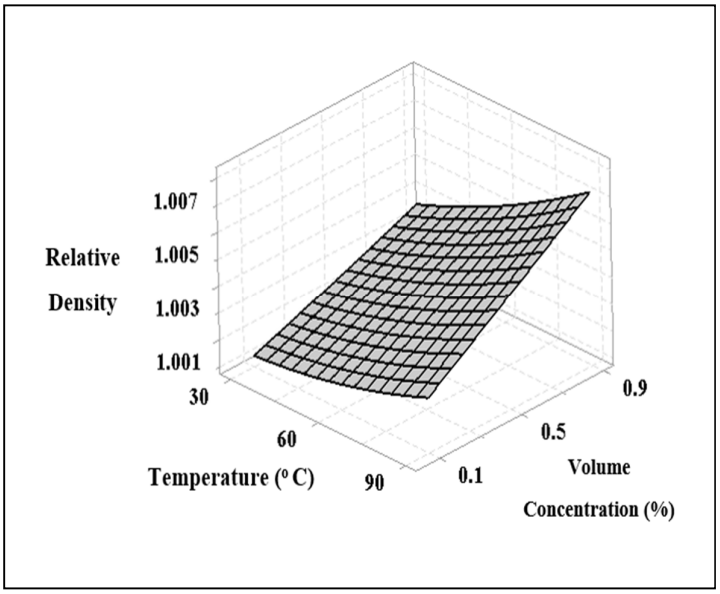

Fig. 9(b). Interaction of temperature and volume concentration on Relative density - Surface plot

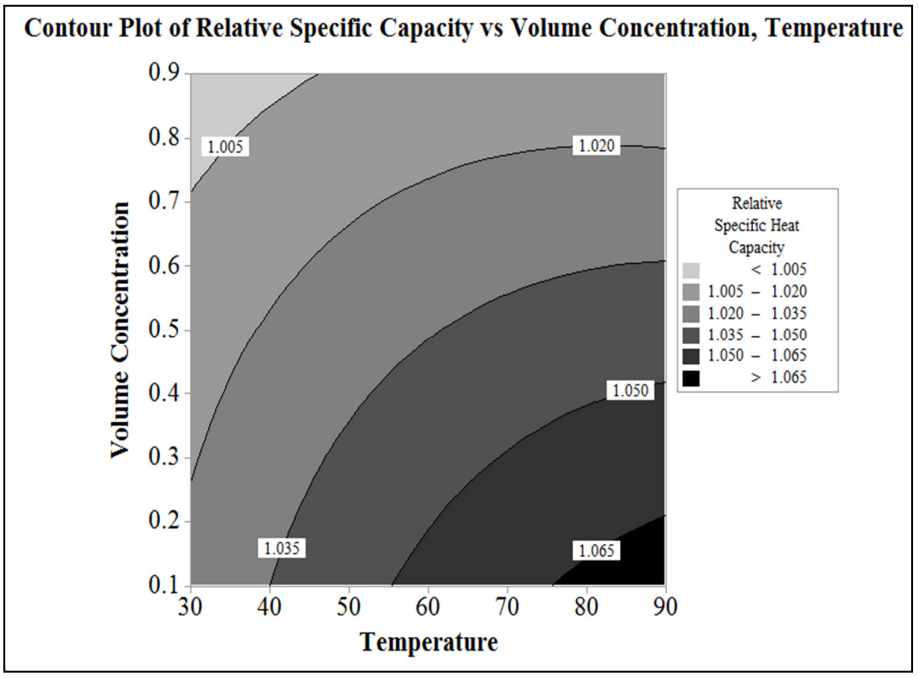

Fig. 9(c). Interaction of temperature and volume concentration on Relative specific heat capacity -Contour plot

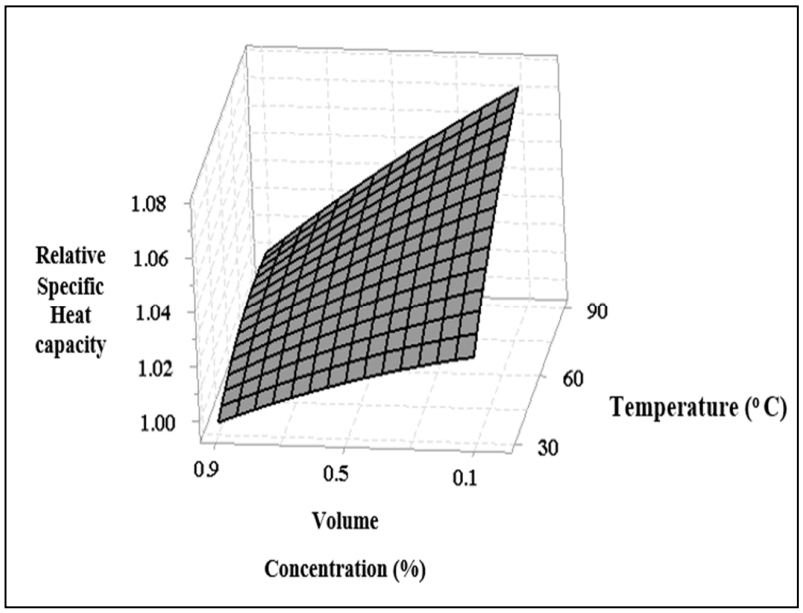

Fig. 9(d). Interaction of temperature and volume concentration on Relative specific heat capacity - Surface plot

4. Conclusions

This paper encloses the density and specific heat capacity evaluation by considering combined experimental-theoretical analysis for CNC based nanofluid. From the analysis, the density observed to have proportional relation with volume concentration and an inverse relation with temperature. Conversely, relative density testifies proportional relation with density and volume concentration. The change of density during heating is believed to be contributed by the weakening of intermolecular adhesion force which is responsible for the reduction in density value at a higher temperature. Besides that, the colloidal suspension phenomenon which spontaneously fills the gaps between CNC-ethylene glycol-water mixture increases the overall mass of the nanofluid and eventually results in higher density at higher volume concentration. The empirical mathematical model developed for relative density has high accuracy in predicting value with a maximum error of 0.26%.

Meanwhile, the specific heat capacity shows a proportional relation with temperature and an inverse relation with volume concentration. The anomaly behind the high heat capacity phenomenon is anticipated due to the interaction between the bulk liquid and nanomaterial which alter crystallization structure in colloidal suspension. The develop theoretical mathematical model capable of predicting relative specific heat capacity with a maximum error of 0.72%. Thus, the proposed empirical model for both thermo physical properties is excellent agreement with the experimental data. The proposed mathematical models for relative density and relative specific heat capacity is suitable to be used for CNC dispersed nanomaterial at volume concentration less than 1% and temperature up to 90°C.

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