

Analysis of Multiwalled Carbon Nanotubes Porosimetry and their thermal conductivity with ionic liquid-based solvents

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ABSTRACT

The suspension of nanoparticles with common heat transfer fluids like Ethylene glycol and water yields nanofluid that exhibits superior thermal properties than their host fluids. Ionic liquids have the potential to demonstrate remarkable thermophysical properties (especially thermal conductivity) that ordinary nanofluids cannot realize. On the other hand, quantity and structure of nanoparticles porosity contributes a major impact on the thermal conductivity of nanofluids. Various investigations have revealed the enhanced thermophysical properties of Multi Walled Carbon nanotubes (MWCNTs) nanofluids containing common solvents or base fluids. However, only limited studies are available on the impact of thermal conductivity in Ionic liquid based nanofluids (Ionanofluids) owing to their high cost and viscosity. Ultrasonication technique is employed for preparing the three different Ionanofluids containing 0.5 Wt.% via two step method to achieve high stability and thermal conductivity without utilizing any surfactants. Experimental investigations are performed for increasing the thermal conductivity of MWCNT/Propylene glycol nanofluid using 1,3-dimethyl imidazolium dimethyl phosphate [Mmim][DMP], 1-ethyl-3-methyl imidazolium octylsulfate [Emim][OSO₄] and 1-ethyl-3-methyl imidazolium diethyl phosphate [Emim][DEP] at a temperature ranging from 295 K to 355 K. The obtained results show that thermal conductivity of MWCNT Ionanofluids incorporated with [Mmim][DMP], [Emim][OSO₄] and [Emim][DEP] increased by 37.5%, 5% and 2% respectively. This unique class of ionanofluids shows incredible potential for use as cutting edge heat transfer fluids in high temperature thermal applications.

Keywords: Ionanofluids; multi walled carbon nanotubes; thermal conductivity; heat transfer fluids.

INTRODUCTION

One of the most important requirements of many manufacturing techniques is ultrahigh cooling performance. However, a basic restriction in designing energy efficient heat transfer liquids, which are necessary for ultra-cooling is inherently poor thermal conductivity. This resulted in the development of new class of fluids having high thermal conductivity [1], [2]. One of the feasible paths for improving conventional

coolant thermal conductivity is the addition of nanoparticle to ionic liquids, a unique form of nanofluids called “Ionanofluids” that can dissipate large amount of heat owing to its great thermal conductivity leading to better thermal system efficiency. Nanofluids are suspensions of nanoparticles of less than 100 nm size with traditional heat transfer fluids like water, oil, propylene glycol and ethylene glycol. The main advantage of nanofluids are higher surface area, lower pumping power, higher stability, reduced particle clogging and high potential heat transfer properties [3]. Some of the common nanoparticles used in nanofluids are carbide ceramics (SiC, TiC), oxide ceramics (Al₂O₃, CuO), metals (Ag, Au, Cu), semiconductors (TiO₂, SiC) carbon nanotubes and doped materials. The objective of nanofluid is to obtain the best feasible heat transfer characteristics by standardized dispersion and robust concentration of nanoparticles in base fluids at lowest concentration (< 1% by volume). Salts that are liquid at room temperature is known as ionic liquids and it possess unique characteristics of high thermal stability, low vapour pressure which may be employed as heat transfer fluids in thermal applications where traditional fluids may not be appropriate in implementation due to their limited thermophysical and chemical characteristics. Furthermore, thermal conductivity depends on the maximum possible heat production of nanoparticles which, in turn, is limited by the morphology of their porosity. Usually, variations in processing conditions leading to different pore sizes and structures can also create discrepancies in thermal conductivity. Kreft et al. [4] proved that the thermal conductivity of an autoclaved concrete is mainly dependent on the size, number, distribution and structure of pores via mercury Porosimetry and gas adsorption techniques. Sant et al. [5] reported that increasing the concentration of nanoparticles increases the quantity of small pore that can lead to complicated internal structures of particles affecting the thermal properties. This is why the pore structure and thermal conductivity of the nanoparticles have been examined in many studies over the past decades.

Paul et al. [6] analysed the thermal performance of a circular tube using [C₄mim][NTf₂] ionic liquids and Al₂O₃ nanoparticles via continuum model and Eulerian model. They found that the ionic liquid based nanofluid improved the overall heat transfer coefficient of the tube for certain range of Reynolds number. Oster et al. [7] studied the mechanism behind the enhancement of heat capacity of ionanofluids based on [C₄C₁Im][DCA], [C₄C₁Im][NTf₂], [C₄C₁Pyrr][NTf₂], [C₆C₁Im][PF₆], [C₂C₁Im][C₂SO₄] with different concentration of carbon nanotubes, boron and graphite. They concluded that enhancement of heat capacity of ionanofluids is likely influenced by the interfacial nano layering on the nanoparticle surface. Wu et al. [8] prepared an ionic liquid [OMIM][NTf₂] based TiO₂ nanofluid to determine the traces of acaricides in honey and tea samples that resulted a speedy extraction process and reduction in the use of organic solvent. In a different study, Bose et al. [9] developed ionanofluids [Pyr₁₄ TFSI/TiO₂] based electrolyte that increased the lithium transference number and ionic conductivity in the electrolyte with a higher discharge capacity of 131 mAh g⁻¹ at 25 °C. On the other hand, Wang et al. [10] performed experimental and theoretical analysis on [Bmim][PF₆]/Gold ionanofluids and revealed that heat transport mechanism of ionanofluids depends mainly on the nature of base fluid at low temperature (<33°C) despite the Brownian motion which has been stated by other researchers also.

Recently, Cao et al. [11] experimentally investigated the effect of different ionic liquids on the optical properties of Ferrous oxide nanofluid to improve the absorption properties of a solar collector. Authors inferred that increasing the radius of anion or cation of ionic liquids decreases the absorption property of their proposed nanofluid. Minea et al. [12] provided a comprehensive review on the heat transfer behaviour and

thermophysical properties of ionic liquid based nanofluids which revealed the potential of ionic liquids in heat transfer applications especially under laminar flow conditions. By adding [EMIM][DEP] with an aqueous solution containing MWCNT nanoparticles and water, Xie et al. [13] obtained a 9.7% increase in thermal conductivity over the base fluid with a linear temperature dependency. Liu et al. [14] investigated the thermal stability and thermodynamic properties of graphene based ionanofluids for medium to high temperature applications. At a low concentration of 0.06 wt% of graphene nanoparticles, authors achieved a thermal conductivity enhancement of 22.9% over commercial heat transfer fluid (Therminol VP-1) and also showed a maximum thermal stability of 440 °C. Alina et al. [15] numerically evaluated the natural convection heat transfer efficiency of imidazolium based alumina nanofluids in a square enclosure layered with heated walls. Authors concluded that buoyancy force and viscous force were the main parameters that attributed to the enhancement of natural convection heat transfer. Since thermophysical properties are mainly dependent on the dispersion stability of nanoparticles in the base fluid, Jian et al. [16] attempted to modify the molecular structure of graphene nanoparticles using [HMIM][BF₄] ionic liquids to enhance the dispersion stability. Authors found that the modified graphene was more stable than the pristine graphene even at high temperature. Chen et al. [17] improved the optical and thermal properties Silicon nanofluids with addition of [HMIM][BF₄] ionic liquids that resulted in 10.2% thermal conductivity enhancement and 5% specific heat capacity improvement. Many researchers proved that the combination of ionic liquids and nanoparticles demonstrated great potential as heat transfer fluids by their improved thermal properties. Moreover, literature review has shown that only minimal work has been carried out on the thermal conductivity enhancement of nanofluids with ionic fluids. The purpose of this study is to analyse the thermal conductivity and stability of 0.5% of MWCNT nanofluid based ionic liquids.

METHODS AND MATERIALS

Materials

Multi-walled carbons nanotubes in dry powder form with an average particle size of 24 nm were procured from Sigma Aldrich. Propylene glycol and ionic liquids such as [EMIM][OSO₄], [EMIM][DEP] and [MMIM][DMP] (Sigma Aldrich, USA) were used as the base fluid [18]. The thermophysical properties of the nanoparticle is shown in Table 1 [19]. The reason for choosing MWCNT is because of their remarkable thermal conductivity. In Figure 1 and 2, the images of MWCNT nanoparticles and ionic fluids used in this experiment are shown.

Table 1 Properties of nanoparticles

Properties	Specifications
Formula	MWCNTs
Shape Size	Cylindrical Outer Diameter =24 nm Length = ~20 μm
Purity	>98%
Appearance	Powder
Colour	Black
Surface area	220 m ² /g
Density	0.28 g/cm ³



Figure 1. Photograph of raw MWCNT nanoparticles



Figure 2. Image of ionic liquids (a)EmimOSO₄ (b)EmimDEP (c)MmimDMP

Preparation of ionic liquid based nanofluid

Two step method was employed to prepare four different ionanofluids. The mass concentration of each sample is fixed to 0.5 wt% and it is calculated by Eq. (1). To produce ionanofluids, sufficient nanoparticles have been added to the base fluid which was a combination of Propylene glycol and ionic liquid (80:20). In order to avoid accumulation of nanoparticles and breakdown of agglomerates, an ultrasonic processor of 750 watts was used for all samples at 20 kHz. In view of the reality that elevated sonic time contributes to secondary coalescence of nanoparticles, the suitable period for sonication was first determined. All the samples have been sonicated for 2 hours in this respect.

$$\text{Weight \%} = \frac{\text{Wight of nanoparticles (g)}}{\text{Volume of base fluid (l)}} \quad (1)$$

Surface Morphology

One of the most popular and widely used method for the characterisation of nanostructure and nanomaterials is Field Emission Scanning Electron Microscope (FESEM). To get an idea of shape, size and defects of nanoparticles FESEM, ZEISS Sigma 500 was used. It consists of a Gemini objective lens incorporated with electrostatic and magnetic fields that maximizes the optical performance and the imaging quality even with magnetic materials. In this equipment, Schottky thermal field emitter has been used as an electron source with a maximum resolution of 0.8 nm at 30 kv.

Porosity Measurement

The efficiency of solid particle is known to depend on its surface are and pore morphology. Porosity of standard solid particle is usually measured by adsorption phenomena, which can also be used to evaluate the nanoparticles porosity and surface area. Thus it is important to calculate the adsorption of gases to provide details on the surface and pores of nanoparticles. In this study an Accelerated Surface Area & Porosimetry System (ASAP 2020 Plus, Micrometrics, USA) equipped with twelve gas inlets that permits multiple probe gases was used [20], [21], [22]. With a 1100 °C

furnace, stable and excellent temperature ramp could be achieved without any user intervention. The maximum pressure, resolution, sample temperature ranges from 0 to 950 mmHg, up to 1×10^{-7} torr, ambient to 1100 °C respectively.

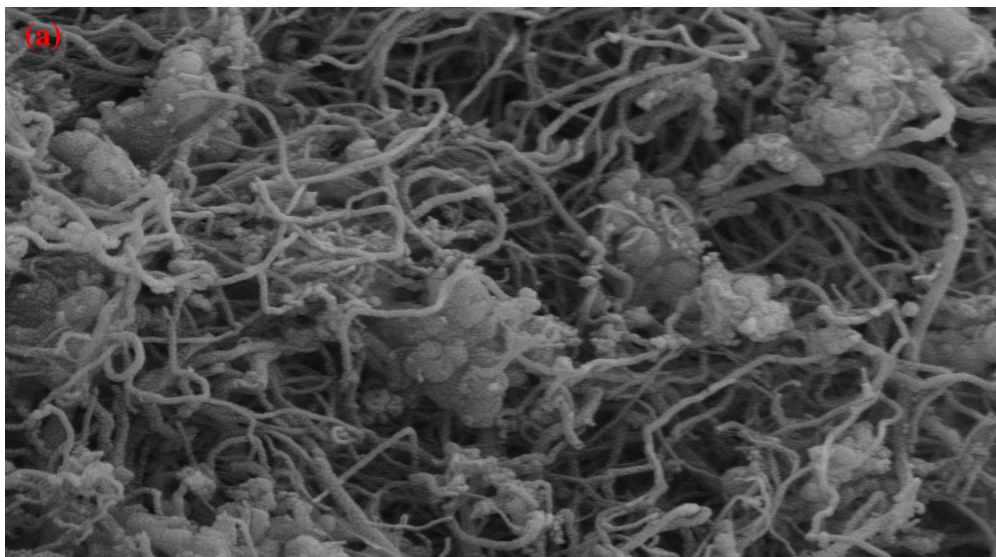
Thermal Conductivity

Temperatures of 295 K to 355 K encompassed with 0.5 wt% concentration were used to determine the thermal conductivity of the as prepared MWCNT ionanofluid. KD2 Pro thermal conductivity meter (Decagon Devices, USA) was used for evaluating the thermal conductivity. It consists of a double needle sensor that measures the thermal conductivity of the ionanofluids using a transient heat source approach. Distilled water's thermal conductivity was measured and contrasted with that of conventional values at a temperature of 295 K to check the precision and feasibility of readings which resulted in a deviation of 0.5%.

RESULTS AND DISCUSSION

Morphological analysis of MWCNT nanoparticles

The structural morphology of MWCNT nanoparticles was analysed using Field Emission Scanning Electron Microscope (FESEM) which is presented in Figure 3. The FESEM image of different parts of the nanoparticle was taken with an electron high tension (EHT) of 6 kV (Figure 3a) and 10 kV (Figure 3b). These FESEM images with different magnifications indicate that the tested Multi walled carbon nanotubes takes the form of a strong agglomerated phase which is not suitable for evaluating its exact size and structure and it can also lead to instability and agglomeration at a significant rate. The tube-like structures assured the cylindrical shape of the nanoparticle with certain diameters ranging from 20 nm to 40 nm and its lengths were found to be uniform.



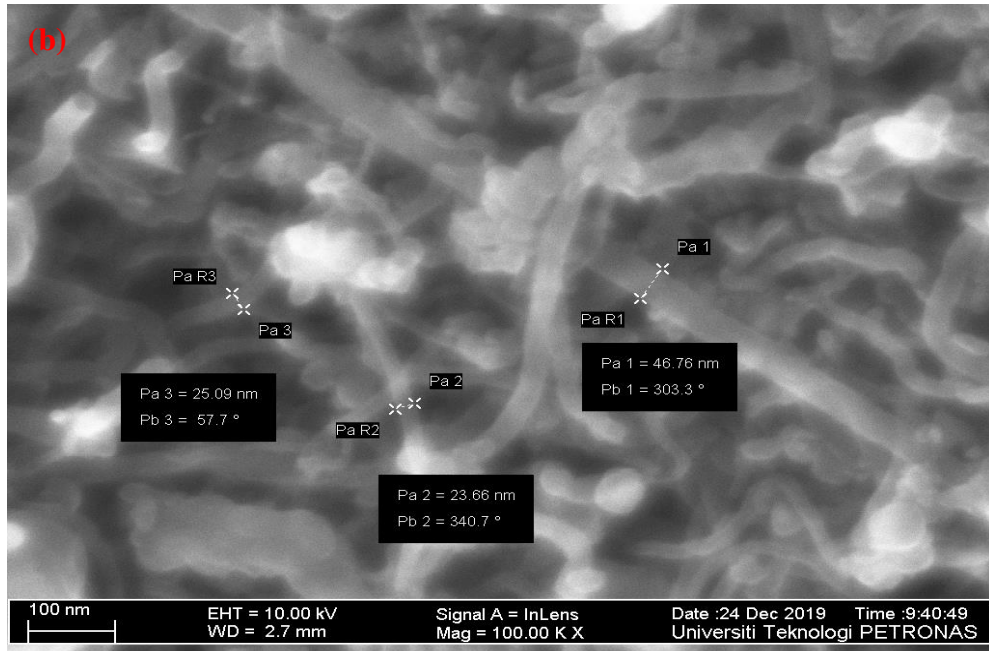
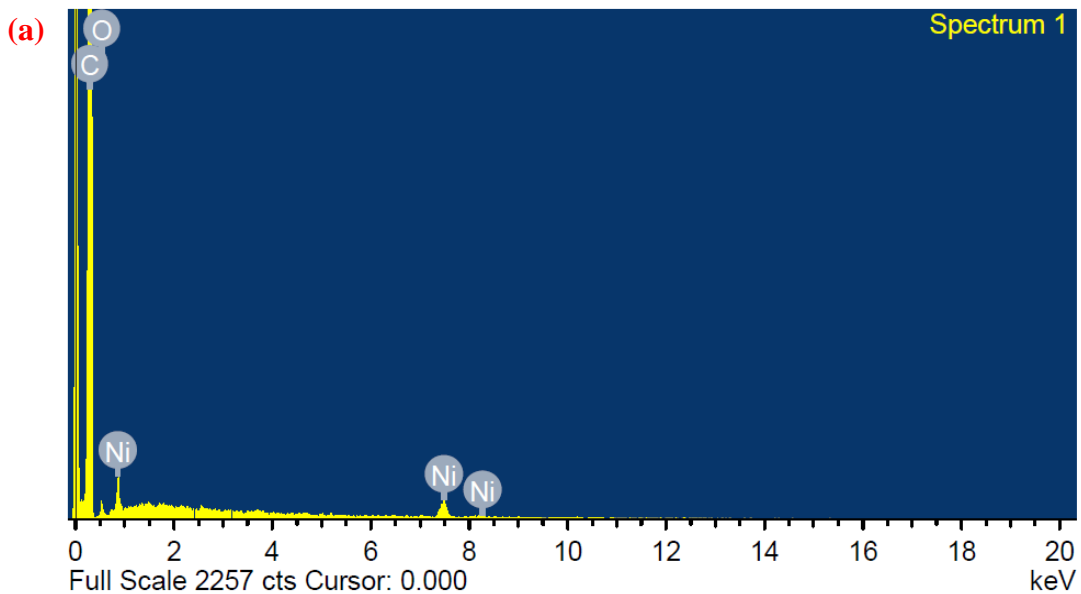


Figure 3. FESEM image of MWCNT nanoparticle at different EHT (a) 6 kV (b) 10 kV

EDX and Mapping

Along with FESEM, Energy Dispersive X-ray spectroscopy (EDX) was also analysed which is presented in Figure 4. In an FESEM equipped with EDX detector, the electron beam induces specific excitations in the specimen which are representatives of the elements in the sample. For elemental identification, characteristic X-rays emitted from samples due to hole decay can be used.



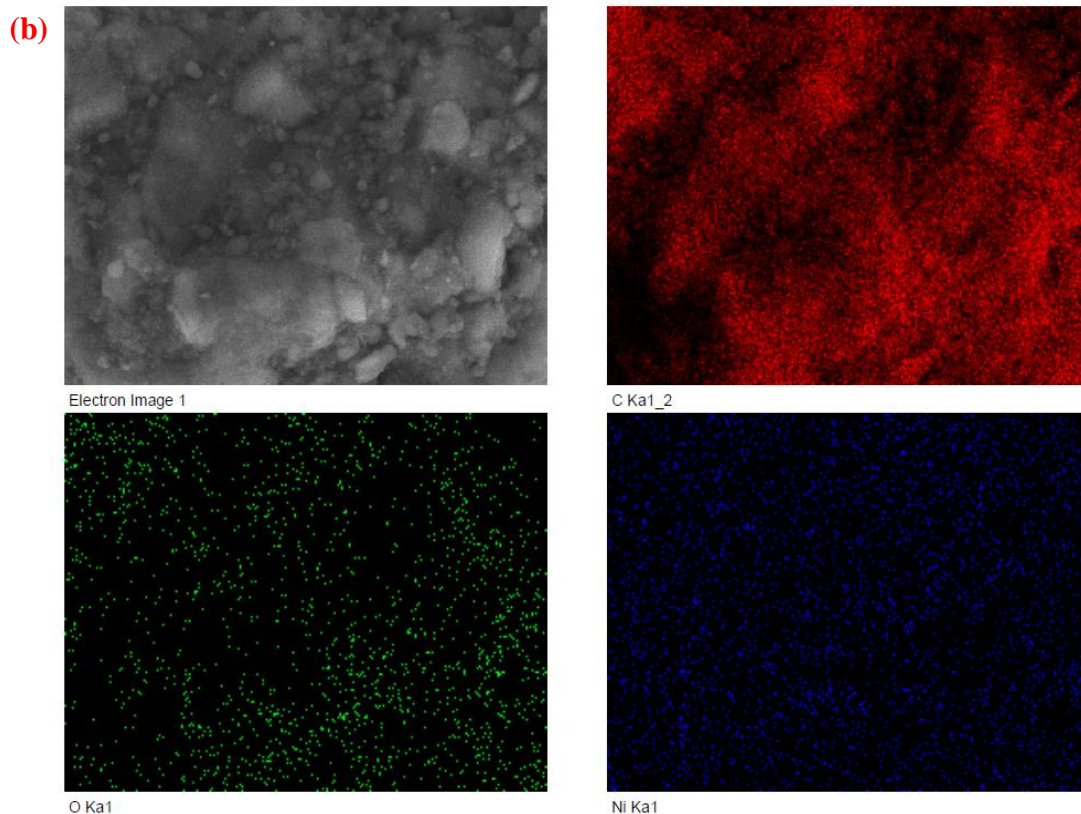


Figure 4. EDX and Mapping analysis of MWCNT nanoparticles

The presence of various atomic elements was revealed in the EDX analysis. The detected elements are carbon (94.04%), Oxygen (3.75%) and Nickel (2.20%). Figure 4(a) confirms the presence of carbon, oxygen and nickel which was automatically detected by the system. Figure 4(b) contains the spectrum area and mapping of that area where the distribution of atomic elements is displayed.

Surface area and Porosimetry analysis

The two significant parameters that affects the quality and effectiveness of nanomaterials are surface area and porosity. Surface area and porosity plays an important role in processing, purification, blending, surface reactivity, dissolution rate and stability that can be interpreted with adsorption and desorption isotherms plots. To gain adsorption and desorption isotherms, and data on the surface and porosity of MWCNT nanoparticle, Accelerated Surface Area and Porosimetry System (ASAP 2020, Micromeritics USA) was used. The measurements of the surface area with the distributions of pore amounts and pores were carried out with the standard gas ultra-high purity (UHP) nitrogen. This device was also utilised to calculate parameters such as BET and Langmuir surface regions, average and maximum pore density, BJH (Barrett Joiner Halenda) pore size distribution and micro-pore structure. Adsorption is the process of binding molecules from the gas phase to a solid surface while desorption is the process of extracting molecules from the solid particles. Figure 5 clearly indicates the experimental isotherms of the MWCNTs that ensured the characteristics of pure mesoporous (20 – 500 Å) material and also illustrated a well-defined cylindrical pore channel. Furthermore, the hysteresis loop of the desorption branch attributed the

equilibrium phase transition where BJH method could be used to determine the exact pore size.

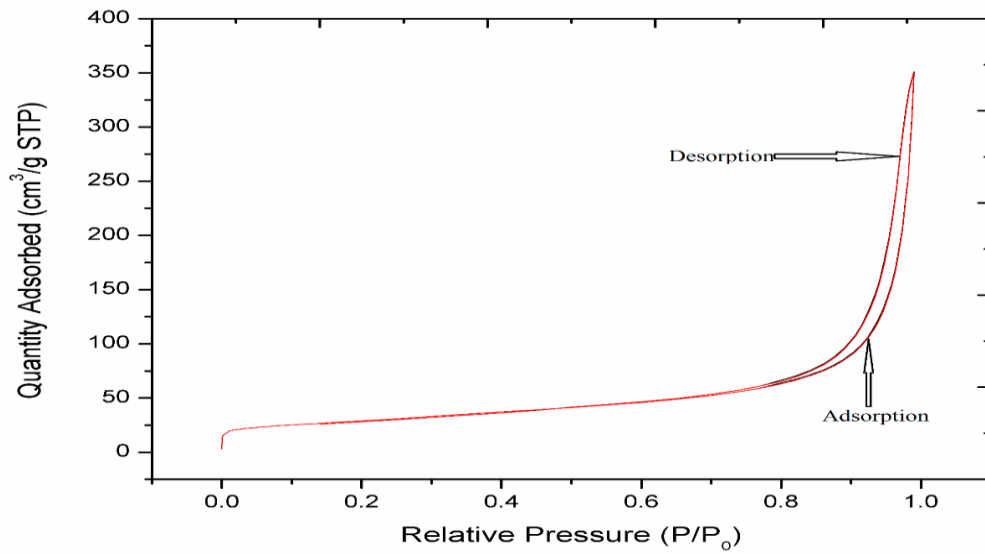


Figure 5. Adsorption and Desorption isotherms of MWCNT nanoparticles

From the ASAP analysis the MWCNT nanoparticles resulted an average surface area of 100 m²/g with pore volume and size of 0.5 cm³/g and 20 nm respectively. Table 2, Table 3 and Table 4 displays the measured surface area, pore volume and pore size at different parameters.

Table 2 Measured surface area of MWCNT nanoparticles

Parameters	Surface Area (m ² /g)
Langmuir Surface Area	121.9848
Desorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm width (BJH)	104.5659
Single point surface area at P/Po = 0.199916132	100.8505
BET Surface Area	100.7346
Adsorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm width (BJH)	100.186
t-Plot External Surface Area	84.8438
t-Plot Micropore Area	15.8908

Table 3 Pore volume of MWCNT nanoparticles at different specifications

Parameters	Pore Volume (cm ³ /g)
Adsorption cumulative volume of pores between 1.7000 nm and 300.0000 nm width (BJH)	0.546542
Desorption cumulative volume of pores between 1.7000 nm and 300.0000 nm width (BJH)	0.544906
Single point desorption total pore volume of pores less than 114.8210 nm width at P/Po = 0.982845033	0.516129
Single point adsorption total pore volume of pores less than 114.2328 nm width at P/Po = 0.982755021	0.406318
Micropore volume t-plot	0.007771

Table 4 Size of pores measured

Parameters	Pore size (nm)
Average pore width in Adsorption (BET)	16.13422
Average pore width in Desorption	20.49460

(BET)	
BJH Adsorption average pore width	21.8211
BJH Desorption average pore width	20.8445

Thermal conductivity

The measured thermal conductivity of propylene glycol base fluid [23] is compared with ASHRAE [24] and Bejan [25] for PG/Water (60:40) and PG respectively is presented in Figure 6. Thermal conductivity measurements were taken for 4 different samples of MWCNT nanoparticles dispersed in propylene glycol and ionic liquids (80:20) with a temperature range of 295K to 355K.

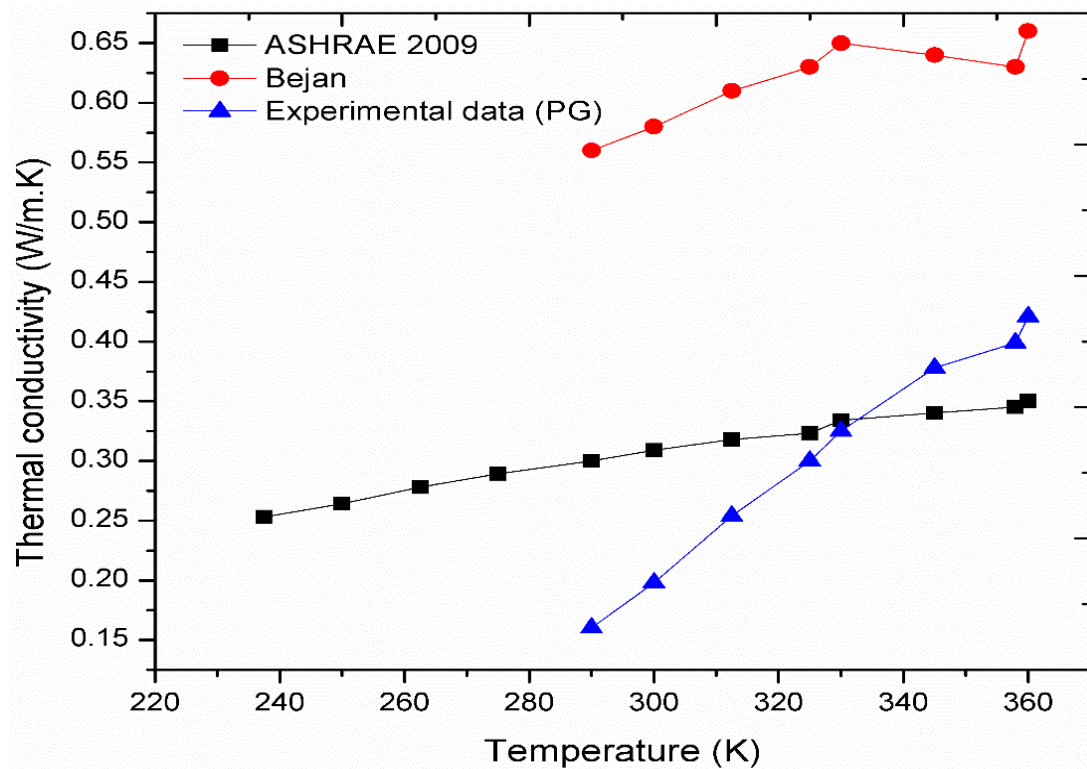


Figure 6. Thermal conductivity validation of reference fluids

The thermal conductivity of the formulated nanofluid and ionanofluid are shown in Figure 7 as a function of temperature. Results show that the thermal conductivity of ionanofluids increases as the temperature increases and this increase is more amplified at elevated temperatures. The highest increase in thermal conductivity was observed for MWCNT/PG/MmimDMP at 355 K which is 37.5% higher than the propylene glycol base fluid. This important improvement in thermal conductivity can be ascribed to the increase in particle movement owing to the rise in temperature and an increase in the rate of interactions with nanoparticles [26] and an increase in energy levels [27]. The other factors that contributed to the thermal conductivity enhancement were nanometre sized particles, high surface area and weaker molecular bonds.

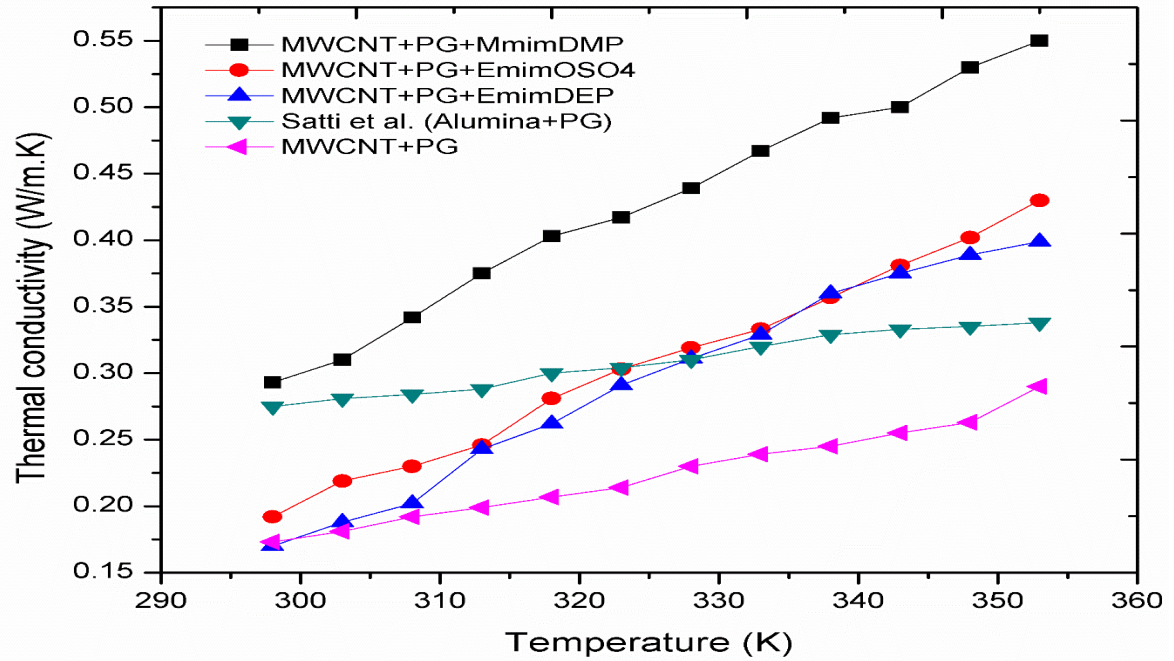


Figure 7. MWCNT ionanofluids thermal conductivity at different temperatures

Stability

Nanoparticles tends to aggregate after a certain period of time that affects the thermophysical properties of the ionanofluids due to the occurrence of intermolecular forces of attraction. Ionanofluids stability has to be considered vigorously as it was an important factor to enhance the thermal conductivity of nanofluids. As presented in Figure 8, the visual inspection of the prepared samples showed stability for 72 hours except MWCNT/PG nanofluid which does not contain any ionic liquids. The ionic liquid based nanofluids have not shown any major sedimentation or aggregation within the specified time period.

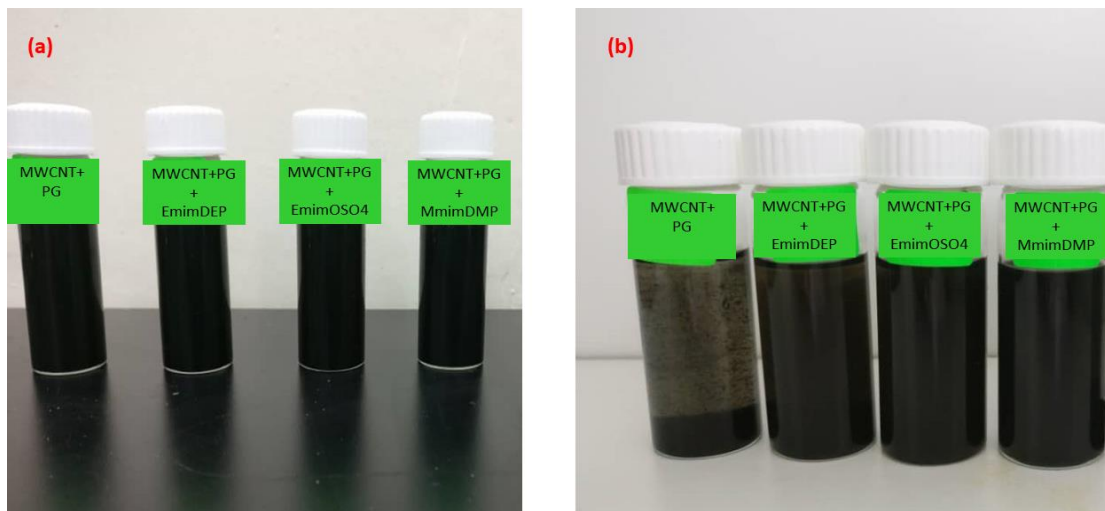


Figure 8. Photograph of prepared samples (a) Sample after immediate preparation (b) Sample after 72hours

CONCLUSIONS

This research analysed the Porosimetry of Multiwalled carbon nanotubes along with the effect of ionic liquids in MWCNT nanofluids on thermal conductivity and stability with a mass concentration of 0.5 wt%. Overall the surface area, pore volume and pore size of the studied MWCNTs ranged from 15 to 121 m²/g, 0.0077 to 0.54 cm³/g and 16.13 to 20 nm respectively. From the results, it was found that MWCNT/PG/MmimDMP showed a maximum thermal conductivity of 0.54 W/m.K at 355K and it was also observed to be more stable without any sedimentation when compared with other fluids. This study revealed that nanofluids' thermal conductivity depends primarily on the stability of the nanoparticles in the base fluid. Moreover, less concentration of ionic liquids increased the thermal conductivity of the prepared samples that might help researchers to enhance other thermophysical properties of nanofluids without surfactants.

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Nomenclature

MWCNT	Multi walled carbon nanotubes	Wt%	Mass fraction
IL	Ionic Liquids	T	Temperature
PG	Propylene Glycol	K	Thermal conductivity
[Mmim][DMP]	1,3-dimethyl imidazolium dimethyl phosphate	ρ	Density
[Emim][OSO4]	1-ethyl-3-methyl imidazolium octylsulfate	μ	Viscosity
[Emim][DEP]	1-ethyl-3-methyl imidazolium diethyl phosphate	C_p	Specific heat capacity
[C4mim][NTf2]	1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide		
[Pyr14][TFSI]	1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl) imide		
[OMIM][NTf2]	1-octyl-2,3-dimethylimidazolium bis(trifluoromethylsulfonyl) imide		
Al ₂ O ₃	Aluminium oxide		

TiO ₂	Titanium dioxide
SiC	Silicon Carbide