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Experimental Studies on Thermo-Physical Properties of Nanocellulose–Aqueous Ethylene Glycol Nanofluids

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ABSTRACT

The aim of this experimental research study is to evaluate the thermal behaviour of crystal nanocellulose (CNC) nanofluids. Aqueous ethylene glycol (60: 40; W: EG) used as base fluid and 0.1%, 0.3% and 0.5% volume concentrations were designated here. Crystal nanocellulose nanofluids were prepared by two-step method. Thermal conductivity, viscosity, density, specific heat capacity and pH were assessed with standard measurement method and equipment. In this present work, thermal conductivity of 0.3% CNC nanofluids was increased of about 25% at 80^{0}c; viscosity decreased; 0.1% nanofluid exhibit lowest viscosity performance at 80^{0}c. In addition to this, 0.5% CNC nanofluid performed better declination of specific heat with increasing temperature up to 70^{0}C and lowest error percentage in density measurement. However, some data of the experiments were distracted.

Keywords:
Crystal Nanocellulose; Thermal Conductivity; Viscosity; Density; Specific Heat; pH

1. Introduction

Needs and luxury of human being both completely depends on energy, as energy is the lifeboat of modern world. Today's dramatic rising trend of energy consumption which is making a conflict among national and international community and with that global climate change challenge is now a crucial issue [1-2]. To bypass the energy scarcity and to secure the energy supply, a stable and sustainable source of energy is highly demandable. In this respect, technology and innovation can play a vital role by increasing global energy security [3]. Moreover, energy is the fundamental input to economic activities and therefore human success & social developments entirely depends on it. Besides, energy is the major part of industrialization as technological improvement, productivity and production enhancement, mechanizing and automation, technique and information exchange through industrial sector to the globe [4-6]. Today global energy supply depends over 80 % on fossil fuel [7]. Due to depletion and negative impact on to the environment; it is fire urgent to take necessary actions to resolve these problems. In this circumstance, alternative of fossil fuels or

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renewable energy is inevitable [8]. Renewable energies are generally eco-friendly [9] and obtained from those resources which are essentially inexhaustible and abundant [10]. Today nano technology is attracting more attention of many researchers to solve different problems in different field such as physics, chemistry, engineering, biology etc. In respect of fluid mechanics, nanofluid can be used associated with thermal energy enhancement properties [11]. Nanofluid is the fluid with nano sized solid particles which may be metallic or non-metallic solid particles can change the transport properties and heat transfer characteristics of the base fluid [12-13]. Thermal conductivity and heat transfer coefficient increases with increase in volume fraction and mass flow rate of nanofluids [14-15].

Though, nanoparticle is the most auspicious and important invention for today's science and engineering field, it also has some adverse effects such as possible toxicity of nanoparticles towards the environment which poses the potentiality of hazardous substances for plants and human health as well. In recent studies mentioned that nanotoxicity of metal oxide particles especially oxidative stress-introduced the flow that directs the inflammatory responses. Besides, researchers investigated that there should be ecological problem when plants would be captivated by nanoparticles and as a result human being may be attacked [16-20]. Therefore as an alternative, over the last few years nano cellulose attract more attention of the researchers due to their some impressive characteristics such as biodegradability, excellent mechanical properties, low density, availability of renewable resources and most significantly eco-friendly attribute as well [21-23]. Nanocellulose manufactured from wood-based cellulose and mainly in two groups such as cellulose nanofibrils (CNF) and cellulose nanocrystal (CNC). Generally CNC is formed by hydrolysing the amorphous region of cellulose with acid and then followed by centrifugation, dialysis and sonication and then finally crystal nanocellulose produced [24-25]. Theoretically crystalline region in nanocellulose is stabilized by hydrogen bonds between hydroxyl groups. Therefore, nanocellulose has higher strength and stiffness in structure. Some literatures mentioned nanocellulose has good mechanical properties as well [26-27].

The aim of this research study is to evaluate different thermal properties of crystal nanocellulose nanofluid based on aqueous ethylene glycol (60: 40; W: EG) with various volumetric concentrations. Therefore, nanofluid preparation, measurement of different thermal properties such as thermal conductivity, viscosity, density, stability, pH and specific heat capacity have been done followed by comparing the resulting data with standard data and statistical analyses.

2. Experimental Method and Materials

2.1 Nanofluid Sample Preparation

Nanofluid preparation is the first and fundamental step of any kind of nanofluid experimental studies. The crystal nanocellulose used in this study purchased from Blue Goose Biorefiners Inc. supplier. The specification of CNC has been presented in Table 1 which was provided by the supplier and density of CNC nanoparticles is around 1.5–1.6 g/cm^3 [28]. Transmission Electron Microscope (TEM) used for the microstructural characterization of CNC nanofluids. TEM images of CNC nanofluids at X50,000 and X100,000 shown in Figure 1. Images showed the average size of CNC nanoparticles is 41-137 nm. Here two step method of nanofluid preparation used. The nanocellulose was dispersed in the base fluid (60 water: 40 ethylene glycol ratio) at various volume concentrations such as 0.1 %, 0.3 % and 0.5 %. The required mass of nanocellulose corresponding to the volume concentration was calculated in Eq. (1) [29] and weighed utilizing a high precision (0.0001 g) electronic balance.

$$Vol\% = \frac{W_{np} / \rho_{np}}{W_{np} / \rho_{np} + V_{bf}} \quad (1)$$

The samples were stirred for good mixing by using magnetic stirrer for half an hour. Finally, to produce highly stable and homogenous nanofluids, the mixed samples placed in ultrasonic processor for 1 h. named Fisherbrand model number-FB1505 which is similar equipment conducted by several researchers previously in experimental studies [30-31].

Table 1
Specification parameters of CNC [32]

Parameter	Value
Crystallinity index	80%
Crystal length	100-150 nm
Crystal diameter	9-14 nm
Hydrodynamic Diameter	150nm

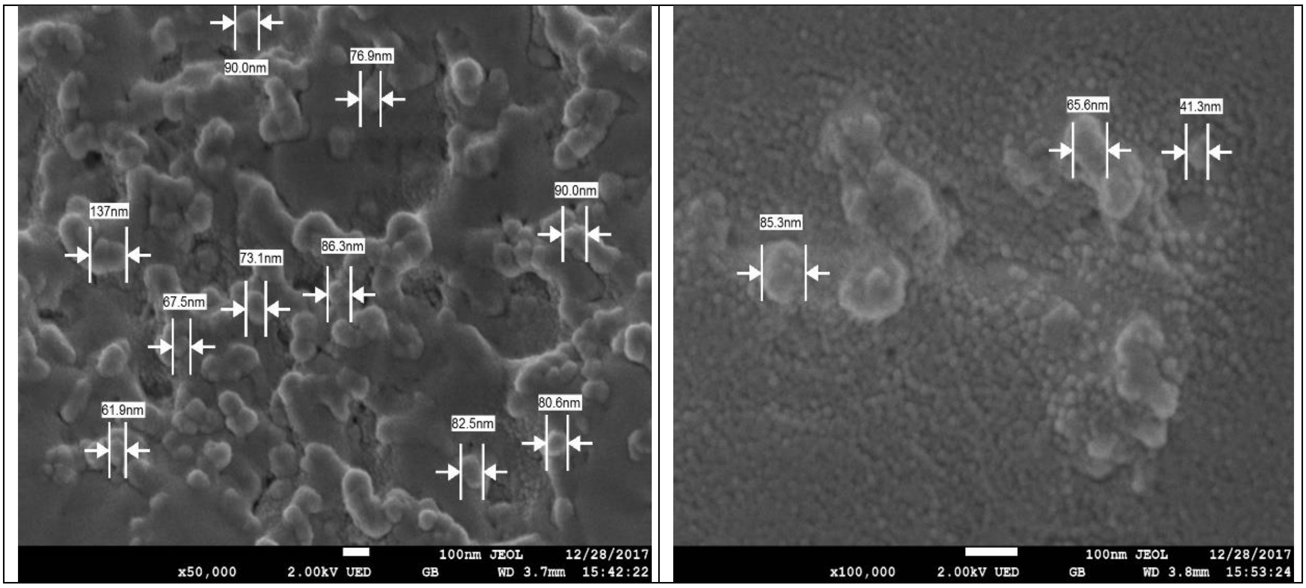

Fig. 1. TEM images of X50,000 and X100,000 magnification of CNC nanofluids

2.2 Measurement of Thermal Conductivity

Thermal conductivity of base fluid and CNC-nanofluid at various concentrations was measured by Thermal Property Analyser (Decagon Devices, Inc., USA). The transient hot-wire method was the operating principle of that device. KD2 Pro consists of a handheld controller and a sensor. Here 60 mm long with 1.3 mm diameter single-needle KS-1 sensor used in accordance with test sample to measure thermal conductivity. This sensor can measure the thermal conductivity in between 0.002-2.00 W/m-k with an accuracy of ±5 %. The experiment was performed at a temperature between 30 ^{0}C to 80 ^{0}C (controlled condition). Memmert water bath with an accuracy of 0.1 ^{0}C used to maintain the temperature by electric current supplied. The sensor is inserted into the test sample bottle and the bottle is immersed into the water bath. To prevent the bending of sensor, it must be installed in vertical with the cap of bottle. Before starting the actual sample measurement, the sensor was validated by measuring the thermal conductivity of glycerine (k= 0.282 W/m-k at 20 ^{0}C); solution provided by the manufacturer. Moreover, the thermal conductivity measurement verified by measuring the base fluid (60 % water and 40 % ethylene glycol) and compared with predetermined values of ASHREE standard (American Society of Heating, Refrigerating and Air Conditioning Engineers). The maximum deviation between base fluid and ASHREE data was 3.2 %. The deviation result determines the authenticity of the Analyser to perform the measurement. Each reading was measured with 15-minute interval time for the consistency of the data. To ignore experimental error, 20-time measurement readings and average did for every concentration of the sample. Previously many researchers used KD2 Pro Thermal Property Analyser to measure the thermal conductivity of nanofluids in experimental studies [33-36].

2.3 Measurement of Dynamic Viscosity

Dynamic viscosity of nanofluids was measured with various volume concentrations at a temperature range of 30^0 to 80 ^{0}C. A Brookfield RST, Coaxial Cylinder rheometer used in this study. RST coaxial cylinder rheometer is connected to a circulating water jacket for considering the temperature range (stipulated temperature range of 30^0 to 80^0C) along with other purposes used. Rheometer is capable for measuring viscosity range of 0.0001 to 5.4×10^6 Pa. s and temperature range of -20^0 to +180 ^{0}C. Experiment done under steady state condition. The method of measuring was Rotational measurement under controlled shear rate (CSR) and shear rate was 260 (1/s). To validate the rheometer, the viscosity of base fluid was measured and then compared the obtained values with predetermined data of ASHREE standard. The fluid amount of 15.7 ml is required to measure the viscosity and the reading is assembled in computer connected with RST rheometer. Here twenty times reading obtained and averaged to minimize the experimental error. Earlier several studies conducted viscosity measurement using the Brookfield rheometer [37-39].

2.4 Measurement of Specific Heat

Differential Scanning Calorimetry (DSC) is a highly sensitive technique can be used to measure the specific heat capacity of both solid and fluids [40-41]. In this study DSC (model DSC 8000) also used for measuring the specific heat manufactured by PerkinElmer, Inc. Specific heat capacity has been evaluated of base and CNC nano fluids at a temperature range of 25^0 to 90 ^{0}C to get the actual values of specific heat at 30^0, 50 ^{0}, 70^0 and 80 ^{0}C temperature. DSC 8000 is double-furnace thermal analyser and temperature range of -180^0 to 750 ^{0}C. Measurement solution was taken in an aluminium pan weighed on electrical balance (precision: 0.0001) and covered with a lid (aluminium) and then sealed by universal crimper press. Prior to actual sample measurement an empty pan/lid and the pan/lid filled with sapphire reference were placed in DSC to obtain the data of baseline and reference. Afterwards the sample pan/lid was mounted in DSC with an empty pan/lid as a reference. Then the temperature range was set with 10^0C/min temperature gradient following the standard DSC test method ASTM-E1269. Required time was around 6 min for every sample. This test has been done for all volume concentrations of nanofluid and base fluid. The resulting values are collected in a computer connected with DSC. Previously many researches used DSC for conducting the specific heat measurement test of nanofluids [42-43].

2.5 Measurement of Density

Density is an influential property of nanofluids which affects the pumping power, friction factor, Reynolds number and so no. In this study, to measure the density of CNC nanofluids with different volume concentrations digital density meter used alike with previous studies of various researchers

[44-46]. Here KEM (model DA-640) density meter manufactured by Kem Kyoto Electronics Co. Ltd. used. This meter has density (gm/cm^3) measuring range of 0.0000-3.000 with ±0.0001gm/cm^3 accuracy and density (gm/cm^3) repeatability is 0.00005. The ambient condition for using this meter is about temperature range of 5-35 ^{0}C and humidity 85 % RH or below. The method for measuring the density follows ASTM D4052-18 which is acknowledged as Standard test method for density, relative density and API gravity of liquids by digital density meter.

2.6 Measurement of pH

For the measurement of pH of CNC nanofluids with different volume concentration pH meter used. A considerable number of experimental studies utilized pH meter to assess the pH of nanofluids [47-48]. Here Mettler Toledo pH meter used (model five easy) to measure the pH of CNC nanofluids along with base fluid. The pH measurement range of pH meter is 0-14 with accuracy of ± 0.1 and pH resolution is 0.01. The method for evaluating the pH follows the electrometric method that is APHA 4500H^+ B. The basic principle of this electrometric method is to determine the activity of the hydrogen ions by potentiometric measurement using standard and reference electrode.

3. Results and Discussion

3.1 Stability Analysis of CNC Nanofluids

Sedimentation observation (Qualitative method) has been done to examine the stability of CNC nanofluids at various volume concentrations such as Kadirgama *et al.,* [28] conducted the qualitative method to measure the stability of nanofluids in thermal analysis of SUS 304 stainless steel using nanocellulose/ethylene glycol based study. The sedimentation observed after each fifteen day. No aggregation of CNC nanoparticles occurred at the bottom of test tube even after forty-five days of nanofluids preparation. This observation demonstrates the high stability of nanofluids. Whereas aggregation or clogging of nanoparticles degrades the thermal properties such as thermal conductivity, density, viscosity, specific heat capacity as stability of nanofluids has significant effect on thermal conductivity directly or indirectly [49].

Furthermore, UV-vis spectrum of nanofluids at 0.1%, 0.3% and 0.5% vol. concentration formed for stability measurement and showed in Figure 2. This is done by UV-Vis Spectrometer (Perkin Elmer Limited, UK). From this spectrum, it can be detected that the peak value of absorbance for all samples lies in the range of 231-233 nm of wavelength [50].

3.2 Thermal Conductivity Analysis of Nanofluids

The thermal conductivity of base fluid (60 % water and 40 % EG) and CNC nanofluids with different volume concentration (0.1 %, 0.3 % and 0.5 %) measured at four different temperature such as 30^0, 50^0, 70^0 and 80 ^{0}C. Figure 3 shows the thermal conductivity synopsis of base fluid and various volume concentrated CNC nanofluids.

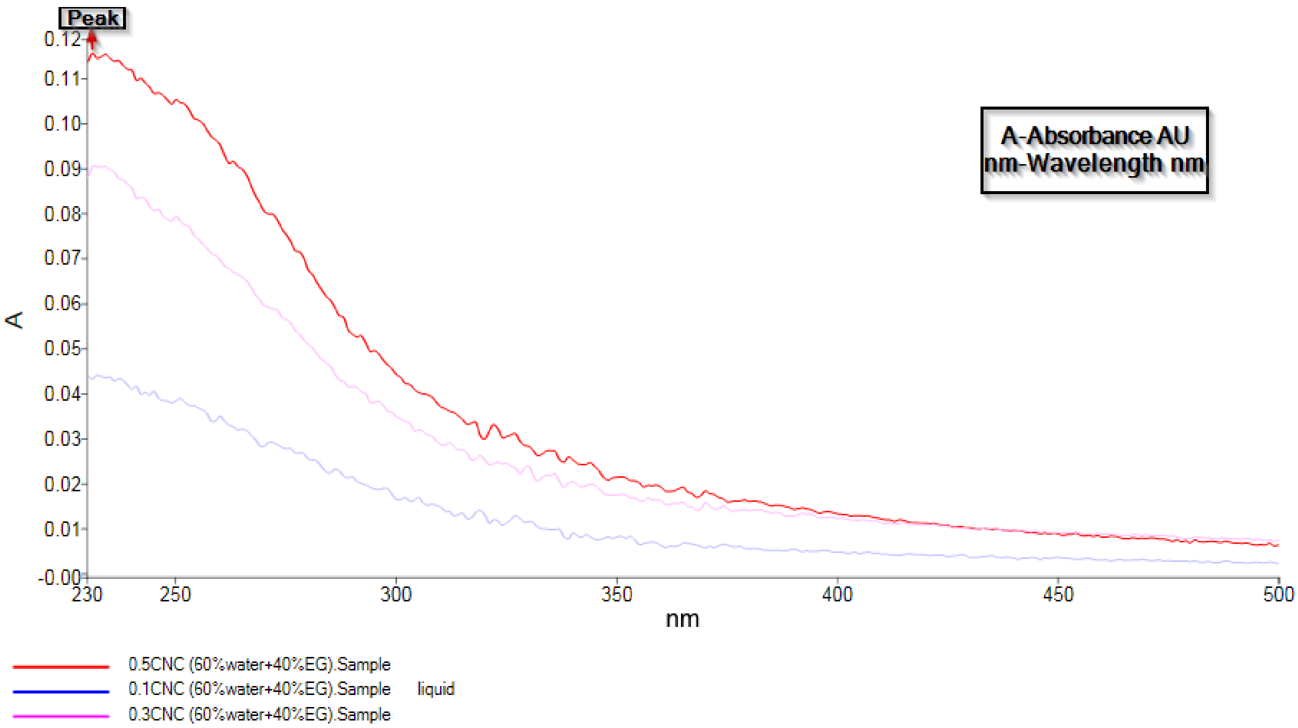

Fig. 2. UV-vis spectrum for CNC nanofluids

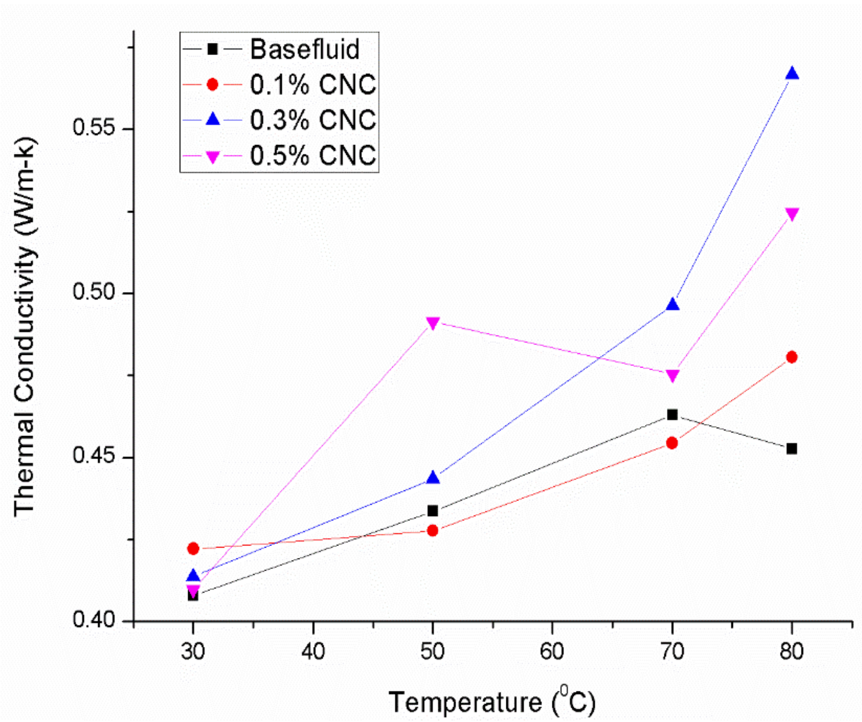

Fig. 3. Thermal conductivity of CNC nanofluids at different temperatures

The result revealed that thermal conductivity of 0.1 % CNC nanofluids increased more than other two volume fraction of nanofluids and base fluids at 30 ^{0}C. Besides, thermal conductivity surged highly for 0.5 % volume concentration CNC nanofluid at 50 ^{0}C. In addition, thermal conductivity improved in maximum at 80 ^{0}C for 0.3 % CNC nanofluid. Moreover, all volume concentration of CNC nanofluids show the increasing trend at 80 ^{0}C temperature, while base fluid shows the downward trend of thermal conductivity. However, thermal conductivity of 0.1 % CNC nanofluids is slightly lower at temperature of 50 ^{0}C and 70 ^{0}C compare with base fluid. Figure 3 also illustrates that at any temperature thermal conductivity enhancement is not linear with continuous increment of volume concentration percentage of CNC nanoparticles. This similar phenomenon also observed by Wei, Zou and Li [51]. They found thermal conductivity enhancement is not even with increased volume concentration at a temperature of 20^{0}C. Ramachandran *et al.,* [52] studied the thermal properties of water-ethylene glycol based CNC nanofluids and found addition of CNC nanoparticles can improve thermal conductivity and viscosity as well. It was also observed that the increasing temperature enhances thermal conductivity of CNC nanofluids. Thermal conductivity enhancement pattern can be discussed in accordance with the theory of Brownian motion. The collision between particles strengthened at higher temperature generating an increment of Brownian diffusion which reinforces the enhancement of thermal conductivity.

3.3 Dynamic Viscosity Analysis of CNC Nanofluids

Initially the viscosity of base fluid (60: 40 W: EG) measured and afterwards viscosity of nanofluids with different volume concentrations (0.1 %, 0.3 % and 0.5 %) determined at four distinct temperatures such as 30^{0}, 50^{0}, 70^{0} and 80 ^{0}C. Experimental viscosity results are shown in Figure 4. Here viscosity increases with increases in volume fractions and decreases when the temperature increased. The maximum value of viscosity found at 30 ^{0}C for 0.3 % volume concentration and the minimum value observed at 80 ^{0}C for 0.1 % volume concentration of CNC nanofluids.

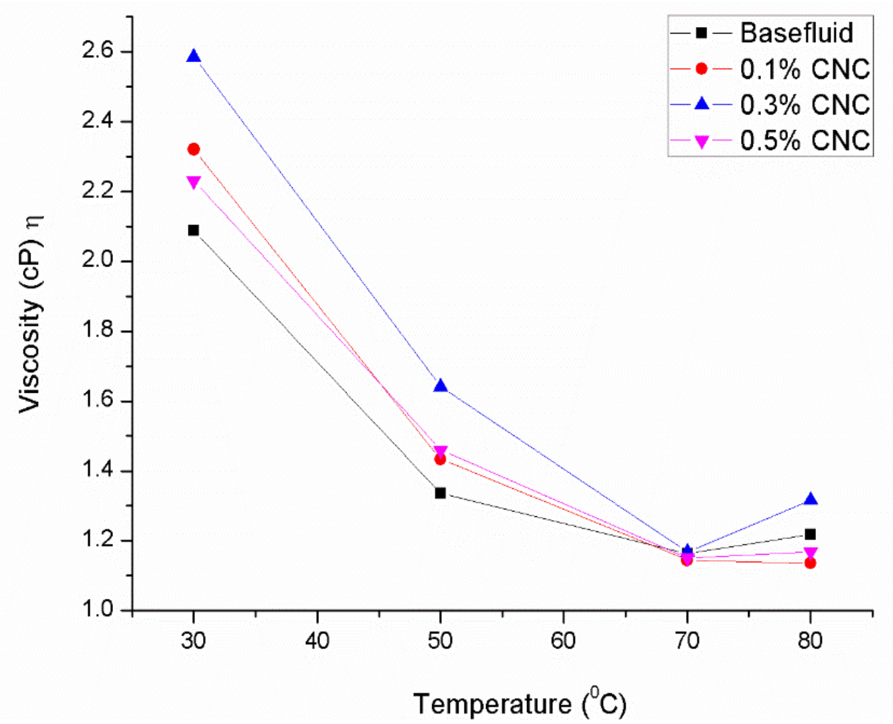

Fig. 4. Temperature dependence of viscosity of CNC nanofluids

Surprisingly viscosity of 0.5 % CNC nanofluid is lower than 0.3% CNC nanofluid at 30 ^{0}C as shown in Figure 4, although this viscosity value of CNC nanofluids is higher comparing with base fluid. The viscosity trend of 0.3 % and 0.5 % CNC nanofluids along with base fluid exhibit upward trend at 80 ^{0}C; although viscosity trend of nanofluids and base fluid is downward with raising the temperature up to 70 ^{0}C and the trend supposed to be in the same direction as well. Perhaps this viscosity phenomenon in this study supports the recommendation of Nguyen *et al.,* [53] viscosity of nanofluid study. According to Newtonian Fluid theory, shear stress (τ) and shear rate (γ) is straight and viscosity remains constant as defined Newtonian fluid [54]. But in the present work, the behaviour of all fluids does not support this Newtonian Fluid theory, as Figure 5 illustrates the disrupted trend of viscosity of 0.1 % CNC nanofluids at 30 ^{0}C. This trend is similar for all kind of nanofluids and base fluid at 30 ^{0}C, 50 ^{0}C, 70 ^{0}C and 80 ^{0}C which specify as non-Newtonian flow behaviour of all fluids.

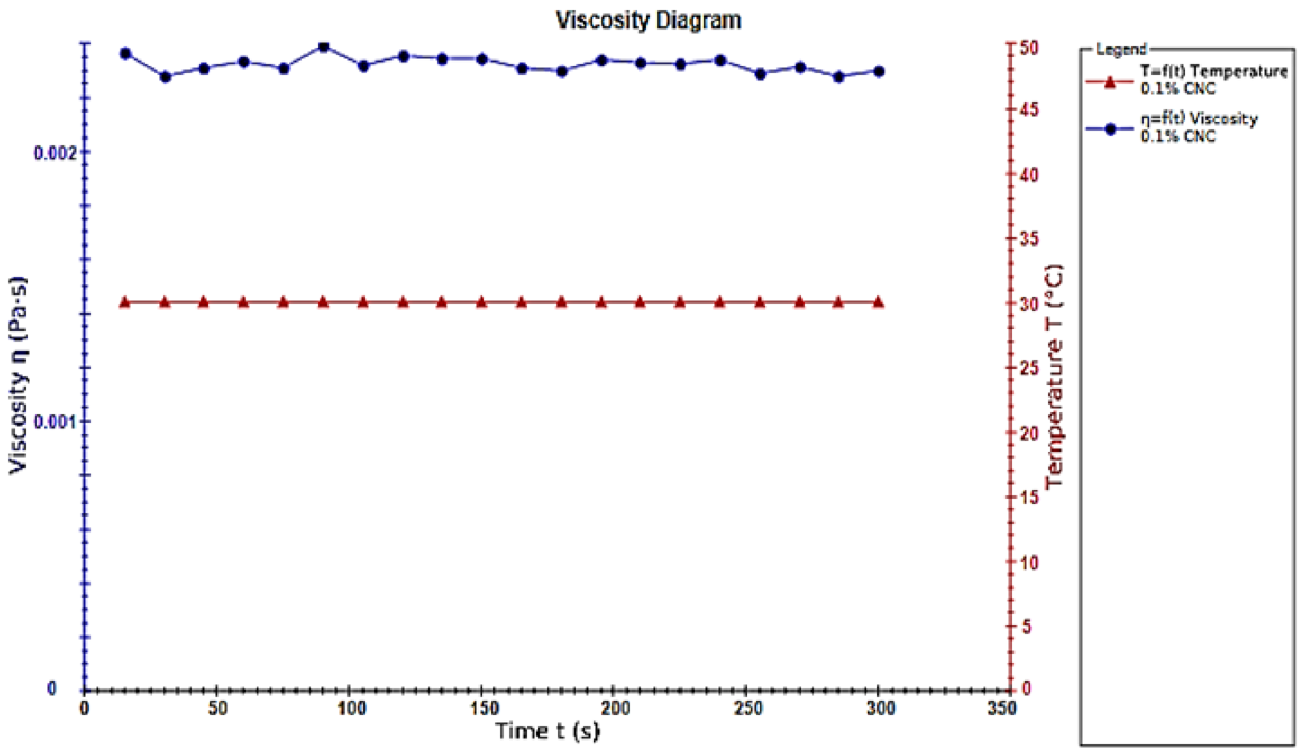

Fig. 5. Flow behaviour of 0.1% CNC nanofluids

3.4 Specific Heat Analysis of CNC Nanofluids

Specific heat capacity characteristics of CNC nanofluids were studied here by Differential Scanning Calorimetry. The results of specific capacity of base fluid and CNC nanofluids are shown in Figure 6. Insufficient numerical and experimental studies had been conducted to determine the specific heat capacity of nanofluids at different temperature and volume concentrations. O'Hanley *et al.,* [55] studied the specific heat capacity of nanofluids with various volume concentrations of different types of nanofluids (water-based alumina, silica, copper-oxide). They also used different size (diameter) of nanoparticles. They revealed specific heat capacity decreased due to increase of volume fractions of nanofluids. Singh *et al.,* [56] concluded the specific heat decreases with increase of nanoparticles concentrations at room temperature and specific heat values is minimum at highest temperature for all nanofluids. Sekhar and Sharma [57] found that the increasing volume concentration of nanofluids (water based aluminium oxide) decline the specific heat capacity of nanofluids owing to increase of thermal diffusivity of nanofluids. They also mentioned the increment of temperature causes the decrement of effective specific heat capacity of nanofluids. On the other hand, Zhou and Ni [58] and Sekhar and Sharma [57] experimentally studied that effective specific heat corresponds to nanoparticles diameter. Effective specific heat improved owing to higher diameter of nanoparticles; as thermal conductivity increases with an increase in particle diameter. Figure 6 presents when the temperature increased, the specific heat of base fluid and CNC nanofluids decreased simultaneously. Furthermore, the adding of CNC nanoparticles in base fluid causes the loss of specific heat capacity. But in this study, there is no gradual degradation of specific heat capacity of nanofluids with augmentation of volume concentrations. Besides that, specific heat capacity of 0.3 % CNC nanofluids is higher than the base fluid; this result is contradictory comparing with other literatures. Moreover, 0.3 % volume concentration of nanoparticles shows higher specific at 80 ^{0}C temperature than rest of the CNC nanofluids concentrations. Here among all other volume concentrations of CNC nanofluids, 0.5 % performs the best result up to 70 ^{0}C temperature.

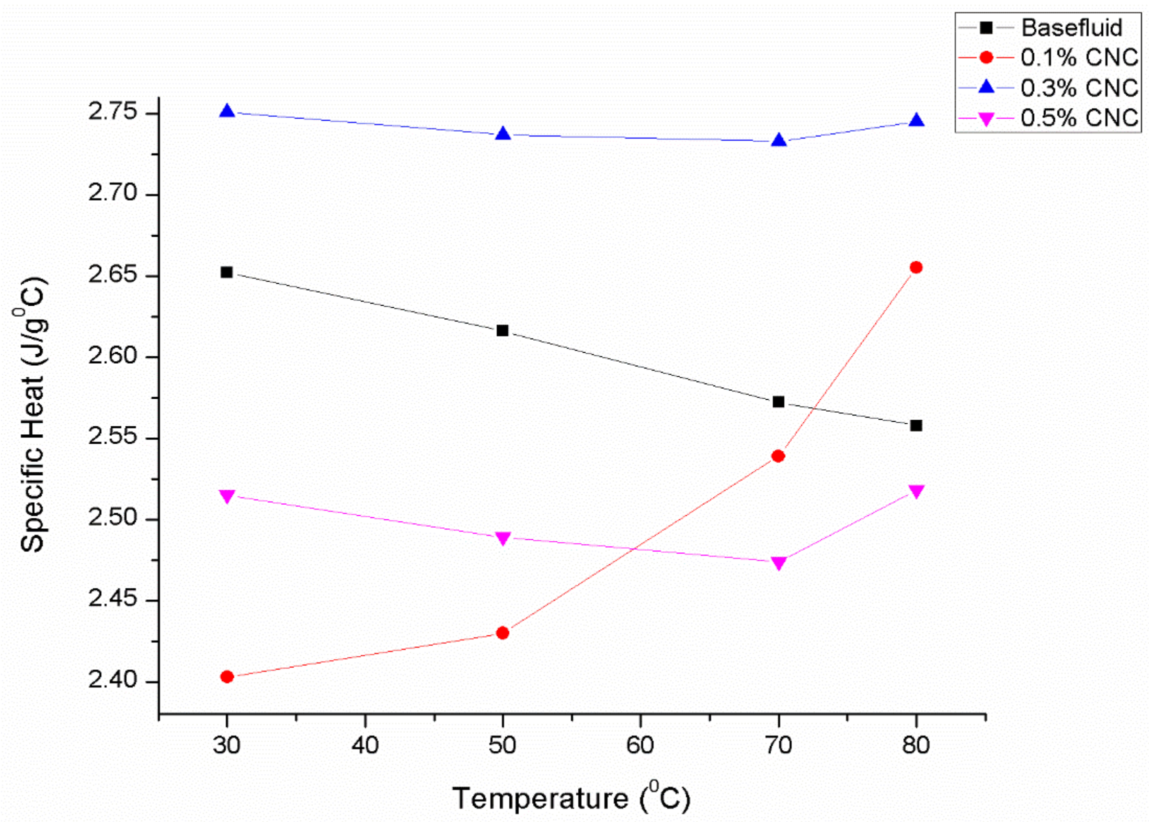

Fig. 6. Effect of temperature on specific heat of CNC nanofluids

3.5 Density Measurement of CNC Nanofluids

Generally, density of nanofluids is equivalent to the volume ratio of nano particles and base fluid. Base fluid performs an important role in the density of nanofluids. Density is also temperature sensitive characteristic of nanofluids. The increasing temperature decreases the density of nanofluids [59]. In this study, due to the deficiency (temperature) of equipment, density of nanofluid only measured at 20 ^{0}C for base fluid and different volumetric concentrations of CNC nanofluids as shown in Figure 7.

The result of density of base fluid is in good agreement with ASHREE data and the deviation is only about 0.77 %. The theoretical density data of all kind of fluids shown in Table 2. Experimental and theoretical density data has deviation within 0.26% which determine a satisfactory agreement between these two types of density values of base fluid and nanofluids as presented in Figure 7.

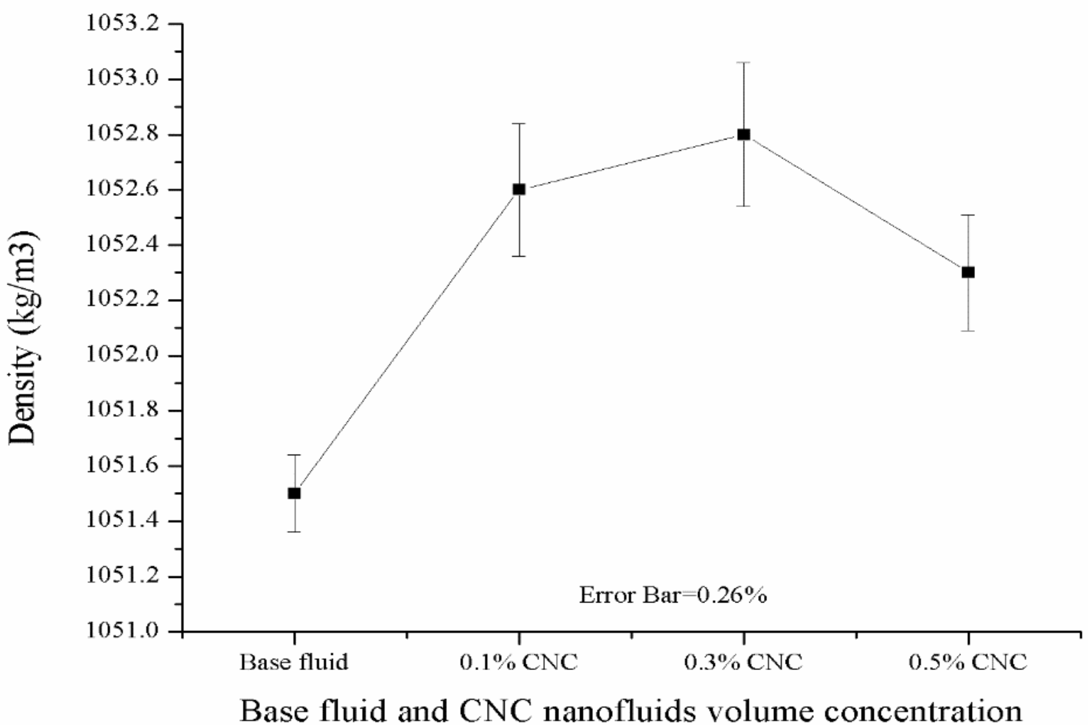

Fig. 7. Density measurement of base fluid and CNC nanofluids

Table 2
Theoretical density values of fluids

Items	Density (kg/m^3)
Base fluid (60: 40; W: EG)	1050 [60]
0.1% CNC nanofluids	1050
0.3% CNC nanofluids	1050
0.5% CNC nanofluids	1050

3.6 pH Analysis of CNC Nanofluids

pH is one of the most important factors of nanofluids as the stability of nanofluids depends on it. The increment of thermal conductivity well depends on the stability and the state of surface charge of nanofluids [61-62]. The more difference between the pH of nanofluids and the pH of isoelectric point (IEP) causes more stable nanofluids and increases efficiency as well [63-64]. Literatures stated pH value of different nanoparticles are not equivalent and has dependency on temperature, volume concentration and particle size [62-63, 65]. Jia-Fei *et al.,* [66] revealed that the small size of nanoparticles performs significantly fluctuations of pH values from 5 to 7. Moreover, low pH value performs good heat transfer property of nanofluids. Because in low pH condition, hydration forces among the particles increases in the suspension which results the mobility enhancement of nanofluids in the suspension to lead the heat transport process [67]. On the contrary, Goudarzi *et al.,* [64] experimentally concluded that higher pH values of nanofluids increases more efficiency than lower pH values. Figure 8 demonstrates that pH values of 0.1 % vol. concentration of CNC nanofluids are decreased than base fluids. On the other hand, pH value of 0.3 % CNC nanofluid at 30 ^{0}C and 50 ^{0}C is equal with base fluid as well as pH value of 0.5 % CNC nanofluids increased at 30 ^{0}C and 70 ^{0}C comparing with base fluid. But pH values of all CNC nanofluids decrease at 80 ^{0}C temperature.

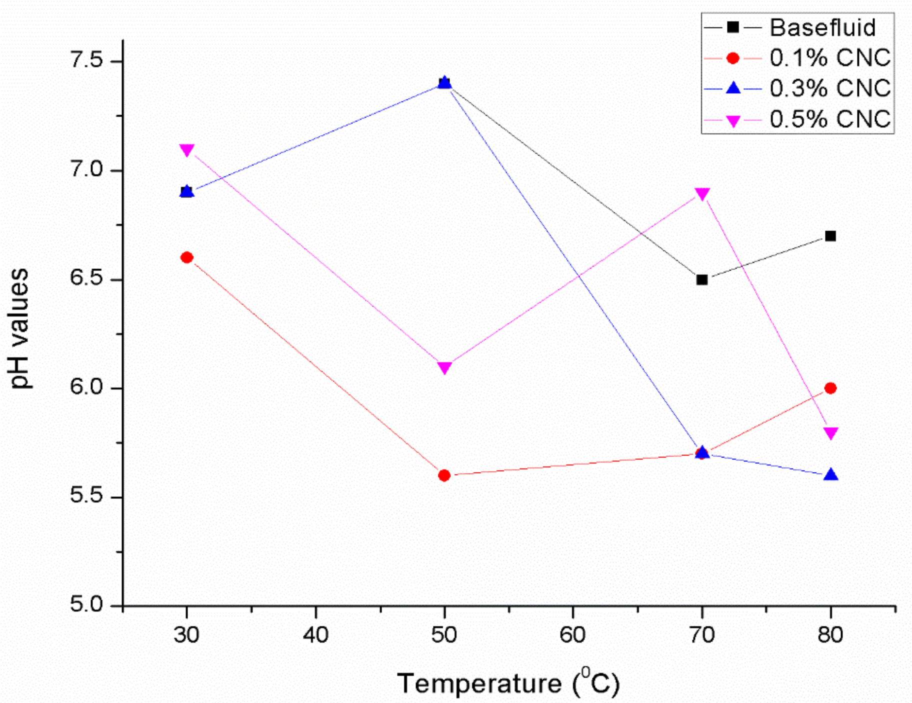

Fig. 8. pH values of different concentration of CNC nanofluids

4. Conclusions

Throughout the research work, stability of CNC nanofluids was very good and no sedimentation occurred; thermal conductivity has been increased for the addition of CNC nanoparticles to the aqueous ethylene glycol (60: 40; W: EG) base fluid. 0.3 % volume concentration of CNC nanofluids maintained the gradual increment of thermal conductivity within this temperature range, maximum thermal conductivity achieved at 80^{0}C and the improvement was of about 25 %. Experimental results of viscosity showed the decrement with increasing the temperature of CNC nanofluids. In case of specific heat, 0.5 % volume concentration performed excellent results than other concentrations and base fluid up to 70 ^{0}C temperature. The resultant data of density of CNC nanofluids were in good agreement with theoretical data and finally pH values of CNC nanofluids were in range of 5 to 7.5 confined with in this temperature range. However, some experimental values show the contradictory results compared with CNC nanofluids related previous literatures. The resultant data of thermal-physical properties of crystal nanocellulose nanofluids show the interesting and promising approach in respect of thermal engineering applications.

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