



Stability and Thermo-Physical Properties of Ethylene Glycol Based Nanofluids for Solar Thermal Applications

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ABSTRACT

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This article summarizes research involving the evaluation of the thermo-physical properties of ethylene- glycol-based solar thermic fluids oxidized multi-walled carbon nanotubes. Nanofluids were prepared with Ethylene glycol and water as base fluids in 100:0, 90:10 and 80:20 ratios. Base fluids of three categories were dispersed with surfactant-assisted multi-walled carbon nanotubes (MWCNTs) and oxidized MWCNTs in the weight fractions of 0.125, 0.25, and 0.5 percentages to check the influence of surface modification technique on the thermophysical properties. The variation in zeta potential is studied to examine the dispersion stability during 2 months. Thermal conductivity and dynamic viscosity were measured by hot disk method and Anton paar viscometer, respectively. Significant enhancement of thermal conductivity by 15 to 24 % was observed when the base fluids are dispersed with oxidized MWCNTs. In the case of nanofluids dispersed with surfactant-assisted MWCNTs, the improvement is significantly less compared to oxidized MWCNTs. Nanofluids' dynamic viscosity is found to be higher compared to base fluids in the temperature range of 50 to 70 °C. A comprehensive mathematical equation suitable for all weight fraction of MWCNTs and volume percentages of Ethylene glycol was developed, which can forecast the temperature range. The correlation could fit well with the experimental data in reasonable limits.

1. INTRODUCTION

Over past decades, numerous studies have shown that nanofluids can be used in multiple fields; For instance, heat exchangers, electronic cooling, tribology, engine cooling systems, and solar thermal systems. Their use could boost the heat transfer capability of industrial equipment. The nanofluid is characterized as a mixture of one or more diverse types of nanomaterials in a base fluid. Currently, several types of nanomaterials are being studied by researchers [1-24] for dispersing in base fluids. Nanomaterials currently employed by researchers have classified as 1) carbonaceous materials with a very high thermal conductivity such as graphene, CNTs, diamond etc. 2) metallic nanoparticles like aluminium (Al), Copper (Cu), gold (Au), iron (Fe), silver (Ag), etc. 3) metal oxides or compounds such as copper oxide (CuO), Alumina (Al₂O₃), titania (TiO₂), zinc oxide (ZnO), silicon carbide (SiC), silica (SiO₂) etc

Ethylene glycol – water mixtures can be used as thermic fluids for solar water heating. These fluids can also be used as residential baseboard heaters, heat exchangers, and industries. Ethylene glycol has the advantage of possessing tunable properties by way of diluting with water, thus varying the thermo-physical properties to suit applications. The thermophysical properties of ethylene – glycol water mixtures for use as thermic fluids are shown in Table 1.

Several studies were conducted to assess carbon nanotubes' effect on the thermophysical properties of nanofluids. All researchers witnessed that with a rise of volume fraction, there

is a surge in nanofluid thermal conductivity, density, and viscosity while the specific heat decreased. Also, the thermal conductivity and specific heat improved with the increase of temperature.

Table 1. Properties of mono-ethylene glycol – water mixtures for use as thermic fluids

Thermo-physical properties	Mono-ethylene glycol– water (80:20)	Mono-ethylene glycol– water (90:10)	Mono-ethylene glycol– water (100)
Freezing point, °C	-12.22	-28.88	-43.33
Boiling point, °C	122.22	133.4	194.44
Viscosity, cP (at room temperature)	4.608	6.422	9.090
Specific heat, kJ/kg K (at room temperature)	3.14	2.93	2.72
Thermal conductivity, W/m K (at room temperature)	0.328	0.259	0.225
Density, kg/m ³ (at room temperature)	1085	1090	1100

All researchers witnessed that with a rise of volume fraction, there is a surge in nanofluid thermal conductivity, density and viscosity while the specific heat decreased. Also, the thermal conductivity and specific heat improved with increase of temperature. On the contrary, the viscosity and density were observed to have reduced as temperature increases. The major deficiency in the studies of most of the researchers is the surface modification technique and use of surfactant which would result in the formation of copious amount of foam.

1.1 Present studies

Most of the researchers did not emphasize the issue of nanofluids' stability, which is the key task of using nanofluids is the low stability of the suspensions. The major deficiency in most of the researchers' studies is the surface modification technique and use of surfactant, which would result in the formation of a copious amount of foam. Surface functionalization of MWNTs utilizing acid treatment produces functional groups over the surface of CNTs improving ethylene glycol's solubility. The novelty of the present work is the evaluation of the stability of prepared nanofluids for two months. A comparison of nanofluids dispersed with surfactant-assisted MWCNTs and oxidized MWCNTs in terms of thermal conductivity enhancement is made. Unlike previous the studies, the present studies are intended for application of EG- Water mixtures as thermic fluids. Hence, the ratio of Ethylene glycol water mixtures is taken as 100%, 90% and 80% with the boiling point ranging from 120 to 194°C. Dynamic viscosity and thermal conductivity is evaluated in the temperature range of 50 to the near boiling point of the EG – water mixtures. Comprehensive correlations were predicted for Dynamic viscosity and thermal conductivity in terms mass fraction of MWCNTs, percentage of water, temperature.

2. MATERIALS AND METHODS

2.1 Materials

Multi-walled Carbon Nanotubes prepared using CVD process was purchased from M/s Cheaptubes Inc., USA. The MWCNTs are highly entangled with a purity of 95 % and are of 30–50 nm diameter & 3-15 μm length. All other chemicals and surfactant used Cetrimonium Bromide (CTAB) are purchased from M/s Sigma Aldrich are analytical grade.

2.2 Surface modification of multi-walled carbon nanotubes

Several studies have established that dispersion and stability are vital features in improving nanofluids' thermophysical properties, particularly for thermal conductivity. The dispersion stability of the nanofluids evaluated the final properties of nanofluids. Pristine multi-walled carbon nanotubes are incredibly hydrophobic and are not dispersible in polar solvents like water, Ethylene glycol, alcohols etc. Due to hydrophobicity, nanotubes clusters would form due to agglomeration and would settle down in the liquid medium resulting in loss of properties. The common practice of the researchers is to use a surfactant to disperse CNTs in base fluids. Surfactants escalate the foaming tendency of fluids resulting in decreased heat transfer rates.

Chen et al. [1] and Hou et al. [3] showed that the pristine carbon nanotubes, when purified, become high-quality carbon

nanotubes with open ends. Studies of Rosca et al. [4], Vaisman et al. [5] and Chiang et al. [6], demonstrated that acid treatment is the most appropriate method to purify and create polar groups on the surface of the MWCNTs to make them hydrophilic.

In the present studies, the multi-walled carbon nanotubes were surface modified with two techniques 1) surfactant-assisted surface modification and 2) oxidation of MWCNTs to produce carboxyl and carbonyl groups on the surface. In the first method, a surfactant Cetrimonium Bromide (CTAB) and MWCNTs are taken in equal weight ratios and dispersed in solvent methanol using an ultra-sonicator for 10 minutes. The solvent and allowed to evaporate, and the resultant surfactant-assisted MWCNTs were recovered. For oxidation of MWCNTs, the nanotubes are refluxed in a mixture of 4 Molar H₂SO₄ and 4 Molar HNO₃ (3:1 volume ratio) for 3 hours. After reflux, the residue was thoroughly washed with distilled water till seven pH and dried over-night in an oven at 60°C.

The processed MWCNTs were characterized using FESEM for structure and Fourier Transform infrared spectroscopy for the occurrence of functional groups. Figures 1a & 1b shows the HRSEM image of pristine and oxidized CNTs. From Figure 1b shows that the purification process has disentangled the MWCNTs and could open the tips.

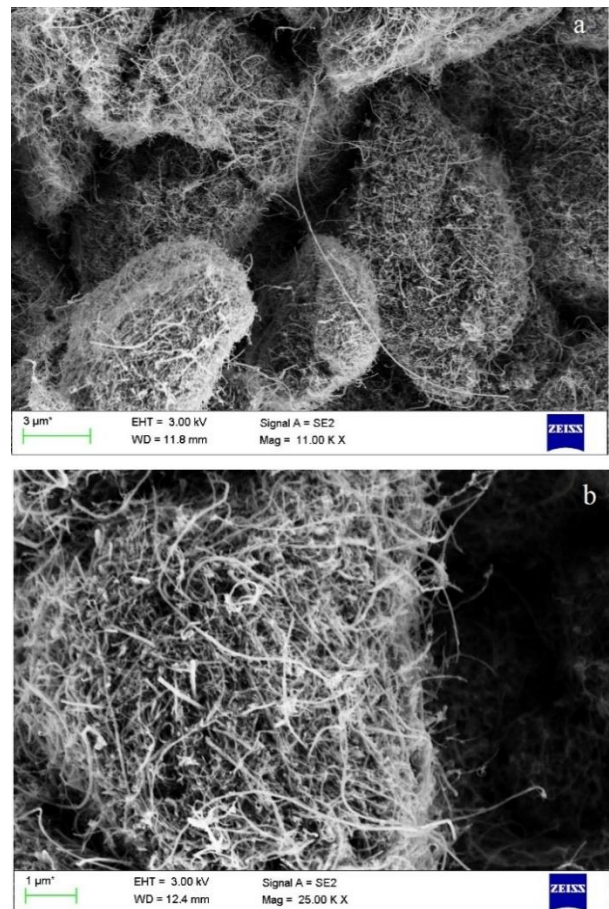


Figure 1. FESEM Image of a) as-received multi-walled carbon nanotubes b) oxidized multi-walled carbon nanotubes

Figure 2 shows the FTIR Spectrum of pristine MWCNTs and MWCNTs oxidized with acid mixtures. Unlike pristine MWCNTs shown in Figure 2a, the modified MWCNTs in Figure 2b display spikes on the spectrum at 1125 and 1740 cm⁻¹, indicating carbonyl and carboxyl groups formed on the MWCNTs. These hydrophilic groups create MWCNTs that

are highly dispersible in water. High dispersibility is also a precursor for property enhancement compared to unstable nanofluids. The stability measurement by zeta potential is discussed in the next section.

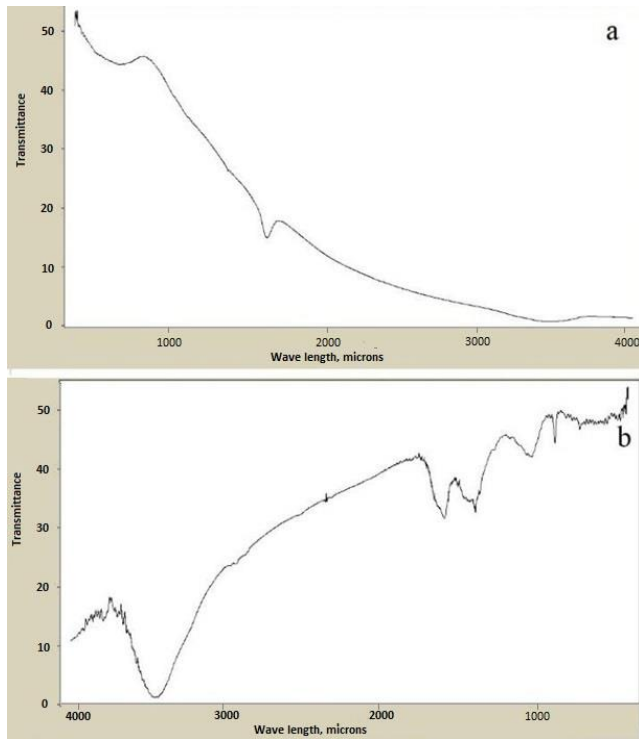


Figure 2. FTIR analysis of a) Pristine MWCNTs b) oxidized MWCNTs

2.3 Preparation of base fluids and nanofluids

Table 2. Coolant configuration of base fluids

S. No	Composition
1	100 % Ethylene glycol
2	90 % Ethylene glycol+10% distilled water
3	80 % Ethylene glycol+20% distilled water

Table 3. Nanofluid configuration

S. No	Composition
1	100 % Ethylene glycol+ (0.125, 0.25 and 0.5 %) surfactant assisted MWCNTs
2	90 % Ethylene glycol+10% distilled water+(0.125, 0.25 and 0.5 %) surfactant assisted MWCNTs
3	80 % Ethylene glycol+20% distilled water+ (0.125, 0.25 and 0.5 %) surfactant assisted MWCNTs
4	100 % Ethylene glycol+ (0.125, 0.25 and 0.5 %) oxidized MWCNTs
5	90 % Ethylene glycol+10% distilled water+(0.125, 0.25 and 0.5 %) oxidized MWCNTs
6	80 % Ethylene glycol+20% distilled water+ (0.125, 0.25 and 0.5 %) oxidized MWCNTs

3 types of thermic fluids with the composition of as given below are selected in the current study. The details of base fluids and nanofluids are shown in Tables 2 and 3.

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than ten, including all the sections. Please make sure that the whole text ends on an even page. Please do not insert page numbers. Please do not use the Headers or the Footers because they are reserved for the technical editing by editors.

3. STABILITY OF NANOFLUIDS

Colloidal fluids are assessed for stability through variation of zeta potential of the fluid. The zeta potential specifies the amount of repulsion of charged particles (OH⁻, COOH⁻ groups existing in MWCNTs and the base liquid). A threshold value of ±20 specifies developing stability of the solution and values exceeding 25 and under -25 indicate good nanofluids' good stability. Ethylene glycol's zeta potential–water mixtures mixed with oxidized MWCNTs is appraised to assess their stability over 60 days.

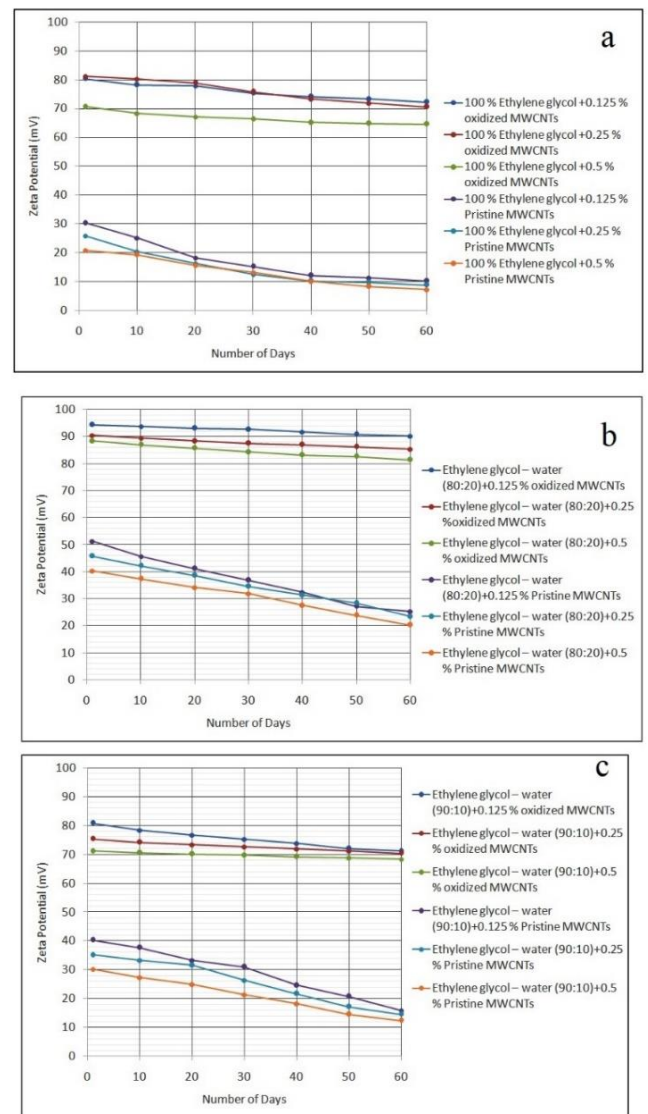
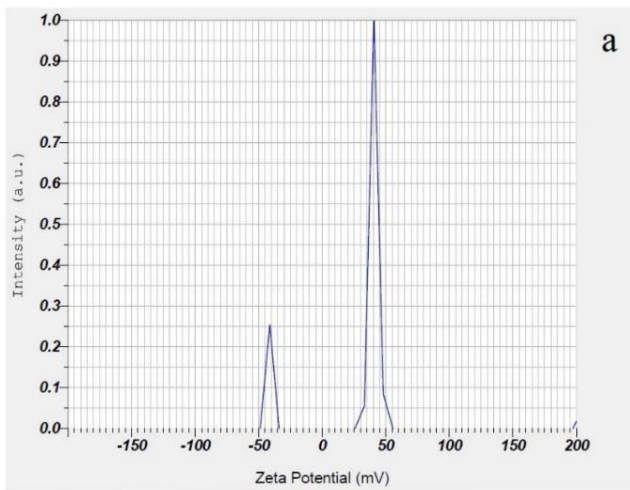


Figure 3. Zeta potential variation of nanofluids with pristine and oxidized MWCNTs for two months

All the nanofluids are tested for stability for two months, and the results are as shown in Figure 3 for the varying composition of ethylene- glycol – water mixtures. The results in Figure 3 conclude that compared to pristine MWCNTs, the surface-modified MWCNTs could remain more stable in the

coolants. There is a massive difference in the zeta potential from the first day to 60th day in the case of coolants dispersed with pristine MWCNTs. With the dispersion of oxidized MWCNTs, the zeta potential difference over two months is minimal, indicating high stability of the suspensions. This enhanced stability is due to carbonyl and carboxyl groups on the MWCNTs which link with polar groups of Ethylene glycol and water molecules allowing MWCNTs to remain stable in the nanofluids.

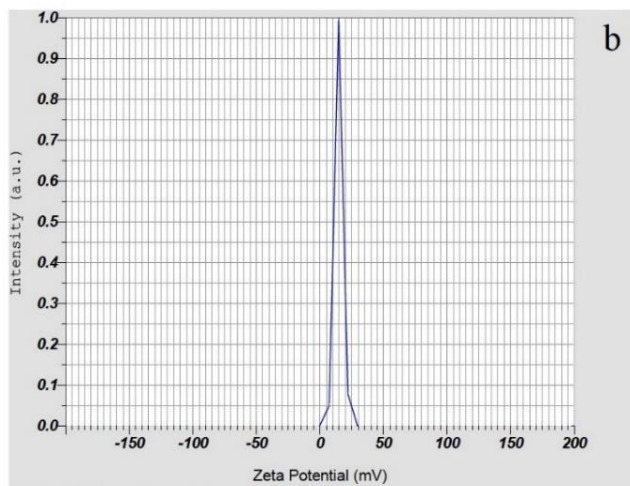
Further, Figures 4 & 5 show the values of zeta potential of selected coolants. Figure 4 shows the zeta potential of nanofluids dispersed with pristine MWCNTs and it can be seen that the zeta potential is very low on the first day as well as after two months. In Figure 5, which shows the zeta potential variation of nano fluids from the first day to the 60th day, dispersion of oxidized MWCNTs resulted in excellent stability.



Calculation Results

Peak No.	Zeta Potential	Electrophoretic Mobility
1	41.0 mV	0.000318 cm ² /Vs
2	-41.0 mV	-0.000318 cm ² /Vs
3	---	---

Zeta Potential (Mean) : 20.8 mV
Electrophoretic Mobility mean : 0.000161 cm²/Vs

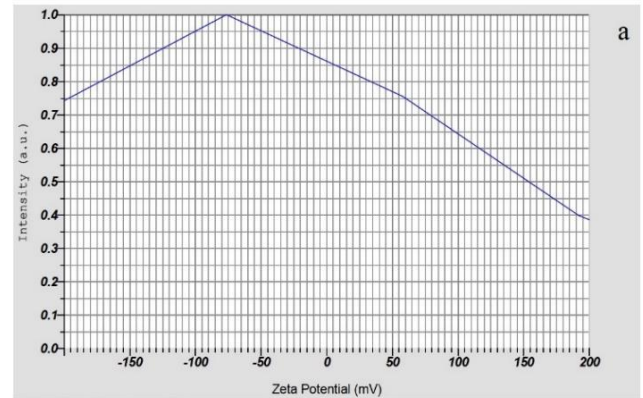


Calculation Results

Peak No.	Zeta Potential	Electrophoretic Mobility
1	15.0 mV	0.000116 cm ² /Vs
2	---	---
3	---	---

Zeta Potential (Mean) : 15.0 mV
Electrophoretic Mobility mean : 0.000116 cm²/Vs

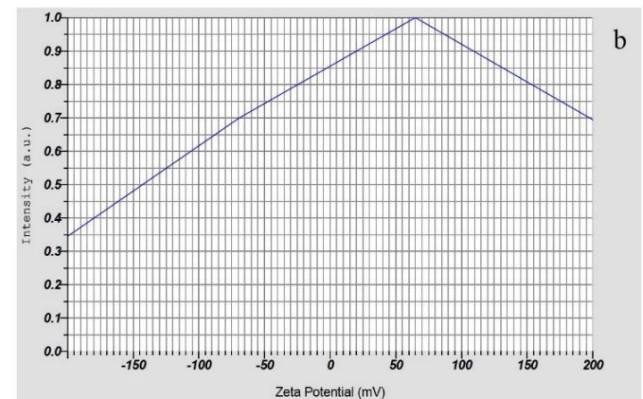
Figure 4. Zeta potential of Ethylene glycol – water mixture (90:20) dispersed with pristine MWCNTs a) first day b) after two months



Calculation Results

Peak No.	Zeta Potential	Electrophoretic Mobility
1	-70.6 mV	-0.000027 cm ² /Vs
2	---	---
3	---	---

Zeta Potential (Mean) : -70.6 mV
Electrophoretic Mobility mean : -0.000027 cm²/Vs



Calculation Results

Peak No.	Zeta Potential	Electrophoretic Mobility
1	64.5 mV	0.000024 cm ² /Vs
2	---	---
3	---	---

Zeta Potential (Mean) : 64.5 mV
Electrophoretic Mobility mean : 0.000024 cm²/Vs

Figure 5. Zeta potential of Ethylene glycol – water mixture (90:20) dispersed with oxidized MWCNTs a) first day b) after two months

4. THERMO-PHYSICAL PROPERTY EVALUATION

Evaluation of Thermophysical properties Viz., thermal conductivity, specific heat and dynamic viscosity are very important for assessing thermic fluids' effectiveness. The evaluation of liquids' thermal conductivity is a challenge due to the predominant convective heat transfer in fluids during the measurement. In present studies, thermal conductivity is measured by the Hot Disk method which eliminates errors while measuring liquid thermal conductivity. Kapton sensor 7577 was selected for testing while keeping a short measurement time to reduce convection. Three sets of experiments with different measurement times were conducted on the samples and the average values are reported.

To measure dynamic viscosity ANTON PAAR MCR 302 Rheometer is used for fast and accurate strain control of fluids resulting in very accurate measurements. The samples are tested at different temperatures in three trials and average values are taken. The sample is ensured to be free from air and loaded in the rheometer and the values were noted at steady-state temperature conditions.

All the samples are measured for dynamic viscosity and thermal conductivity in the temperature limits from 50 to near boiling point.

5. RESULTS AND DISCUSSIONS

Figure 6 depicts the variation of thermal conductivity of solar thermic fluid Ethylene glycol - water mixtures with temperature for varying percentage of water and weight fraction of multi-walled carbon nanotubes. It can be found that there is a lessening of thermal conductivity as the temperature increases. For the cases of Ethylene glycol - 10% Water and Ethylene glycol - 20% water, there is an initial spike in thermal conductivity followed by a decrease when the temperature increased. The fluids dispersed with MWCNTs performed well in cases of all weight fractions and all percentages of water.

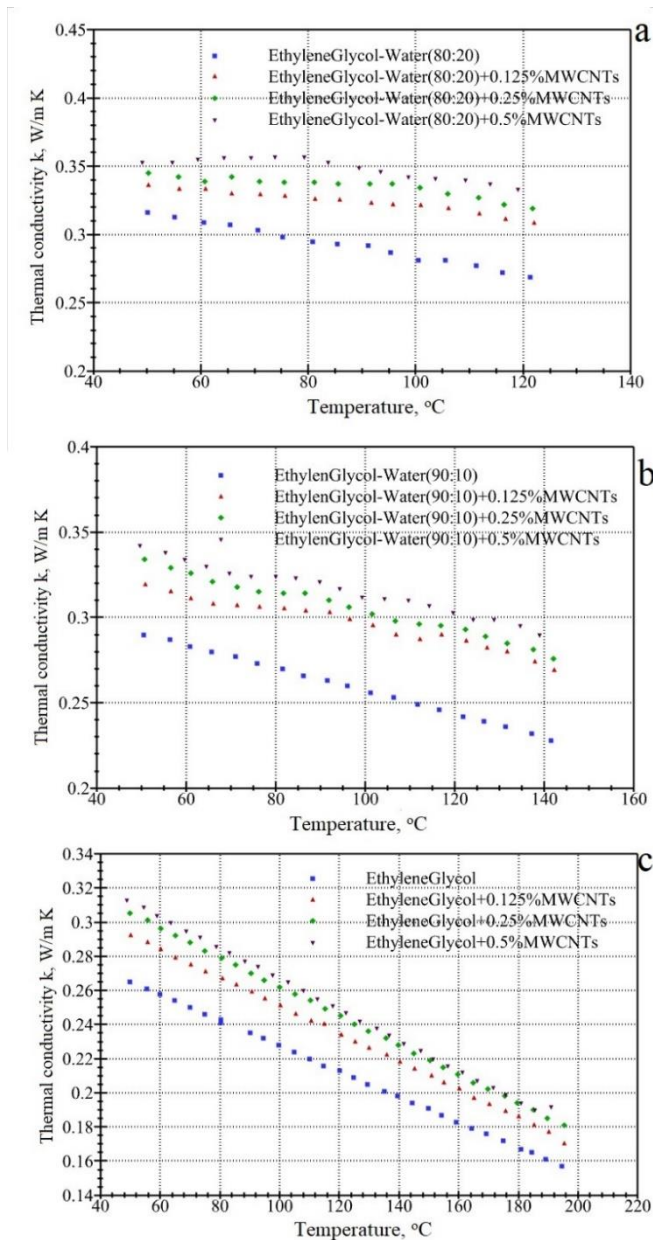


Figure 6. Variation of thermal conductivity with temperature for different Ethylene glycol –water mixture concentrations

The results established that a higher percentage of MWCNTs resulted in higher TC of the nanofluids, as can be perceived from the below figure. The TC of nanofluids for all percentages of water shown significant improvement compared to corresponding base fluids. Furthermore, it can be established that the 80% Ethylene glycol– 20% water

combination has shown the highest thermal conductivity increase compared to all other fluids. Meanwhile, pure Ethylene glycol displayed a slightly low enhancement in TC. The influence of temperature on thermal conductivity improvement is also noteworthy. The improvement in TC is more at higher temperatures. The maximum percentage improvement for pure Ethylene glycol, Ethylene glycol – 10% water mixture and Ethylene glycol – 20% of water mixtures is 18%, 24% and 26%.

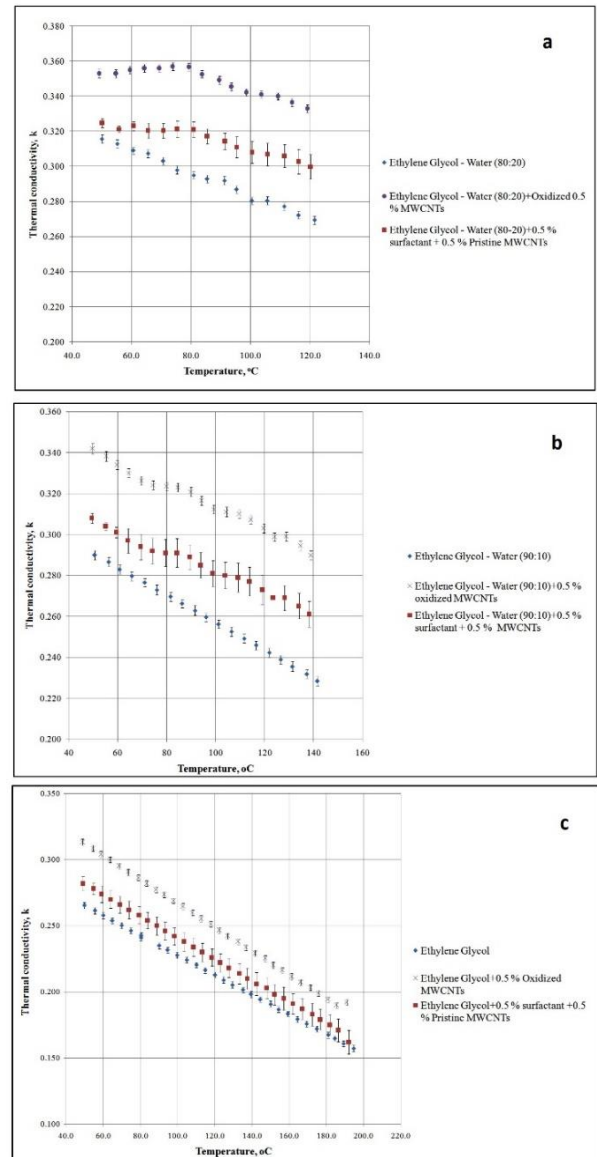


Figure 7. Comparison of thermal conductivity of nanofluids dispersed with surfactant-assisted MWCNTs and oxidized MWCNTs

In Figure 7, a comparison of thermal conductivity enhancement of nanofluids dispersed with surfactant-assisted MWCNTs and oxidized MWCNTs. It is found that surfactant-assisted MWCNTs could not much improve the thermal conductivity compared to oxidized MWCNTs at the higher temperature (above 70°C) due to the formation of foaming. Further, the instrumentation error is more during the measurement due to the intervention of bubbles in the foam. In all, taking into account the error in instrumentation, the effect of dispersion of surfactant-assisted MWCNTs is negligible.

Figure 8 displays the deviation of dynamic viscosity of different nanofluids with temperature. Figure 8a shows the variation of dynamic viscosity for Ethylene glycol – 20% water mixture in the temperature range of 50 to 125°C. Figure 8b shows the variation of dynamic viscosity for Ethylene glycol – 10 % water mixture range of 50 to 150°C and Figure 8c show the change of dynamic viscosity of pure Ethylene glycol in the temperature limits of 50 to 175°C. This disparity in maximum temperature is due to the decrease of boiling point with the addition of water. The dynamic viscosity of all fluids shows a decreasing trend with an increase of temperature. A common characteristic with nanofluids is an evident increase in dynamic viscosity with the dispersion of MWCNTs compared to base fluids. The improvement is substantial in nanofluids' dynamic viscosity compared to the equivalent base fluids at lower temperatures.

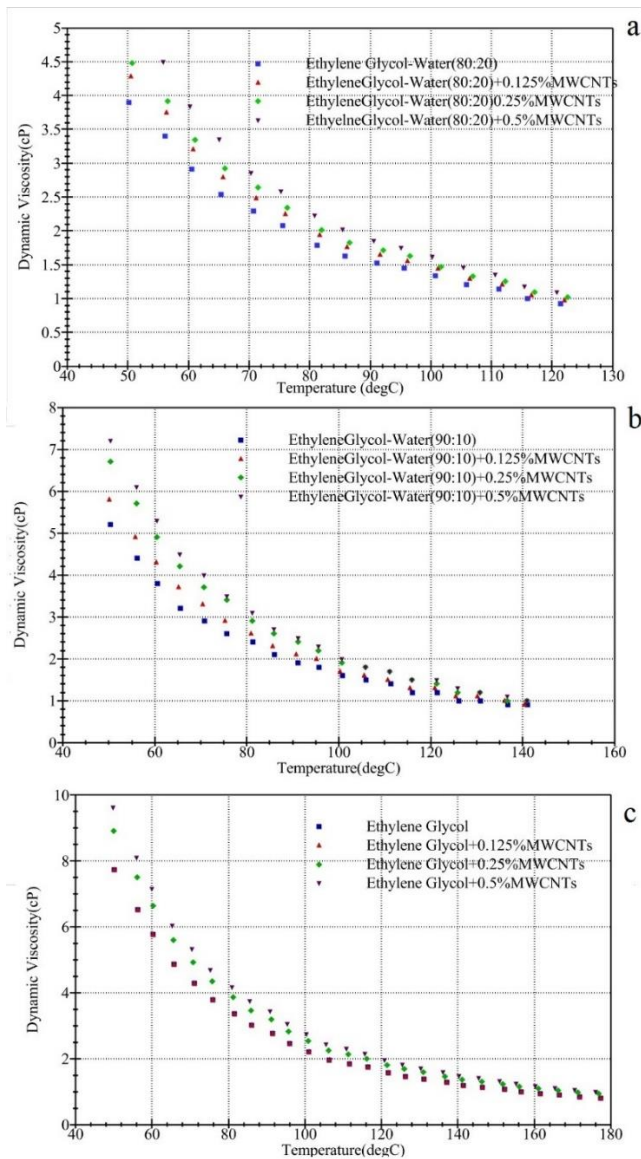


Figure 8. Variation of Dynamic Viscosity with temperature for different Ethylene glycol –water mixture concentrations

5.1 Regression analysis

The data is taken to a statistical tool, and regression of experimental data points is done with dependent variables as temperature, percentage of ethylene glycol and the

concentration of the MWCNTs. The experimental values of thermal conductivity and dynamic viscosity were separately examined to develop the property assessment regression equations. The equations developed are as shown in Eqns. (1) and (2).

$$\frac{k_{nf}}{k_b^{0.75}} = 1.25 \left[1 + \frac{T}{T_{max}} \right]^{-0.18} (1 + \phi)^{0.4} \left(1 + \frac{\alpha}{100} \right)^{-0.7} \quad (1)$$

$$\frac{\mu_{nf}}{\mu_b^{0.9}} = 1.303 \left[1 + \frac{T}{T_{max}} \right]^{-0.7} (1 + \phi)^{0.7} \left(1 + \frac{\alpha}{100} \right)^{0.25} \quad (2)$$

The equations could forecast the thermal conductivity and viscosity of Ethylene glycol – water based solar thermic fluids in volume percentage from 80 to 100% and MWCNTs weight percentage between 0.125 to 0.5%.

5.2 Validation of experimental values predicted from correlations

Figure 9 shows the plot with values predicted from equation 1 for thermal conductivity juxtaposed with the experimental data. Figure 10 display the plot with predicted values of correlation equation 2 for dynamic viscosity contrasted with the experimental data. The outcomes revealed that the predicted values of correlation were in good agreement with the experimental data. It can also be found that correlation can forecast both the thermal conductivity and viscosity of nanofluids.

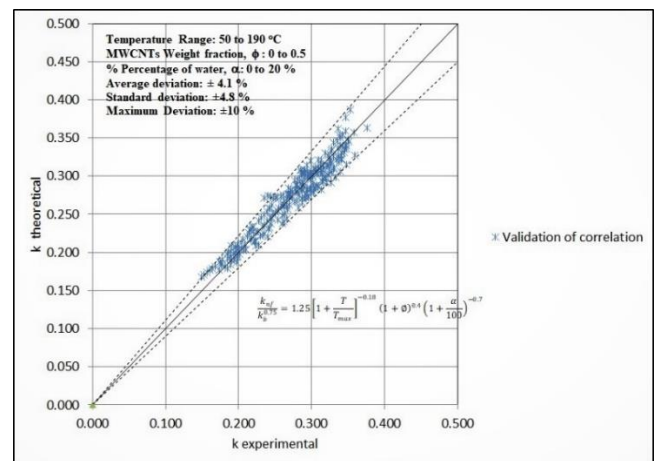


Figure 9. Validation of correlation equation developed for predicting thermal conductivity

Figure 9. depicts the validation of correlation for thermal conductivity proposed in Eq. (1). The proposed Eq. (1) fits the data with an average deviation of 4% and standard deviation of 4.80%. The equation is in agreement with the current experimental data and within ±10% deviation. The equation is valid in the range of $0 \leq \phi \leq 0.5\%$ and $50 \leq T \leq 190^\circ\text{C}$.

Figure 10 shows the validation of correlation for dynamic viscosity proposed in Eq. (2) and could fit the data with an average deviation of 5.1% and standard deviation of 6.3% and all experimental data is within ±11% deviation. The Eq. (2) is valid in the range of $0 \leq \phi \leq 0.5\%$ and $50 \leq T \leq 150^\circ\text{C}$.

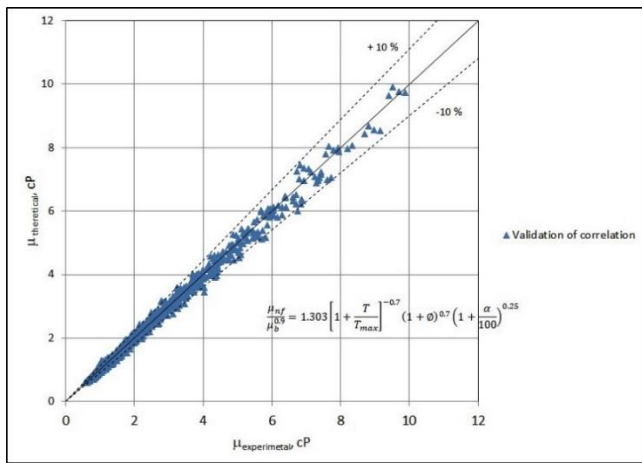


Figure 10. Validation of correlation equation developed for predicting dynamic viscosity

6. CONCLUSIONS

The following conclusions can be made from the study.

- 1 The functionalization of MWCNTs could graft carboxyl and carbonyl groups on their surface, which makes them stable in Ethylene glycol–water mixtures for longer periods compared to pristine MWCNTs.
- 2 The zeta potential analysis of nanofluids revealed that oxidized MWCNTs could remain stable in Ethylene glycol–water mixtures compared to pristine MWCNTs.
- 3 Dispersion of oxidized MWCNTs improved the thermal conductivity in the range of 10 to 20% for all the Ethylene glycol –water ratios.
- 4 The influence of the surface modification technique on thermal conductivity enhancement is found to be significant. The thermal conductivity enhancement of nanofluids dispersed with oxidized MWCNTs is very high compared to nanofluids dispersed with surfactant-assisted MWCNTs.
- 5 Nanofluids' dynamic viscosity is found to be higher compared to base fluids in the temperature range of 50 to 70°C. Nevertheless, at higher temperature ranges, there is minimal variation in viscosity of base and nanofluids.
- 6 A comprehensive correlation for thermal conductivity and viscosity has been developed which could predict in all temperature and weight fraction ranges.

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