DISPERSION STABILITY AND RHEOLOGICAL CHARACTERISTICS OF WATER AND ETHYLENE GLYCOL BASED ZINC OXIDE NANOFLUIDS

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With advancement of nanoscience, "nanofluids" are becoming quite popular among thermal engineers. High thermal conductivity, relatively less settling speed, and higher surface area of nanoparticles are a few key promoting properties. The last two decades have seen dramatic progress towards using nanoparticles in industrial applications. However, the stability and rheological characteristics of prepared nanofluids have serious effects on their transport characteristics, but unfortunately, this has not found proper attention from researchers. In this study, stability and rheological characteristics of ZnO nanoparticles within deionized water, ethylene glycol, and their blends have been extensively tested. Stability was observed using UV-vis spectroscopy, while the viscosity was measured with the help of a rheometer. The data was collected with 0.011-0.044 wt. % loading of nanoparticles, while experiments were conducted within 15-55°C temperature range. Better stability was recorded when nanofluids were prepared with pure ethylene glycol. Experiments showed that the viscosity increased with particle loading, whereas the effect of surfactants appeared to be insignificant. Research results were used to assess predictions of different viscosity models. Experimental data was overpredicted by Einstein, Brinkman, and Batchelor's models.

Key words: *Ethylene glycol, Deionized water, Nanoparticles, Nanofluid, Stability, Viscosity, Zinc oxide*

1. Introduction

High-tech industries like microelectronics, transportation, manufacturing, and defense are facing a huge challenge in the field of cooling. The heat dissipation quest is rapidly increasing in microelectronics. Heat transfer intensification is needed to fulfill the demands of modern high-tech electronic devices [1]. Thermophysical properties of the working medium have a significant impact on fluid flow and thermal characteristics [2]. Nanofluids (NFs), a new kind of heat transfer medium and consisting of dilute dispersion of nanometer-sized metallic and non-metallic particles in engine oil, ethylene glycol (EG), deionized water (DIW) and distilled water (DW), have gained significant recognition in the present era [3][4]. The potential use of NFs in solar energy, refrigeration systems [5], medical applications [6], chemical reactions, and automobiles [7] has attracted the attention of many researchers.

Nanoparticles (NPs), dispersed in different working mediums, exhibit quite different properties, and the resulting NFs are supposed to show enhanced thermal and heat transfer characteristics than the host base fluids [8][9]. The thermal conductivity of NFs prepared with various base fluids has been widely investigated; however, other critical factors like dispersion stability, morphology, and rheological behaviour are less investigated and reported [10]. The use of NPs for heat transfer enhancement increased the viscosity of the working medium under consideration [11]. NFs are considered stable when the NPs are well dispersed in base fluid and do not tend to aggregate at a significant rate. NPs are prone to hard aggregate due to their large surface area and high surface energy. The stability of the nanofluid is a complex phenomenon and depends on various parameters (particle size, Brownian motion, Van der Waal forces, particle sedimentation, thermodynamically instability, etc.). As particles get aggregated, hydrodynamic size, morphology, and concentration changes, and all this has a significant effect on the thermophysical properties of NFs [12]. Particle agglomeration also results in choking heat transfer systems and degradation in overall thermal performance. Every attempt should be exercised to make stable fluid to overcome foretasted consequences [13][14].

NPs of metal oxide materials such as Al_2O_3, CuO, SiO_2, TiO_2, and ZnO normally exhibit higher thermal conductivity than base fluids even in very dilute concentrations [15]. The utilization of such mediums is normally reported to enhance heat transfer performance [16]. Although the thermal conductivity of other metallic and non-metallic materials such as Al, Ag, Cu, SiC, CNT, SWCNT, MWCNT, and graphene is higher than those of metal oxides, the later are preferred due to their greater resistance to oxidation and less particle sedimentation due to lower density [17]–[20].

Stability of water-based ZnO and CuO NFs (0.1-0.5 wt. %) have been reported by Ponmani et al. [21] using xanthan gum (0.4 %) as a surfactant. Experimental investigations showed an increase in the average hydrodynamic size of NPs from 500-2000 nm for ZnO NFs and 250-1000 nm for CuO NFs. An improvement of 53.0 % in thermal conductivity has been recorded in the case of 0.5 wt. % ZnO nanofluid. Sughanti and Rajan [22] investigated the stability of 0.2-2.0 vol. % ZnO/DW NFs in 25-55°C temperature range using probe and bath sonication. Sodium hexametaphosphate (SHMP) was used as a stabilizing agent. The authors recorded a reduction in the hydrodynamic size of aggregates for an ultrasonication period of up to three hours. This bigger size was speculated due to the formation of loose aggregates after the surfactant was partially desorbed. The viscosity of NFs was also observed to be increased with the formation of these agglomerates. Raykar and Singh [23] stabilized aqueous ZnO NFs using acetylacetone at particle loading of 0.075-0.50 wt. %. They reported an enhancement in the dispersion stability of NFs for up to one year, while thermal conductivity enhanced up to 40 % for 0.5 wt. % of NPs at 30 °C. In another study [24] enhanced stability of ZnO/DW NFs using SHMP as a surfactant was observed in a pH range of 8.58-9.38 and at surfactant to NPs ratio of 1:2 and 1:6, respectively. Gallego [25] investigated the variation in thermal conductivity, viscosity, and density of ZnO/EG solutions in a volume concentration of 1.0 and 6.2 % and at a temperature range of 10-60 °C. Viscosity measured for volume concentration up to 4.7 % confirmed the Newtonian behaviour of the NFs.

While many metal oxide NPs have been widely studied, to the best of the author's knowledge from the review of the contemporary literature, the stability and rheology of ZnO based NFs have not been extensively investigated and reported, especially in dilute concentrations. The objective of current research is to investigate the dispersion stability and rheological characteristics of ZnO based NFs in deionized water, ethylene glycol, and their blends with special attention to their application in microelectronic devices generating high heat flux. Acetylacetone was used as a stabilizing agent.

2. Materials and methods

For work reported in this study, ZnO nanoparticles were procured from Sigma-Aldrich® with manufacturer defined particle size less than 100 nm, purity > 97% and of spherical shape. Acetylacetone was also purchased from the same source and used as a stabilizing agent. Deionized water was purchased from the local market. Ultrasonication bath (Elmasonic EASY 30 H, Germany) was used for the dispersion of nanoparticles in the base fluid. X-Ray Diffraction (XRD) (STOE & Cie GmbH, Germany), was used to observe the crystal structure of nanoparticles, scanning electron microscope (SEM) (TESCAN, VEGA3, Czech Republic) was used to explore the shape and size of nanoparticles whereas Fourier Transform Infrared (FTIR) (Perkin Elmer Spectrum Two spectrometer) was used to investigate the identity of nanoparticles in the base fluids.

2.1. Preparation of nanofluids

The preparation of stable NFs is the first step in any nanofluid based application. The main focus of sample preparation is to break up flocs and disperse the particles in different combinations. In the present study, DIW [26] and EG [27] were used as a base fluid for the preparation of NFs. Water is commonly used heat transfer fluid [HTF]; however, its transport properties restrict its utilization for approaching sub-zero temperature conditions as it freezes at 0^{o}C under standard atmospheric pressure conditions [28]. EG is an important organic compound and is used in diverse applications, including energy, plastics, automobiles, and chemicals industry. As an HTF, it is frequently used in car radiators, liquid-cooled computers, chilled water air conditioning systems, as an industrial coolant for gas compressors, and ice-skating rinks. Since water is readily available with comparable properties; however, the same is limited in its applications specifically for sub-zero conditions, anti-freezing agents like EG are therefore mixed with water for coping requirements of cold climatic conditions [29]. It has also been observed that ZnO NPs are highly unstable in pure DIW compared to EG. The dispersion of NPs within EG and its blends with DIW are supposed to enhance the stability of NFs. Additionally, the same will also improve their possible application for very cold climatic conditions.

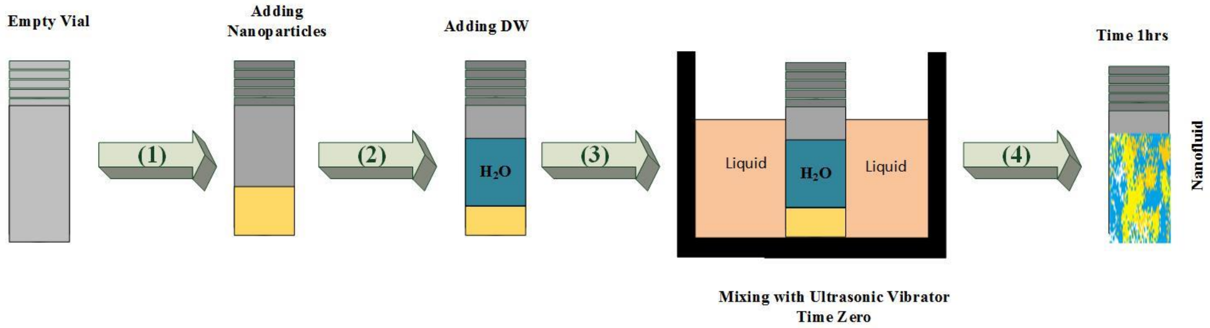


Figure 1. Schematic of nanofluids preparation using two-step method

The ZnO based NFs were prepared using the two-step method in mass concentration of 0.011-0.044 wt.% in DIW, EG, and different blends of DIW and EG. The DIW and EG blends were prepared in the ratio of 80:20, 60:40, 50:50, 40:60, 20:80. The weight of the particles was measured with a highly precise and calibrated analytical balance (Readability = 0.01 mg, Shimadzu AUW220D). An ultrasonication bath (37 kHz, Elmasonic E30H, Germany) was used for the better dispersion of NPs [30]. Ultrasonication is the most accepted physical process to break the NPs aggregates to enhance their stability in prepared NFs [31].

Figure 2. ZnO nanoparticles and ZnO/DIW nanofluids

The NFs were sonicated up to one hour, and acetylacetone was used as a stabilizing agent with DIW based NFs. A schematic of the two-step method is shown in Fig. 1. The original samples of ZnO NPs and NFs prepared in DIW are shown in Fig. 2.

2.2. Characterization of ZnO nanoparticles

An X-ray diffractometer (XRD) was used to examine the crystal structure of ZnO NPs with Cu Kα radiation ($\lambda = 1.5406$ Å) and Bragg angle (2θ) set from 20 to 80^o. Bragg's law measures the angle (θ) between the incident X-ray beam and the crystallographic reflecting plane. Whereas, 2θ is the angle between the transmitted X-ray beam and the reflected beam.

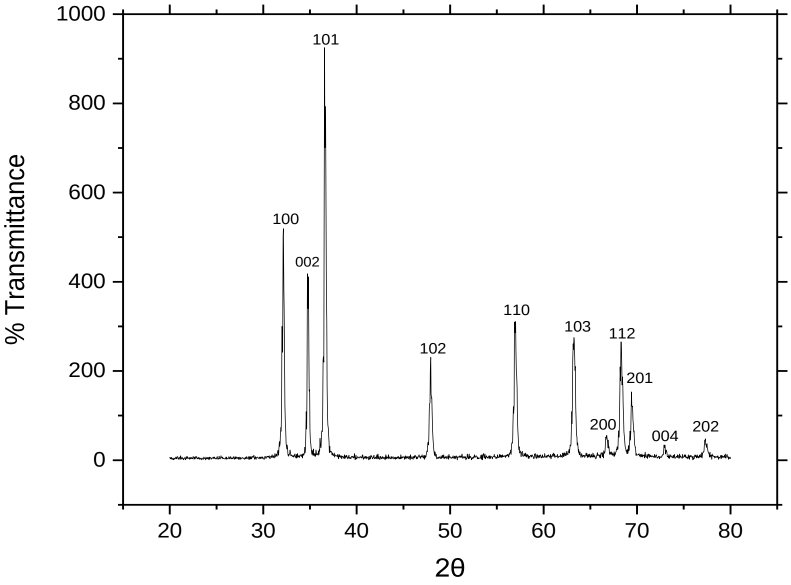


Figure 3. X-ray diffraction pattern of ZnO nanoparticles

The profile of the structure obtained from XRD for as received NPs is shown in Fig. 3. Numerous peaks have been observed in the recorded angle range of 20°-80°. The crystallographic planes of NPs overlap with those recorded in other research investigations. ZnO NPs showed robust diffraction peaks at 32° (100), 34° (002), 36° (101), 47° (102), 57° (110), 63° (103), 66° (200), 68° (112), and 69° (201). The recorded peaks strongly coincide with the hexagonal wurtzite structure of ZnO NPs [32]. The diffraction pattern and interplane spacing have been well-matched with that of the structural database of the Joint Committee on Powder Diffraction Standards (JCPDS), which further reveals the formation of wurtzite ZnO nanocrystals (JCPDS file number: 080-0074) [33].

SEM analysis of gold-coated NPs showed that the particles were predominantly spherical with an average particle diameter/size less than 50 nm, as shown in Fig. 4 and Fig. 5. The finding matched with the supplier's datasheet, and it was confirmed that particles were not pre agglomerated. The minimum size of the NPs makes them a good candidate for NFs preparation with enhanced stability and reduced viscosity.

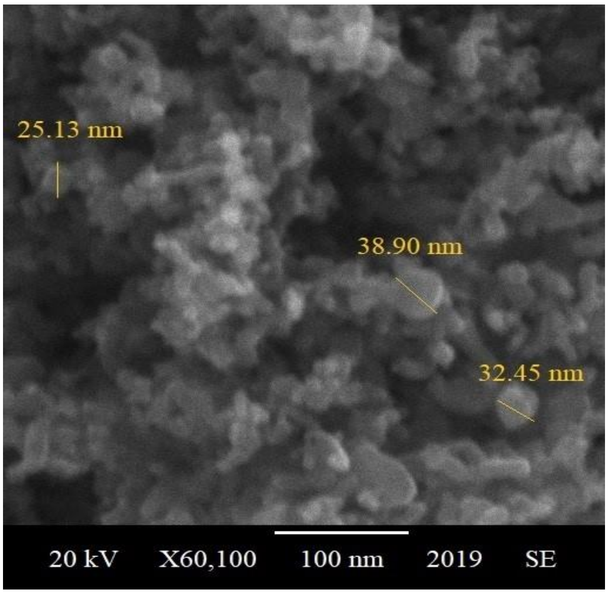


Figure 4. SEM image of ZnO NPs (MAG:60.1k)

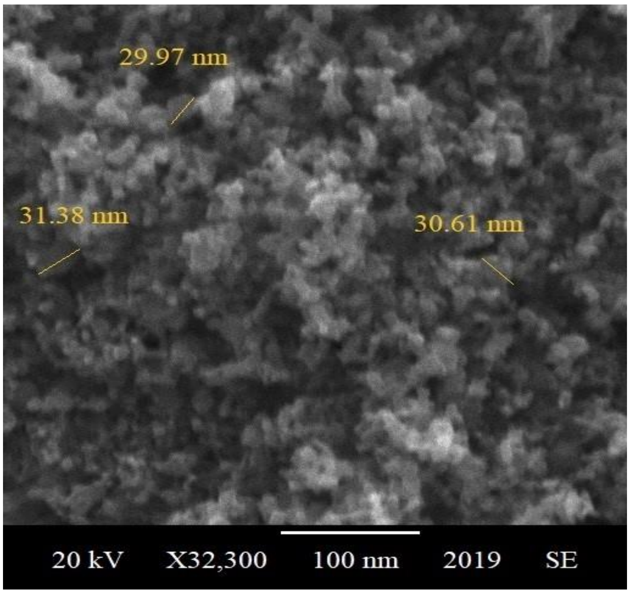


Figure 5. SEM image of ZnO NPs (MAG: 32.3)

2.3. Stability measurement of ZnO nanofluids

Stability is an important parameter and is the real challenge for individuals working with NFs [34]–[36]. From the last two decades, research is in progress for the stabilization of metal oxide NPs using different chemical and physical techniques [37]. The addition of surfactants/capping agents is a chemical technique typically used to control the aggregate size for better dispersion of NPs in a different type of base fluids [38]. The use of capping agents slows down the NPs aggregation in NFs. In the absence of capping agents, particle-particle interaction increases, accounting for the instability of the prepared NFs [39]. How to select a suitable dispersant is a key issue in the world of NFs. In general, when the base fluid is a polar solvent, water-soluble surfactants are used; otherwise, an oil-soluble surfactant is selected [40].

In this study, a UV-vis spectrophotometer was used for evaluating the dispersion stability of prepared NFs in the scanned wavelength range of 300 to 800 nm [41]. It was observed that the peak of absorption was at a wavelength of ~375 nm, which indicated the bandgap transition of ZnO NPs equal to 3.4 eV. All data were recorded at room temperature (22±1°C).

It was observed that ZnO NPs were not stable in pure DIW compared to pure EG and DIW/EG blends. An enhancement in the stability was observed when acetylacetone was used in the case of pure DIW. FTIR analysis confirmed the identity of ZnO NPs in acetylacetone stabilized ZnO/DIW NFs.

2.4. Viscosity measurement of nanofluids

Viscosity is a measure of the ability of a fluid to resist shear and is a critical component in different applications of NFs [42]. RHEOTEST Medingen GmbH RHEOTEST RN 5.1 was used to measure the viscosity of prepared NFs in 0.011-0.044 wt. % particle concentration and 15-55 °C temperature range. Further, the obtained data were used to assess the predictions of well-known Einstein, Brinkman, and Batchelor models [43]–[45]. The famous Einstein model, as given in Eq. (1), has a limitation to be used only for a very low volume concentration of NPs ($\phi \leq 0.02$ %) [43].

$$\frac{\mu_{nf}}{\mu_{bf}} = [1 + 2.5\emptyset] \tag{1}$$

Brinkman model [44], a modified form of Einstein model can be used for volume concentration up to 4.0 % and is described in Eq. (2).

$$\frac{\mu_{nf}}{\mu_{bf}} = \left[\frac{1}{(1-\emptyset)^{2.5}}\right] \tag{2}$$

Batchelor [45] proposed a correlation given in Eq. (3) considering the spherical shape and Brownian motion of the NPs:

$$\frac{\mu_{nf}}{\mu_{bf}} = [1 + 2.5\emptyset + 6.5\emptyset^2] \tag{3}$$

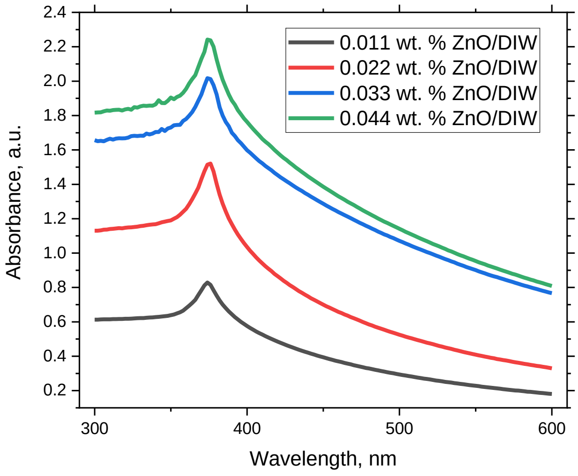
In equations 1, 2 and 3 nf, bf, μ, and ϕ represent nanofluid, base fluid, viscosity, and particle concentration, respectively. One of the common limitations of these models is that they did not consider the effect of temperature change on the viscosity of the NFs.

3. Results and discussion

3.1. Stability investigation of nanofluids

The stability analysis of prepared NFs showed that the peak for the absorbance increased with the increasing concentration of NPs. Maximum absorbance was recorded at a wavelength of ~375 nm for all concentrations of NPs. Figure 6 shows the peak absorbance of 0.829 and 2.237 for 0.011 and 0.044 mass concentrations of freshly prepared NFs in DIW, respectively, while in case of EG, the peak absorbance value was recorded as 1.521 and 3.884 respectively for the same concentrations as shown in Fig. 7. The shift in the absorbance spectrum is due to an increase in the ZnO NPs loading in the base fluids. The research analysis further clarified the drop-in absorbance spectrum with respect to time. This reflects sedimentation and aggregation of NPs within the base fluids.

Figure 8 to Figure 11 graphically shows the results for dispersion stability of ZnO NFs for 0.011-0.044 wt.% concentration in the DIW, EG, and DIW/EG blends over four hours' time span. The results show that there is a more than 50 % drop in the absorbance spectrum in the case of DIW within the evaluation time period, and this is decreased with an increasing proportion of EG and a maximum of 15 % drop in absorbance spectrum was recorded when tested with pure EG. The dilution factor has been employed for higher concentrations of NPs in the stability analysis, where the absorption spectrum went beyond the maximum limit of the UV vis. Spectrophotometer.



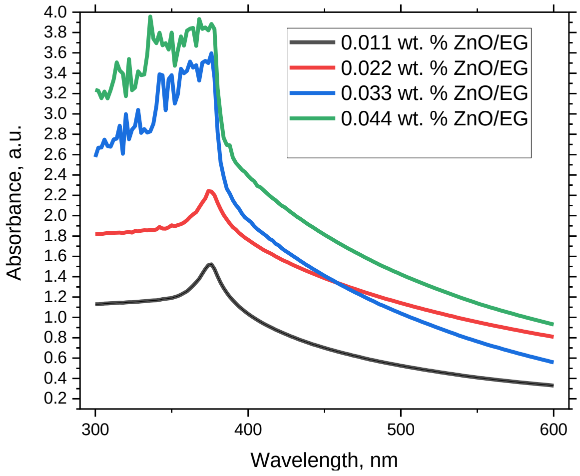
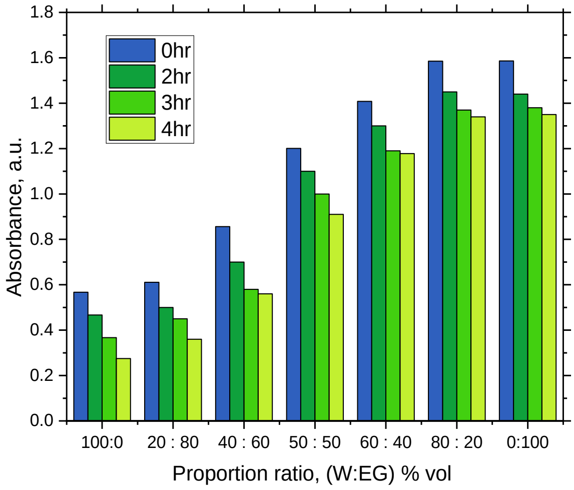


Figure 6. UV Vis. spectrum of ZnO/DIW NFs

Figure 7. UV Vis. spectrum of ZnO/EG NFs



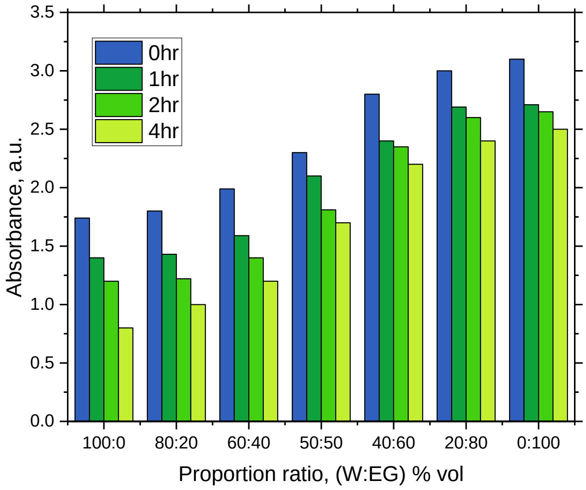
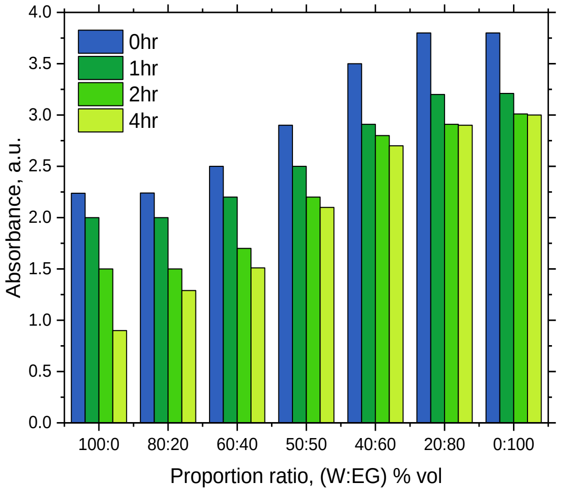


Figure 8. Stability of NFs at 0.011 wt. % of NPs

Figure 9. Stability of NFs at 0.022 wt. % of NPs



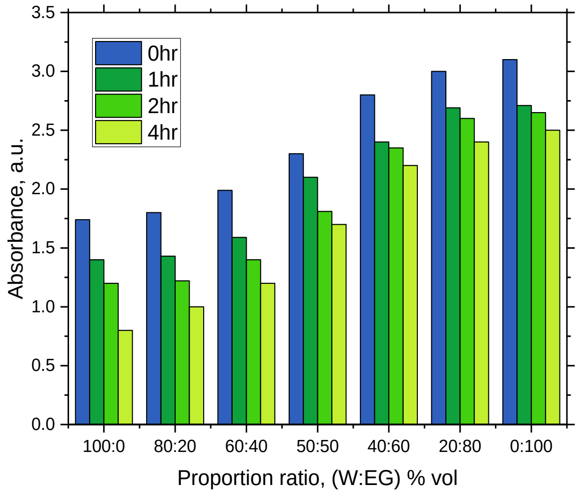


Figure 10. Stability of NFs at 0.033 wt. % of NPs

Figure 11. Stability of NFs at 0.044 wt. % of NPs

The literature review revealed that the stability of NFs could be improved with the utilization of stabilizing agents [46]. Acetylacetone was used as a surfactant in the present study to enhance the dispersion stability of ZnO NFs in DIW at the tested conditions. An addition of acetylacetone in the prepared NFs in volume concentration of 1.0 % turned the milky colour of ZnO/DIW NFs to transparent one showing the enhanced dispersion of ZnO NPs in the DIW. The prepared samples were left unattended for about five months. No sedimentation or aggregation of NPs has been observed in the NFs.

Figure 12 and Figure 13 show the FTIR analysis of pure ZnO NPs and NPs extracted from the prepared NFs. It was observed that the ZnO NPs maintained their unique identity, and this ruled out any chemical or physical change in the properties of NFs for five months. The use of surfactant has not shown any adverse effect on the properties of NPs.

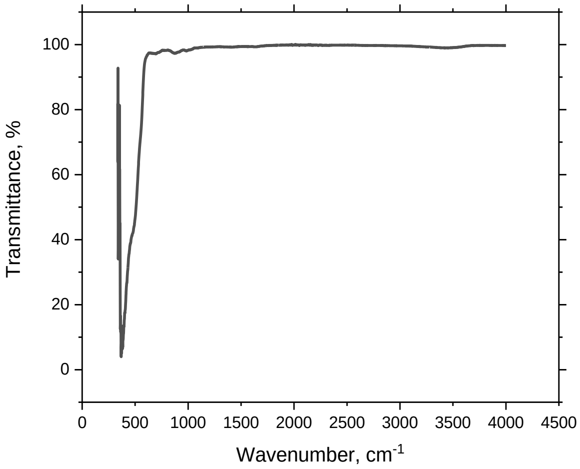


Figure 12. FTIR spectrum of pure ZnO NPs

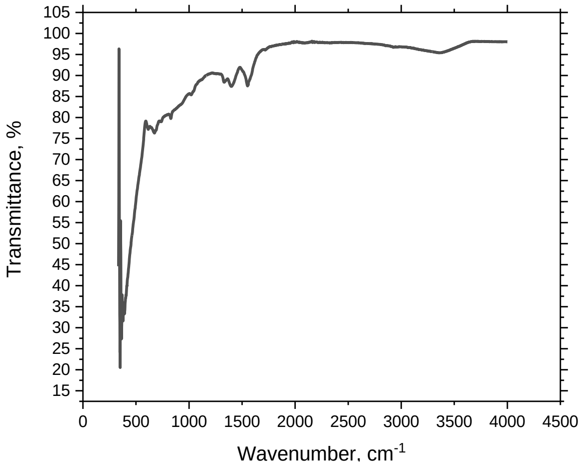


Figure 13 FTIR spectrum of used ZnO NPs

3.2. Viscosity analysis of nanofluids

The rheological characteristics of prepared NFs in the present research have been investigated by varying the shear rate from 0-1000s^{-1}, whereas experiments were done within 15-55°C temperature range. Figure 14 and Figure 15 show a change in shear stress against shear rate for 0.022 wt. % and 0.044 wt. % concentration at a constant temperature of 25 °C. NFs exhibits a slight Newtonian behaviour at higher shear rates for all kind of base fluids.

Figure 16 to Figure 19 shows the variation of viscosity with operating temperature for NFs prepared with different blends of base fluids. It has been noticed that viscosity increased with an increase in the mass concentration of NPs, while the same decreased with the rise in temperature. The change in viscosity was found to be 6.64-6.25%, with the increase in temperature from 15-55°C for 0.044 % mass concentration of NPs. This change was 5.4-3.8%, 28.6-9.5% in the case of DIW to EG ratio of 60:40, 40:60, and 35.5-18.6% for pure EG, respectively, at 0.044 wt. % in a temperature range from 15-55°C. Graphical results show the same qualitative trends (viscosity increase with increasing particle and ethylene glycol concentration), whereas numerical values are unique for each case.

Resistance is developed among fluid layers by the inclusion of NPs in the base fluids and is responsible for the rise in the viscosity of the system [47]. Cabaleiro et al. [48] also reported similar behaviour of ZnO NPs in EG and DIW in 0.025-0.150 wt. % particle concentration and 10 °C to 50 ºC temperature range.

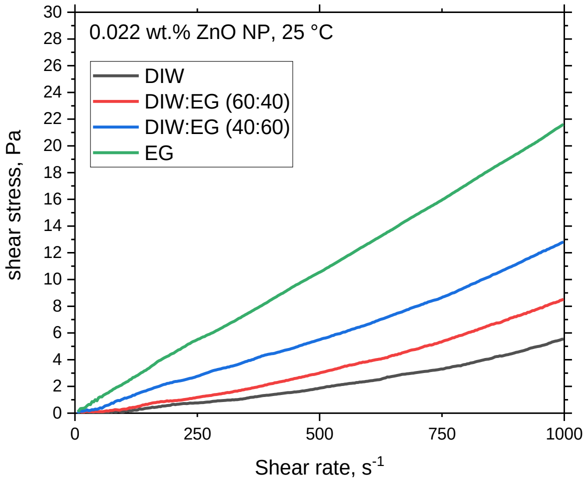


Figure 14. Shear rate vs shear stress at 0.022 wt. %

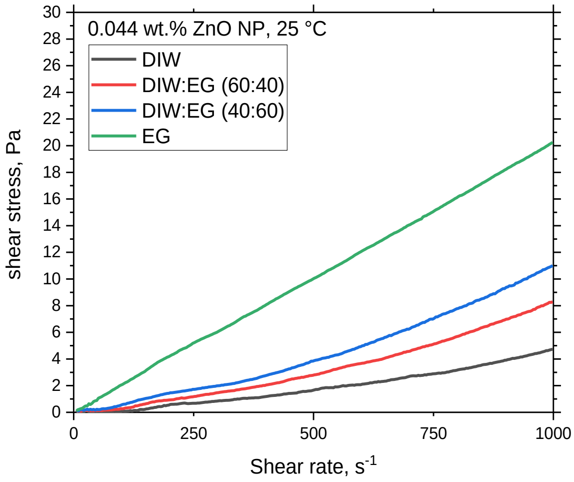


Figure 15. Shear rate vs shear stress at 0.044 wt. %

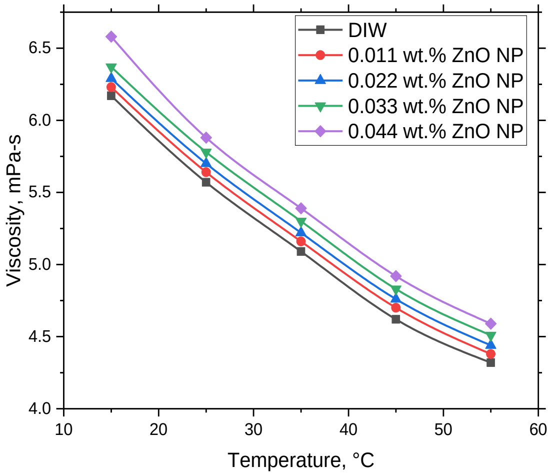


Figure16. Viscosity of DIW based nanofluids at different particles concentration and temperatures

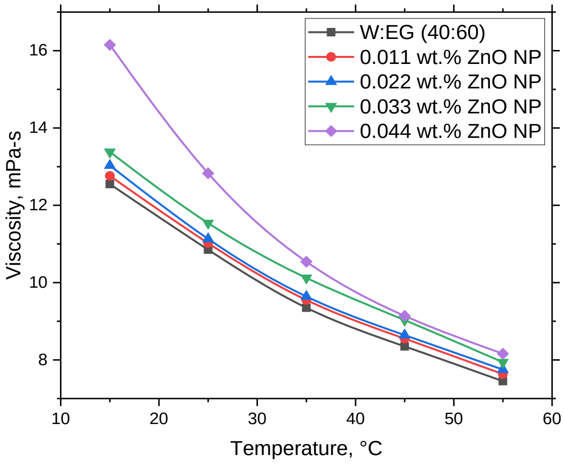


Figure 17 Viscosity of 40:60 DIW/EG based nanofluids at different particles concentration and temperatures

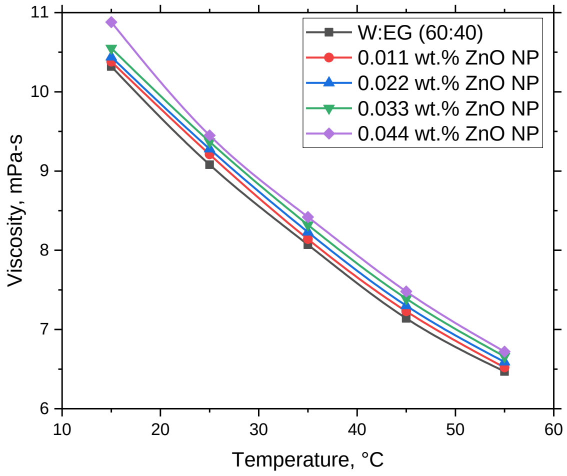


Figure 18 Viscosity of 60:40 DIW/EG based nanofluids at different particles concentration and temperatures

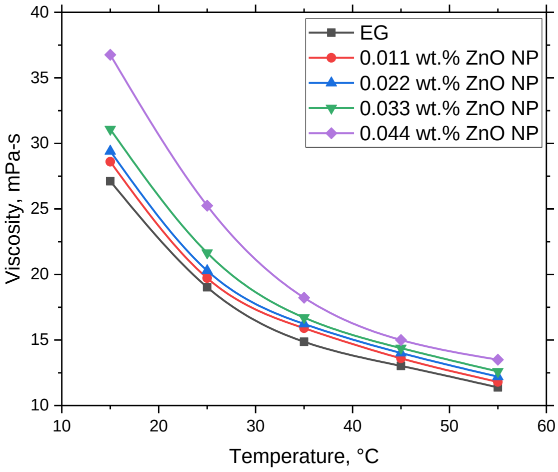


Figure 19 Viscosity of EG based nanofluids at different particles concentration and temperatures

They observed that the viscosity of NFs was increased from 6.0 to 7.8 mPa.s at 10 °C with an increase in concentration, viscosity was reported to decrease from 8 to 2.9 mPa.s for 0.150 wt. % over a decrease in temperature.

The viscosity trend of all kinds of NFs based on EG, DIW, and their blends were directly proportional to the amount of particle loading, but it portrayed an inverse relation with the temperature of the overall system. The decrease in viscosity of the NFs system with the rise in temperature shows that intermolecular forces have become weak with temperature rise. Figure 20 and Figure 21 shows that viscosity of ZnO nanofluid increases on the addition of acetylacetone surfactant, which is a shear thickening behaviour of the fluid as reported in another study [49]. A maximum of 1.6 % rise in the viscosity was recorded at a temperature of 45 °C and a concentration of 0.044 wt.%.

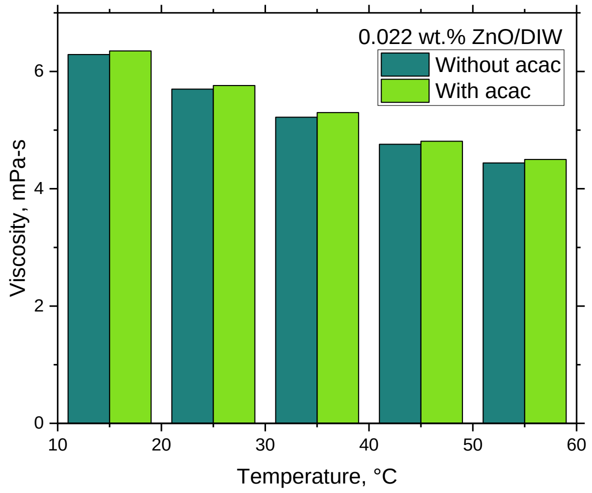


Figure 20. Impact of surfactant on viscosity of ZnO/DIW NFs in 0.022 wt. % concentration

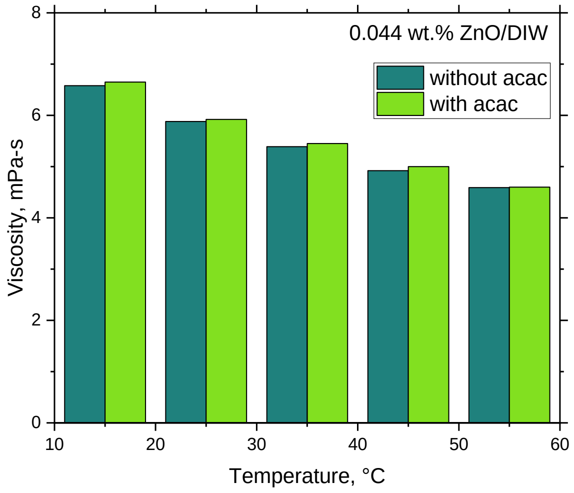


Figure 21 Impact of surfactant on viscosity of ZnO/DIW NFs in 0.044 wt. % concentration

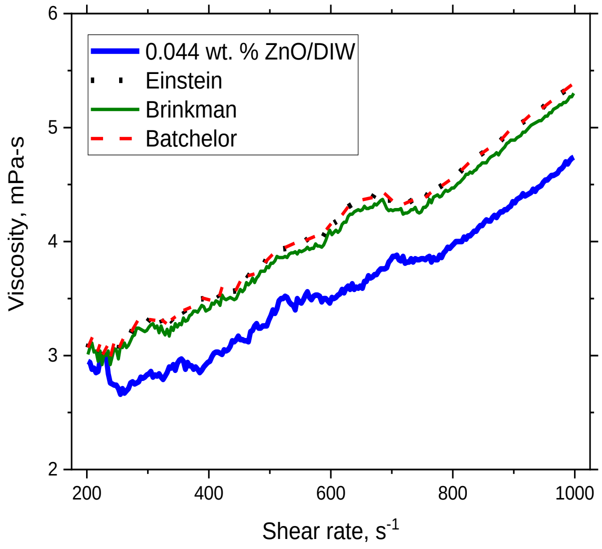


Figure 22. Comparison of viscosity of ZnO/DIW NFs with rheological models [43]–[45]

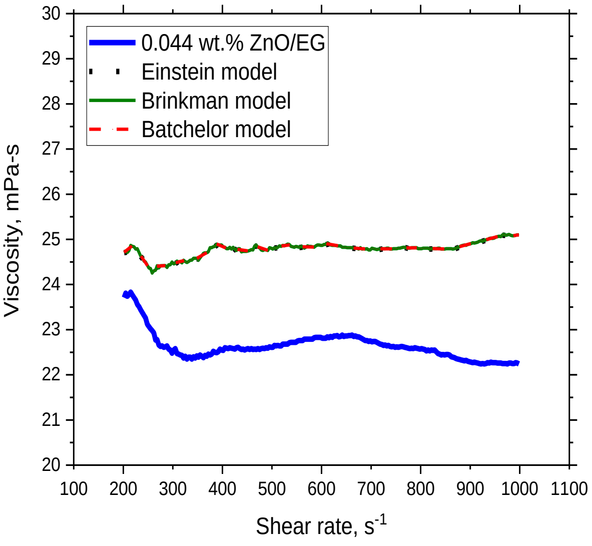


Figure 23. Comparison of viscosity of ZnO/EG NFs with rheological models [43]–[45]

The viscosity of prepared NFs was in close conformance with well-known viscosity models developed by Einstein, Brinkman, and Batchelor [43]–[45]. Figure 22 and Figure 23 portray that approximately 5.0 % variation is observed in the value of the viscosity parameter for 0.044 wt. % ZnO

NPs in DIW as well as EG base fluid at a shear rate of 600 cm^{-1}. The findings of this study matches with those reported by Khodadadi et al. [50], they compared the results of different studies to evaluate significance of stated models.

4. Conclusion

The dispersion stability of ZnO-based NFs prepared by a typical two-step method has been investigated in pure DIW and EG along with their blends using different nanoparticle loadings (0.011-0.044 wt. %) and 15-55°C temperature range. Conclusions from the study are summarized below;

- The stability analysis of prepared NFs revealed that ZnO NPs exhibits less stability in pure DIW; better stability was noticed with EG for the tested time span.
- The spectrometric analysis showed that there was more than 50 % drop in absorbance spectrum (over an extended time period) for experiments conducted with DIW, this drop shrinked and eventually reduced to 15 % for tests carried out with pure EG.
- Acetylacetone was used to enhance the dispersion stability of ZnO NPs in DIW. An enhancement in the stability was recorded, and the prepared NFs were found to be stable for more than five months. FTIR analysis ruled out any adverse effect of surfactant addition on the properties of ZnO NPs.
- The viscosity of NFs increased with an increase in particles concentration and with increasing proportion of EG in DIW, the highest values were recorded with pure EG based NFs. Addition of surfactant adversely affected viscosity of NFs, the maximum rise in viscosity was noticed to be 1.6%.
- Comparison of experimental data showed Einstein, Batchelor, and Brinkman’s models overestimated viscosity of prepared NFs.

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