Analysis of Multiwalled Carbon Nanotubes Porosimetry And Their Thermal Conductivity with Ionic Liquid-Based Solvents

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ARTICLE INFO	ABSTRACT
	The suspension of nanoparticles with common heat transfer fluids like Ethylene glycol and water yields nanofluid 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 achieve. On the other hand, the quantity and structure of nanoparticles porosity affects the nanofluid's thermal conductivity considerably. Various investigations have revealed the improved thermophysical characteristcts of Multiwalled 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 in preparing the three different Ionanofluids containing 0.5 Wt.% via the two-step method to achieve a greater stability and thermal conductivity without utilizing surfactants. Experimental investigations are performed to boost the thermal conductivity of MWCNT/Propylene glycol nanofluid using 1,3-dimethyl imidazolium dimethyl phosphate [Mmim][DMP], 1-ethyl-3-methyl imidazolium octyl sulfate [Emim][OSO4] and 1-ethyl-3-methyl imidazolium diethyl phosphate [Emim][DEP] at a temperature ranging from 295 K to 355 K. The acquired results illustrated that the 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 capacity for use in high temperature applications as conventional heat transfer fluids.

1. Introduction

One of the essential requirements of many manufacturing techniques is ultrahigh cooling performance. However, a fundamental restriction in designing energy-efficient heat transfer liquids

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necessary for ultra-cooling is inherently poor thermal conductivity. Due to this, a new kind of high thermal conductivity fluids was synthesized [1-2]. One of the feasible paths to improve conventional coolant thermal conductivity is the addition of nanoparticle to ionic liquids, a unique form of nanofluids called "Ionanofluids" that can dissipate a large amount of heat owing to its great thermal conductivity, leading to better thermal system efficiency. Coventional heat transfer fluids such as water, oil, ethylene and propylene glycol are suspendend with nanoparticles of less than 100 nm in size to synthesize a high effieient heat transfer fluid called nanofluids. The main advantages of nanofluids are higher surface area, lower pumping power, higher stability, reduced particle clogging and high potential heat transfer properties [3]. Some of the conventional nanoparticles used in nanofluids are carbide ceramics (SiC, TiC), oxide ceramics (Al_2O_3, CuO), metals (Ag, Au, Cu), semiconductors (TiO_2, SiC), carbon nanotubes and doped materials. The objective of nanofluid is to obtain the best feasible heat transfer characteristics through standardized dispersion and robust concentration of nanoparticles in base fluids at the lowest concentration (< 1% by volume). Salts that are liquid at room temperature is known as ionic liquids, and it possesses unique characteristics of high thermal stability and low vapour pressure, which may be employed as heat transfer fluids in thermal applications where traditional fluids may not be appropriate for 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 reliant on the size, number, distribution and structure of pores via mercury porosimetry and gas adsorption techniques. Sant *et al.,* [5] reported that increment in nanoparticles concentration leads to the increase of small pores quantity which 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] investigated the thermal performance of a circular pipe using [C4mim][NTf2] ionic liquids and Al_2O_3 nanoparticles via the continuum model and the Eulerian model. Authors observed that the ionic liquid-based nanofluid improved the total heat transfer coefficient of the tube for a certain range of Reynolds number. Oster *et al.,* [7] examined the factors affecting heat capacity improvement of ionanofluids based on [C4C1Im][DCa], [C4C1Im][NTf2], [C4C1Pyrr][NTf2], [C6C1Im][PF6], [C2C1Im][C2SO4] with different concentrations of carbon nanotubes, boron and graphite. They concluded that the enhancement of ionanofluids' heat capacity is likely influenced by the interfacial nano layering on the nanoparticle surface. Wu *et al.,* [8] prepared an ionic liquid [OMIM][NTf2] based TiO_2 nanofluid to determine the traces of acaricides in honey and tea samples which resulted in a quick synthesize procedure and reduction in the utilization of organic solvent. In a different study, Bose *et al.,* [9] developed ionanofluids [Pyr14 TFSI/TiO_2] based electrolyte that increased the transference number of lithium and conductivity of ions 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. They revealed that heat transport mechanism of ionanofluids depends mainly on the nature of base fluid at low temperature (<33°C) despite the Brownian motion, whereby similar findings have been found in other researches.

Recently, Cao *et al.,* [11] evaluated the impact of a variety of ionic liquids on the optical characteristics of Ferrous oxide nanofluid to improve the absorption properties of a solar collector. The authors inferred that enhancement of anion and cation radius of ionic liquids decreases the absorption property of their proposed nanofluid. Minea *et al.,* [12] thoroughly analysed 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 greater thermal conductivity enhancement of 9.7% juxtaposed to the base liquid with a linear temperature dependency. Liu *et al.,* [14] researched 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, they reached an increase of 22.9% thermal conductivity over commercial heat transfer fluid (Therminol VP-1) which showed maximum thermal stability of 440 °C. Alina *et al.,* [15] numerically evaluated the heat transfer efficiency of imidazolium-based alumina nanofluids under natural convection mode in an oblong casing layered with heated walls. They concluded that the buoyancy force and viscous force were the main parameters attributed to the improvement of natural convection heat transfer. Since thermophysical characteristics are mainly dependent on nanoparticles' dispersion stability in the base fluid. Several researchers intended on enhancing nanofluid's stability with surfactants which resulted in high viscosity affecting the liquid flow. Also, the addition of excessive surfactants changed the original nature of heat transfer fluid leading to less dispersion stability. Therefore, scientists are concentrated on developing a new class of nanofluids consisting of ionic liquids which are environmentally friendly and corrosion resistant. Jian *et al.,* [16] attempted to modify the molecular structure of graphene nanoparticles using [HMIM][BF4] ionic liquids to enhance the dispersion stability. They 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 characteristics of Silicon nanofluids with the addition of [HMIM][BF4] ionic liquids which resulted in 10.2% thermal conductivity enhancement and 5% specific heat capacity improvement. Many researchers proved that the mixture of ionic liquids and nanoparticles is a suitable alternative to be used as fluids for heat transfer attributable to their enhanced pore structure and thermal properties. However, the literature review has shown that only minimal work has been carried out on the Porosimetry of MWCNTs and thermal conductivity enhancement of MWCNT nanofluids with ionic fluids. This research strives to measure the pore size and volume, and the surface area of MWCNTs with a view on the thermal conductivity and stability analysis of 0.5 wt.% of MWCNT nanofluid based ionic liquids.

2. Methodology
2.1 Materials

Multi-walled carbons nanotubes were procured from Sigma Aldrich, in dry powder form with a mean particle size of 24 nm. Propylene glycol and ionic liquids such as [EMIM][OSO4], [EMIM][DEP] and [MMIM][DMP] (Sigma Aldrich, USA) were utilized as base fluids. The significant properties of the studied ionic liquids are analyzed in our previous study [18]. The thermophysical characteristics properties of the nanoparticle are shown in Table 1.

Table 1
Properties of nanoparticles [19]

Properties	Specifications
Formula	MWCNTs
Form	Cylindrical
Dimensions	OD=24 nm, L=20 μm
Purity	>98%
Appearance	Powder
Colour	Black
Surface area	220 m^2/g
Density	0.28 g/cm^3

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.

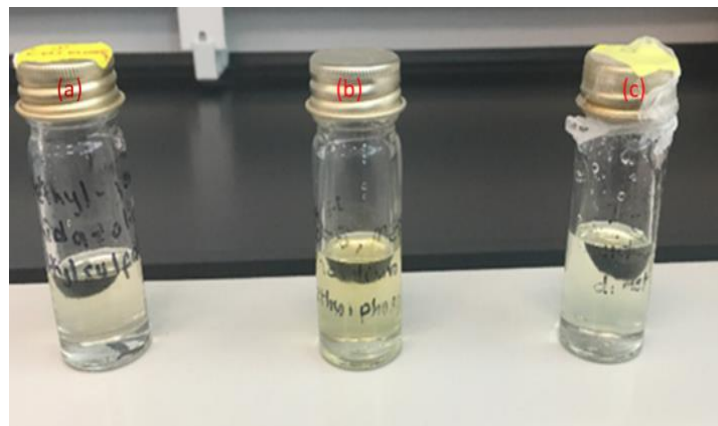
Fig. 1. Photograph of raw MWCNT nanoparticles

Fig. 2. Image of ionic liquids (a)[Emim][OSO4] (b)[Emim][DEP] (c)[Mmim][DMP]

2.2 Preparation of Ionic Liquid-Based Nanofluid

Two-step method was employed to concoct 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 were incorporated to the base fluid, which is a combination of Propylene glycol and ionic liquid (80:20). To avoid the accumulation of nanoparticles and breakdown of agglomerates, an ultrasonic processor of 750 watts was used for all samples at 20 kHz. Because of the reality that elevated sonic time contributes to the secondary coalescence of nanoparticles, a suitable period for sonication was first determined. All the samples were sonicated for 2 hours in this respect.

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

2.3 Surface Morphology

Field Emission Scanning Electron Microscope (FESEM) is one of the most popular methods used for the characterization of nanostructures and nanomaterials. Using FESEM ZEISS Sigma 500, the shape, size, and defects of the studied nanoparticles are analyzed. It consists of a Gemini objective lens incorporated with electrostatic and magnetic fields which maximizes the optical performance

and the imaging quality even with magnetic materials. In this equipment, Schottky thermal field emitter was used as an electron source with a maximum resolution of 0.8 nm at 30 kV.

2.4 Porosity Measurement

The efficiency of the solid particle is known to depend on its surface area and pore morphology. The 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 crucial 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, Micro metrics, USA) which was followed by previous literature [20-22]; equipped with twelve gas inlets that permits multiple probe gases was used. With an 1100 °C furnace, stable and excellent temperature ramp could be achieved without any user intervention. The maximum pressure, resolution, sample temperature in this study ranged from 0 to 950 mmHg, up to 1 x 10-7 torr, and ambient to 1100 °C, respectively.

2.5 Thermal Conductivity

The temperatures between 295K to 355K encompassed with the sample's 0.5 wt.% concentration was constrained to assess the thermal conductivity of the as-prepared MWCNT ionanofluid. KD2 Pro thermal conductivity meter (Decagon Devices, USA) was used to evaluate the thermal conductivity. It consists of a double-needle sensor to measure the thermal conductivity of the ionanofluids using the 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%.

3. Results
3.1 Morphological Analysis of MWCNT Nanoparticles

A field emission scanning electron microscope has been used to study MWCNT nanoparticle's structural morphology, which is presented in Figure 3 and Figure 4. The FESEM image of different parts of the nanoparticle was taken with a high electron tension (EHT) of 6 kV (Figure 3) and 10 kV (Figure 4). These FESEM images with different magnifications indicate that the tested Multiwalled carbon nanotubes take the form of an intense 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 specific diameters ranging from 20 nm to 40 nm, and its lengths were found to be uniform.
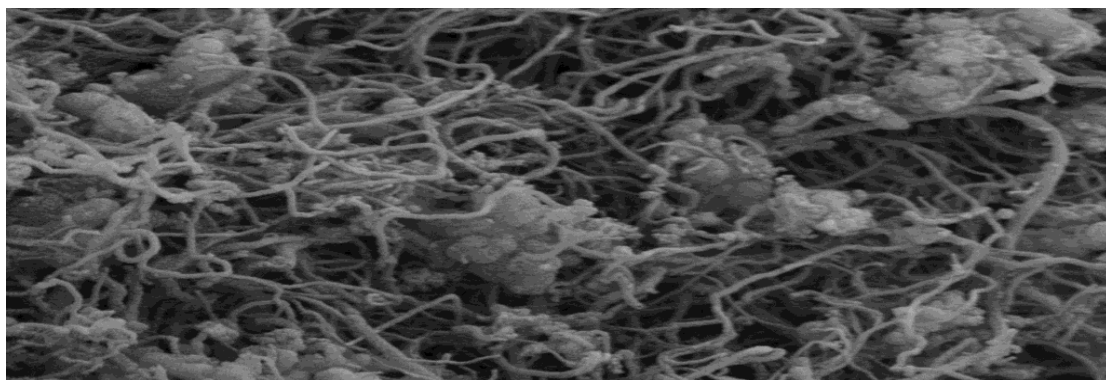

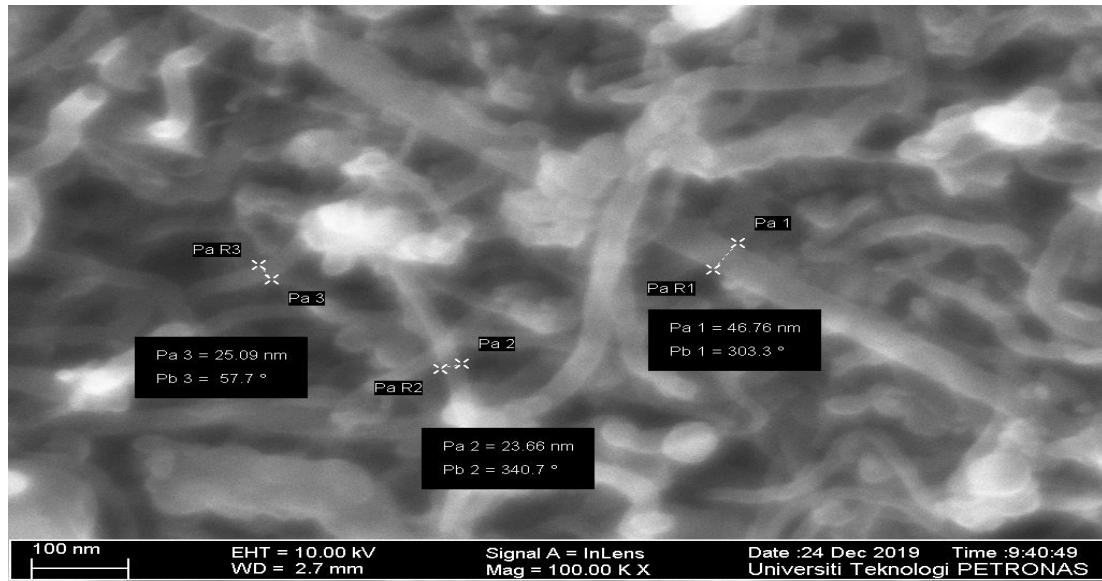**Fig. 3.** FESEM image of MWCNT nanoparticle at 6 kV EHT

Fig. 4. FESEM image of MWCNT nanoparticle at 10 kV EHT

3.2 EDX and Mapping

Along with FESEM, Energy Dispersive X-ray spectroscopy (EDX) was also analyzed, which is presented in Figure 5. In a 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.
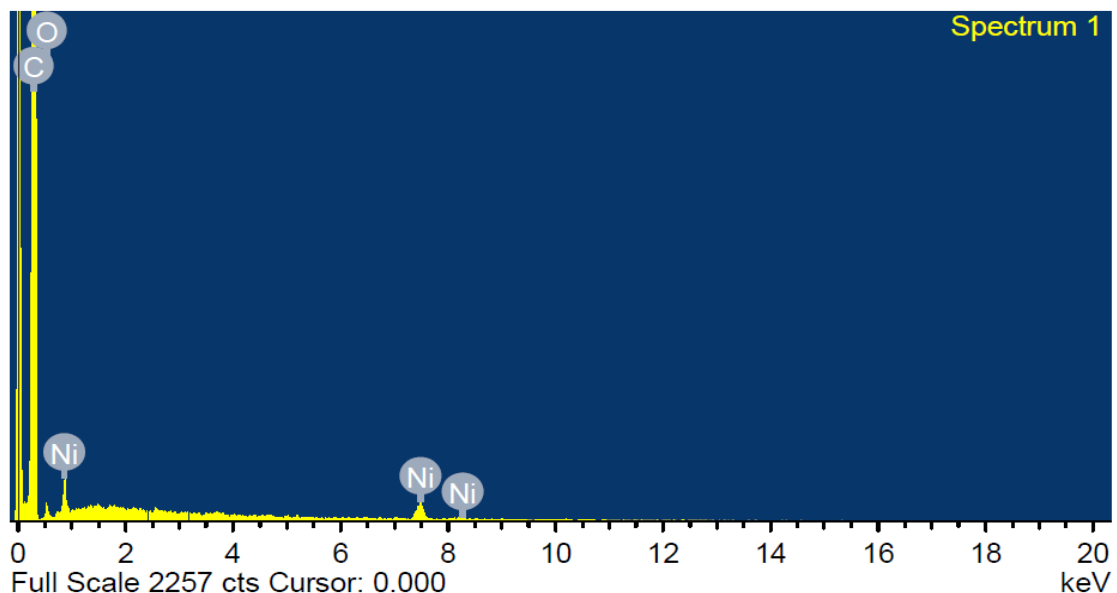

Fig. 5. EDX analysis of the studied MWCNT nanoparticles

The presence of various atomic elements was revealed in the EDX analysis. The detected items are carbon (94.04%), Oxygen (3.75%) and Nickel (2.20%). Figure 5 confirms the presence of carbon, oxygen and nickel, which was automatically detected by the system. Figure 6 contains the spectrum area (Electron image 1) and mapping of that area (C Ka1_2, O Ka1, Ni Ka1) where the distribution of

atomic elements is displayed. In the figure, the red color illustrates the presence of carbon while green color and blue color demonstrates the existence of oxygen and Nickel elements, respectively.
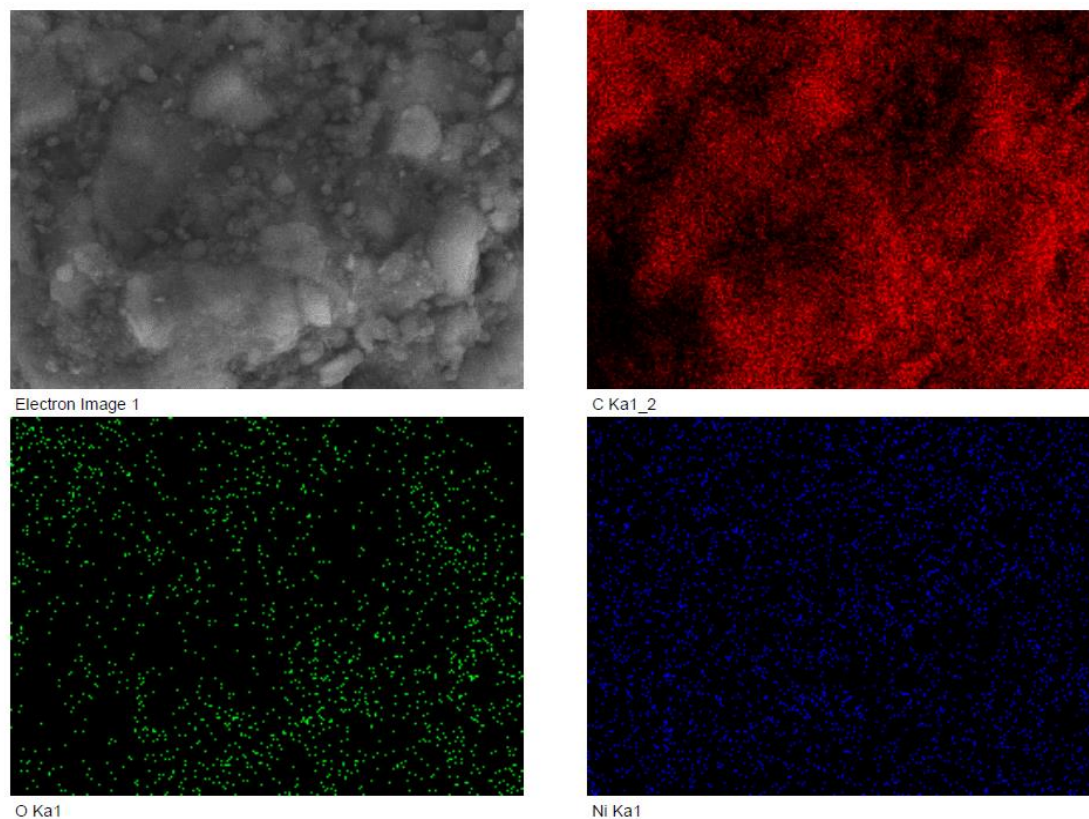

Fig. 6. Mapping analysis of the MWCNT nanoparticle

3.3 Surface Area and Porosimetry Analysis

The two significant parameters that affect the quality and effectiveness of nanomaterials are surface area and porosity. These parameters play an important role in processing, purifying, blending, surface reactivity, dissolution rate and stability, which 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 utilized 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 7 indicates the experimental isotherms of the MWCNTs, which ensured the characteristics of pure mesoporous (20 – 500 Å) material. It also illustrates a well-defined cylindrical pore channel. Furthermore, the hysteresis loop of the desorption branch is attributed to the equilibrium phase transition, where the BJH method is a viable method to be used to measure the exact pore size.
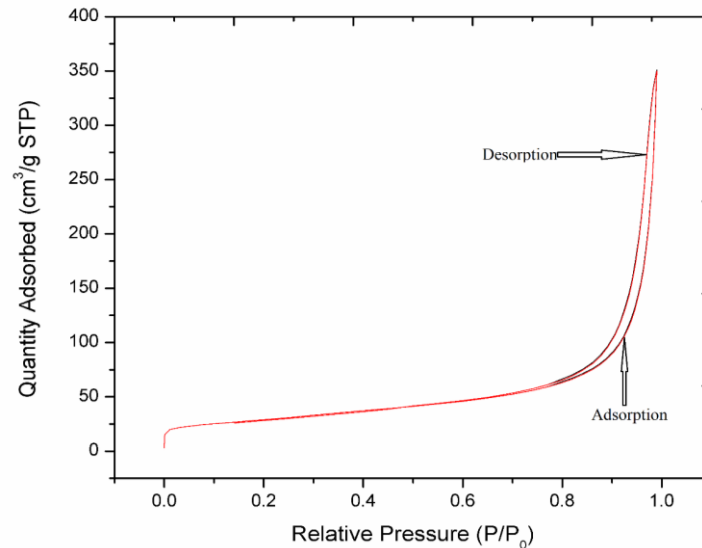
Fig. 7. Adsorption and Desorption isotherms of MWCNT nanoparticles

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

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
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

Table 4
Size of pores measured

Parameters	Pore size (nm)
Average pore width in Adsorption (BET)	16.13422
Average pore width in Desorption (BET)	20.49460
BJH Adsorption average pore width	21.8211
BJH Desorption average pore width	20.8445

3.4 Thermal Conductivity

In order to dissipate heat from thermal systems, fluids with high thermal conductivity are preferred. Propylene glycol with fascinating properties such as incombustibility, non-poisonous, antifreeze, eco-friendly and optimal performance proved to be the most appropriate base fluid for nanofluids [23]. Since propylene glycol is the significant proportionate of the proposed ionanofluid; the measured thermal conductivity of the studied propylene glycol was compared with data from previous literature of ASHRAE [24] and Bejan [25] during the device calibration stage. The studied propylene glycol demonstrated a linear rise in thermal conductivity as a function of temperature. As seen in Figure 8, thermal conductivity improvement was dependent on temperature and resembled the pure propylene glycol behaviour. Thermal conductivity measurements were taken for four different samples of MWCNT nanoparticles dispersed in propylene glycol and ionic liquids (80:20) with a temperature range of 295K to 355K.
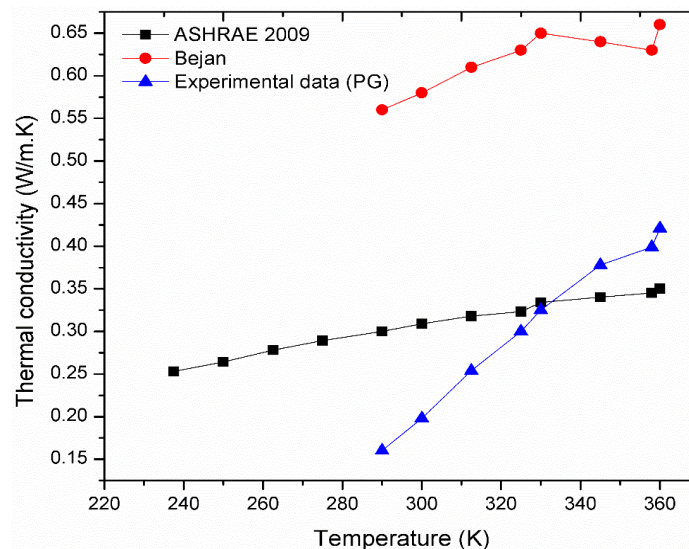

Fig. 8. Thermal conductivity validation of reference fluids with present study

The temperature-dependent thermal conductivity of the formulated nanofluid and ionanofluid is depicted in Figure 9. Results show that the thermal conductivity of ionanofluids increases as the temperature increases, and this increase is more amplified at elevated temperatures. The thermal conductivity rise was seen in MWCNT/PG/[Mmim][DMP] at 355 K, which is 37.5% higher compared to the propylene glycol base fluid. This vital 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 and an increase in energy levels. Other factors that contribute to the thermal conductivity enhancement are nanometer-sized particles, high surface area and weaker molecular bonds. The comparison of thermal conductivity in relation to temperature for both the

measured values and the experimental values from Satti *et al.,* [26] and Dehkordi *et al.,* [27] is depicted in the below figure. There has been a substantial similarity between the calculated values and those derived from previous reports.
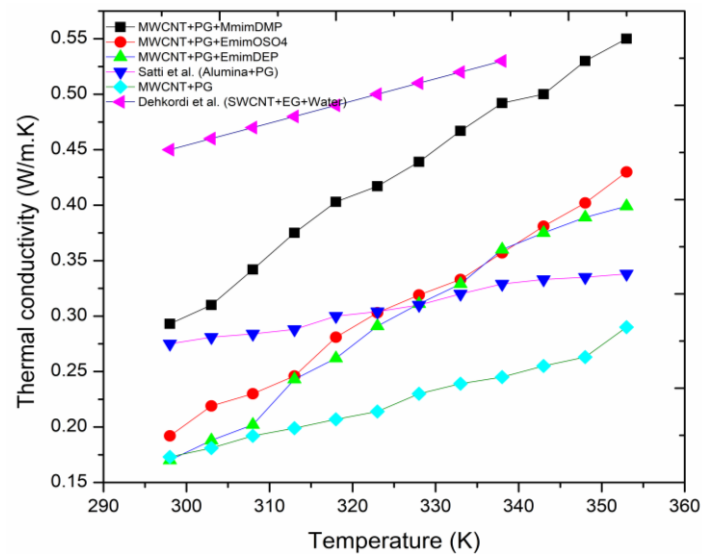

Fig. 9. MWCNT ionanofluids' thermal conductivity at different temperatures

3.5 Stability

Nanoparticles tend to aggregate after a certain period, which 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 has a significant effect on increasing the nanofluid's thermal conductivity. As presented in Figure 10, the visual inspections of the prepared samples show stability for 72 hours except for MWCNT/PG nanofluid, which does not contain any ionic liquid. The ionic liquid-based nanofluids did not show any significant sedimentation or aggregation within the specified time.
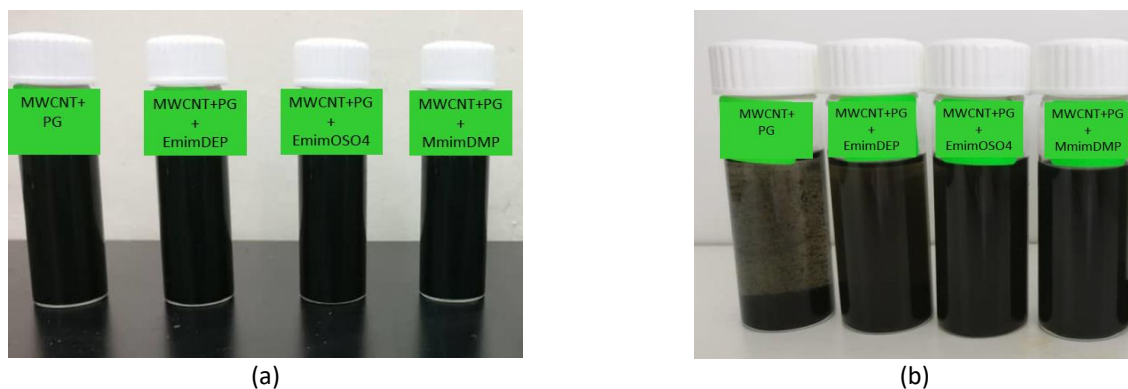

Fig. 10. Photograph of prepared samples (a) Sample after immediate preparation (b) Sample after 72 hours

4. Conclusions

This research analyzed the Porosimetry of Multiwalled carbon nanotubes along with the effect of ionic liquids in MWCNT nanofluids on thermal conductivity and stability with 0.5 wt% mass concentration. Overall, the surface area, pore volume and pore size of the studied MWCNTs ranged from 15 to 121 m^2/g, 0.0077 to 0.54 cm^3/g and 16.13 to 20 nm respectively. From the results, it was found that MWCNT/PG/[Mmim][DMP] showed an utmost 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 thermal conductivity of the nanoparticles in the base fluid depends primarily on the dispersion stability. Moreover, less concentration of ionic liquids ameliorates the samples' thermal conductivity which might help researchers to enhance other thermophysical properties of nanofluids without surfactants.

Acknowledgement

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