DISPERSION STABILITY AND RHEOLOGICAL CHARACTERISTICS OF WATER AND ETHYLENE GLYCOL BASED ZnO NANOFLUIDS

by

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> Research paper https://doi.org/10.2298/TSCI200110187Q

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 rheome*ter. 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, ZnO

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 (NF), 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

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of NF in solar energy, refrigeration systems [5], medical applications [6], chemical reactions, and automobiles [7] has attracted the attention of many researchers.

Nanoparticles (NP), dispersed in different working mediums, exhibit quite different properties, and the resulting NF are supposed to show enhanced thermal and heat transfer characteristics than the host base fluids [8, 9]. The thermal conductivity of NF 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 NP for heat transfer enhancement increased the viscosity of the working medium under consideration [11]. The NF are considered stable when the NP are well dispersed in base fluid and do not tend to aggregate at a significant rate. The NP are prone to hard aggregate due to their large surface area and high surface energy. The stability of the NF 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 NF [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].

The NP of metal oxide materials such as Al₂O₃, CuO, SiO₂, TiO₂, 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 latter 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 NF (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 NP from 500-2000 nm for ZnO NF and 250-1000 nm for CuO NF. An improvement of 53.0 % in thermal conductivity has been recorded in case of 0.5 wt.% ZnO NF. Sughanti and Rajan [22] investigated the stability of 0.2-2.0 vol.% ZnO/DW NF 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 NF was also observed to be increased with the formation of these agglomerates. Raykar and Singh [23] stabilized aqueous ZnO NF using acetylacetone at a particle loading of 0.075-0.50 wt.%. They reported an enhancement in the dispersion stability of NF for up to one year, while thermal conductivity enhanced up to 40% for 0.5 wt.% of NP at 30 °C. In another study [24] enhanced stability of ZnO/DW NF using SHMP as a surfactant was observed in a pH range of 8.58-9.38 and at surfactant to NP ratio of 1:2 and 1:6, respectively. Pastoriza-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 NF.

While many metal oxide NP have been widely studied, to the best of the author's knowledge from the review of contemporary literature, the stability and rheology of ZnO based NF 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 NF in DIW, EG, and their blends with special attention to their ap-

plication in microelectronic devices generating high heat flux. Acetylacetone was used as a stabilizing agent.

Materials and methods

For work reported in this study, ZnO NP 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. The DIW was purchased from the local market. Ultrasonication bath (Elmasonic EASY 30 H, Germany) was used for the dispersion of NP in the base fluid. The X-ray diffraction (XRD) (STOE & Cie GmbH, Germany), was used to observe the crystal structure of NP, SEM (TESCAN, VEGA3, Czech Republic) was used to explore the shape and size of NP, whereas fourier transform infrared (FTIR) (Perkin Elmer Spectrum Two spectrometer) was used to investigate the identity of NP in the base fluids.

Preparation of NF

The preparation of stable NF is the first step in any NF 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 NF. 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 °C under standard atmospheric pressure conditions [28]. The 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 NP are highly unstable in pure DIW compared to EG. The dispersion of NP within EG and its blends with DIW are supposed to enhance the stability of NF. Additionally, the same will also improve their possible application for very cold climatic conditions.

The ZnO based NF 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, and 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 NP [30]. Ultrasonication is the most accepted physical process to break the NP aggregates to enhance their stability in prepared NF [31].

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



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



Figure 2. The ZnO NP and ZnO/DIW based NF



Figure 3. The XRD pattern of ZnO NP

Characterization of ZnO NP

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

The profile of the structure obtained from XRD for as received NP is shown in fig. 3. Numerous peaks have been observed in the recorded angle range of 20-80°. The crystallographic planes of NP overlap with those

recorded in other research investigations. The ZnO NP 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 NP [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].

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

Stability measurement of ZnO NF

Stability is an important parameter and is the real challenge for individuals working with NF [34-36]. From the last two decades, research is in progress for the stabilization of metal oxide NP 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 NP in a different type of base fluids [38]. The use of capping agents slows down

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

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

the NP aggregation in NF. In the absence of capping agents, particle-particle interaction increases, accounting for the instability of the prepared NF [39]. How to select a suitable dispersant is a key issue in the world of NF. 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 NF in the scanned wavelength range of 300-800 nm [41]. It was observed that the peak of absorption was at a wavelength of \sim 375 nm, which indicates the bandgap transition of ZnO NP equal to 3.4 eV. All data were recorded at room temperature (22±1 °C).

It was observed that ZnO NP 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. The FTIR analysis confirmed the identity of ZnO NP in acetylacetone stabilized ZnO/DIW NF.

Viscosity measurement of NF

Viscosity is a measure of the ability of a fluid to resist shear and is a critical component in different applications of NF [42]. The RHEOTEST Medingen GmbH RHEOTEST RN 5.1 was used to measure the viscosity of prepared NF 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 be used only for a very low volume concentration of NP ($\phi \le 0.02\%$) [43]:

$$\frac{\mu_{\rm nf}}{\mu_{\rm bf}} = \left[1 + 2.5\phi\right] \tag{1}$$

Brinkman model [44], a modified form of Einstein model can be used for volume concentration up to 4.0% and is described:

$$\frac{\mu_{\rm nf}}{\mu_{\rm bf}} = \left\lfloor \frac{1}{\left(1 - \phi\right)^{2.5}} \right\rfloor \tag{2}$$

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

$$\frac{\mu_{\rm nf}}{\mu_{\rm bf}} = \left[1 + 2.5\phi + 6.5\phi^2\right] \tag{3}$$

where nf, bf, μ , and ϕ are the NF, 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 NF.

Results and discussion

Stability investigation of NF

The stability analysis of prepared NF showed that the peak for the absorbance increased with the increase in concentration of NP. Maximum absorbance was recorded at a wavelength of \sim 375 nm for all concentrations of NP. Figure 6 shows the peak absorbance of 0.829 and 2.237 for 0.011 and 0.044 mass concentrations of freshly prepared NF 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 NP 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 NP within the base fluids.



Figure 6. The UV-vis. spectrum of ZnO/DIW NF Figure 7. T

Figure 7. The UV-vis. spectrum of ZnO/EG NF

Figures 8-11 graphically shows the results for dispersion stability of ZnO NF 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 NP in the stability analysis, where the absorption spectrum went beyond the maximum limit of the UV-vis. spectrophotometer.

The literature review revealed that the stability of NF 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 NF in DIW at the tested conditions. An addition of acetylacetone in the prepared NF in volume concentration of 1.0% turned the milky color of

1994



Figure 8. Stability of NF at 0.011 wt.% of NP



Figure 10. Stability of NF at 0.033 wt.% of NP



Figure 9. Stability of NF at 0.022 wt.% of NP



Figure 11. Stability of NF at 0.044 wt.% of NP

ZnO/DIW NF to transparent one showing the enhanced dispersion of ZnO NP in the DIW. The prepared samples were left unattended for about five months. No sedimentation or aggregation of NP has been observed in the NF.

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

Viscosity analysis of NF

The rheological characteristics of prepared NF in the present research have been investigated by varying the shear rate from 0-1000 1/s, whereas experiments were done within 15-55 °C temperature range. Figures 14 and 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. The NF exhibits a slight Newtonian behaviour at higher shear rates for all kind of base fluids.





Figure 12. The FTIR spectrum of pure ZnO NP





at 0.022 wt.%

at 0.044 wt.%

Figures 16-19 shows the variation of viscosity with operating temperature for NF prepared with different blends of base fluids. It has been noticed that viscosity increased with an increase in the mass concentration of NP, 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 NP. 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 EG concentration), whereas numerical values are unique for each case.

Resistance is developed among fluid layers by the inclusion of NP 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 NP in EG and DIW in 0.025-0.150 wt.% particle concentration and 10-50 °C temperature range.

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

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Figure 16. Viscosity of DIW based NF at different particles concentration and temperatures



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



The viscosity trend of all kinds of NF 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 NF system with the rise in temperature shows that intermolecular forces have become weak with temperature rise. Figures 20 and 21 shows that viscosity of ZnO NF 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.%.

The viscosity of prepared NF was in close conformance with well-known viscosity models developed by Einstein, Brinkman, and Batchelor [43-45]. Figures 22 and 23 portray that approximately 5.0% variation is observed in the value of the viscosity parameter for 0.044 wt.% ZnO NP in DIW as well as EG base fluid at a shear rate of 600 1/cm. 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.



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



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





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

Conclusions

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

- The stability analysis of prepared NF revealed that ZnO NP 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 NP in DIW. An enhancement in the stability was recorded, and the prepared NF were found to be stable for more than five months. The FTIR analysis ruled out any adverse effect of surfactant addition on the properties of ZnO NP.

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- The viscosity of NF increased with an increase in particles concentration and with increasing proportion of EG in DIW, the highest values were recorded with pure EG based NF. Addition of surfactant adversely affected viscosity of NF, 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 NF.

Acknowledgment

The authors like to thank Department of Mechanical, Manufacturing and Mechatronics Engineering, University of Engineering and Technology, Lahore (New Campus) for providing funds and facilities to execute the present research work. We are also thankful to Higher Education Commission-Pakistan for providing free access to different journals and libraries.

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Paper revised: April 26, 2020

Paper accepted: May 11, 2020

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