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Influence of the Base Liquid and Particle Size on the Thermo-Physical Characteristics of MoS_2 Micro- and Nanofluids

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Abstract

The present work investigates the formulation and evaluation of thermo-physical properties of water (W) and water/ethylene glycol (W/EG) based nanofluids and microfluids (NFs/MFs) made with the dispersion of Molybdenum(IV) sulfide nanoparticles and microparticles (MoS_2 NPs/MPs) with focus on the effect of the particle size and the base liquid. A series of stable NFs and MFs with particle concentrations 0.25, 0.5 and 1wt% were prepared by dispersing MoS_2NPs and MPs in W and W/EG as the base liquids. Thermal conductivity (TC) and viscosity of MoS_2NFs and MFs, as the most important thermo-physical properties influencing the performance of heat exchange fluids, were measured at 20 ºC. The results for TC and viscosity of W based MoS_2NF-MFs were compared with the same NFs-MFs with W/EG base liquid to reveal the impact of the base liquid. The experiments showed that W/EG based NFs-MFs exhibit higher heat transfer efficiencies than that of W-based suspensions. Moreover, NFs with MoS_2NPs exhibited higher TC than those of MFs containing the same particle concentration and at the same temperature, which helps to identify the effect of NP size. The best result was obtained for water-based NF with 1wt% MoS_2NP, demonstrating a TC enhancement of ~12.4 % with ~10.6 % viscosity increase at 20 ºC.

Keywords: *Heat transfer; Nanofluids, MoS_2; Nanoparticles; Microparticles; Thermal conductivity; Viscosity.*

1. Introduction

The necessity for transport of heat using an appropriate heat-exchange fluid is quite common in industries including engineering devices, machine and plants producing energy. Heat transfer through a fluid takes place by convection, which can be improved by improving the thermal conductivity of the heat-exchange fluid. It has been demonstrated that suspension containing solid particles may display an enhanced TC [1] as the TC of solids is significantly higher than that of liquids. Large particles, however, can easily sediment which is not desirable in heat-exchange fluids. To tackle this challenge, new suspensions containing nanoparticles (NP), defined as nanofluids (NFs) [2], have been formulated offering possibility of enhancing heat-transfer performance of conventional heat-exchange fluids. Compared to micron sized particles (MP), NPs offer higher surface area, which is shown to enhance heat-transfer properties of suspensions [3]. Various micro and nanostructured materials have been employed to fabricate NF/MF in several base liquids for heat-exchange purposes. Carbon based materials (such as carbon nanotubes and Graphene nanoplatelets) [4-5] or metals such as Ag [6], Cu [7-10], and ceramics including alumina (Al_2O_3) [11], iron oxide (Fe_3O_4) [12], copper oxide (CuO) [13], titania (TiO_2) [14], silica (SiO_2)

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[15], mesoporous SiO_2 [16], cerium oxide (CeO_2) [17], zinc oxide (ZnO) [18], and silicon carbide (SiC) [19-21] are some of the materials the effect of which on heat-exchange has been studied.

A wide range of liquids can be used as base liquids, though water (W), ethylene glycol (EG) and mixture of water and EG (W/EG) are the most relevant ones. As for the formulation of NFs-MFs there are two major routes defined as two-step, or one-step formulation. In the two-step formulation route, which is the most commonly adopted ones due to its use, solid particles are pre-synthesized, separated, dried and then dispersed in the base liquids of interest [22], making possible the use of commercial nanoparticles in the formulation. In the one-step route the NPs-MPs are directly fabricated in the base liquid [23-25], minimizing the number of process steps, and also providing a better control over particle size and morphology -whenever possible. The composition of dispersed particulate materials influences the thermo-physical properties of NFs-MFs, while other factors such as particle morphology (size and shape) [26], composition of the base liquid [27-28], NF-MF preparation method [29], stability of suspension and surfactants [30] can also significantly influence the TC and viscosity of NFs-MFs. Among all enlisted factors, although the role of particle size and base liquid composition on thermo-physical properties of NFs-MFs is indisputable, there is no detailed study on their effect on TC and viscosity. Limited number of studies have been reported with huge discrepancy in the reported data [31]. Furthermore, in many of the earlier reports the true impact of particle size and base liquid on TC and viscosity are difficult to ascertain, due to the use of additives, surfactants or stabilizers. The impact of base liquid composition is not very well documented and its role on TC of NFs for different viscosities is still debated. NFs with Al_2O_3 NPs in different base liquids by Xie et al. [32-33] proved that enhancement in TC of NFs is more pronounced if base liquid with lower TC is employed. SiC NFs in water (W) and W/EG mixture with various particle concentration, particle sizes and pH have been reported Timofeeva et al. [34]. Their investigations revealed that the TC enhancement is higher in W/EG based NFs compared to the same NFs in W. We recently reported on the effect of base liquid on TC and viscosity on two NF systems containing α-SiC NP in different base liquids of W and W/EG mixture [35]. The study showed the W/EG based NFs exhibited higher heat-exchange efficiencies than similar W-based NFs. About the effect of particle size on TC and viscosity of NFs, some studies showed that the TC enhancement of NFs could be improved when smaller NPs is used [36] while there are also other reports with the opposite trend [37]. Our studies on the effect of base liquid on thermo-physical properties of EG and diethylene glycol (DEG) based NFs containing Cu NPs showed EG based NFs displayed more promising characteristics than that of DEG based NFs-MFs [38].

Nevertheless, there is still a serious need for a systematic study on the effect of base liquid and particle size on thermo-physical properties of NFs-MFs. For this purpose, we designed an experimental work using nanostructured and micron sized MoS_2 particles stabilized in different base liquids to study the role of these factors on the TC and viscosity of NFs/MFs. So far, few studies have been conducted to enhance thermal characteristics of conventional heat transfer fluids using nanostructured MoS_2. Recently, Zeng et al [39] studied the application of MoS_2 NF for heat-exchange applications where engine oil based (B350) NFs by dispersing stearic acid as surface modified spherical MoS_2 NPs with size in the range of 50-100 nm. They studied the TC of NFs, though the changes in viscosity of NFs was not reported. Su et al. [40] reported on the stability and TC of W-based and oil-based MoS_2 NFs (with NP loading in the range of 0.01 and 0.5 wt. %), with no mention of viscosity. Su et. al. reported on nanofluids in W, vegetable lubricant (LB2000) and unsaturated polyhydric alcohol ester (PriECO6000) as the base liquids by dispersing commercial ~30 nm MoS_2 NPs, where no systematic report on the effect of base liquid effect and

particle sizes on thermophysical properties of MoS_2 NFs/MFs was reported. Thermophysical properties EG based NFs and MFs containing MoS_2 NPs and MPs at 20 ^oC was recently reported by our group [41].

In the present work, MoS_2 NPs and MPs of different size ranges were stabilized in water (W) and water/ethylene glycol (W/EG) base liquids via a two-step method and their TC and viscosity properties were analyzed. For a direct comparison with this report, MoS_2 NP-MP loading, the ranges of particle size and the NF-MF formulation procedure were kept the same [41]. Here we have focused on the comparison between TC and viscosity (experimentally) obtained during this work with EG NFs [41]. In fact, study and reporting the role of particle size and different base liquids on the TC and viscosity of MoS_2 NFs/MFs is the novelty of this work. There is no such study in the literature yet, which experimentally investigates and reports the effect of particle size and base liquid on TC and viscosity of MoS_2 NFs and MFs. This work allows us to compare the effect of base liquid as well as the particle sizes (nanoscale vs micron) on the thermo-physical properties of MoS_2 NFs-MFs. Our findings on the physicochemical and thermophysical properties of NFs-MFs are presented in detail.

2. Materials and Methods

Molybdenum(IV) sulfide nanoparticles/microparticles (MoS_2 NPs-MPs) and EG were acquired from Sigma Aldrich (Germany) and were used as received, without any further purification. MoS_2 NPs-MPs were utilized to prepare a series of NFs-MFs with 0.25 wt%, 0.5 wt% and 1 wt% NPs-MPs loading. For this purpose, MoS_2 NPs and MoS_2 MPs were dispersed in distilled water (DW) as the base liquid (two-step preparation method) to obtain MoS_2 NF-MF. In order to obtain stable NFs-MFs, the suspensions were mixed by ultrasonic mixing and the pH of the NFs was adjusted to ~9.5 -to achieve strong negative charge on its surface that prevents agglomeration [42]. In order to prepare W/EG (50/50 by wt%) based MoS_2 NFs and MFs, the same formulation procedure was followed, using W/EG as the base liquid, so that three NFs and three MFs with varying MoS_2 NPs and MPs were prepared. All NFs-MFs, that are listed in Table 1, showed long-term stability without any visual precipitation. Since one of our aims was to study the real effect of MoS_2 NPs and MPs on thermo-physical properties of NFs-MFs, the use of surfactant or surface modifiers were strictly avoided. Morphology and microstructure of MoS_2 NPs-MPs was studied by using Scanning Electron Microscopy (FEG-HR SEM, Zeiss-Ultra 55) working at 20 kV accelerating voltage. A closer investigation of MoS_2 particles size and morphology was performed using Transmission Electron Microscopy (TEM, JEOL 2100) at 200 kV accelerating voltage. Surface bound groups of as-received commercial MoS_2 NPs-MPs was studied was studied by Fourier Transform Infrared (FT-IR) Spectroscopy in the range of 400-4000 cm-1 (Nicolet Avatar IR 360 spectrophotometer) via KBr pellet method in the transmission mode. Average hydrodynamic/solvodynamic particle size distribution was estimated by dynamic light scattering (DLS, Beckmann-Coulter Delsa Nano C system). The TC of NFs and MFs was evaluated using TPS 2500 system as described earlier [43]. The validity of the TPS system was tested using thermodynamic properties of water (IAPWS reference) and comparing with the reference; the measurement accuracy for distilled water was within ± 2% [44]. Finally, the viscosity of NFs and MFs was assessed using DV-II+ Pro- Brookfield viscometer, after verifying the validity on distilled water; the measurement accuracy was found within ± 4% [44].

Table 1. Compositional details of the fabricated W and W/EG based MoS_2 NFs and MFs at pH 9.5.

Sample ID	Particle ID	Base Liquid	NP/MP loading (wt %)
Nano- MoS_2-W	MoS_2 NP	W	0.25
Nano- MoS_2-W	MoS_2 NP	W	0.5
Nano- MoS_2-W	MoS_2 NP	W	1
Micro- MoS_2-W	MoS_2 MP	W	0.25
Micro- MoS_2-W	MoS_2 MP	W	0.5
Micro- MoS_2-W	MoS_2 MP	W	1
Nano- MoS_2-W/EG	MoS_2 NP	W/EG	0.25
Nano- MoS_2-W/EG	MoS_2 NP	W/EG	0.5
Nano- MoS_2-W/EG	MoS_2 NP	W/EG	1
Micro- MoS_2-W/EG	MoS_2 MP	W/EG	0.25
Micro- MoS_2-W/EG	MoS_2 MP	W/EG	0.5
Micro- MoS_2-W/EG	MoS_2 MP	W/EG	1

3. Results and Discussion

3.1. Structural Analysis

To study the crystal structure of MoS_2 NPs/MPs, X-ray powder diffraction (XRPD) was performed and the result is displayed in Figure 1. Based on that MoS_2 NP and MoS_2 MPs are nearly identical and display hexagonal phase of MoS_2 (ICDD Ref pattern# 98-002-4000). All main diffraction peaks are indexed against the crystallographic data, as marked by the relevant Miller indices in Figure 1.

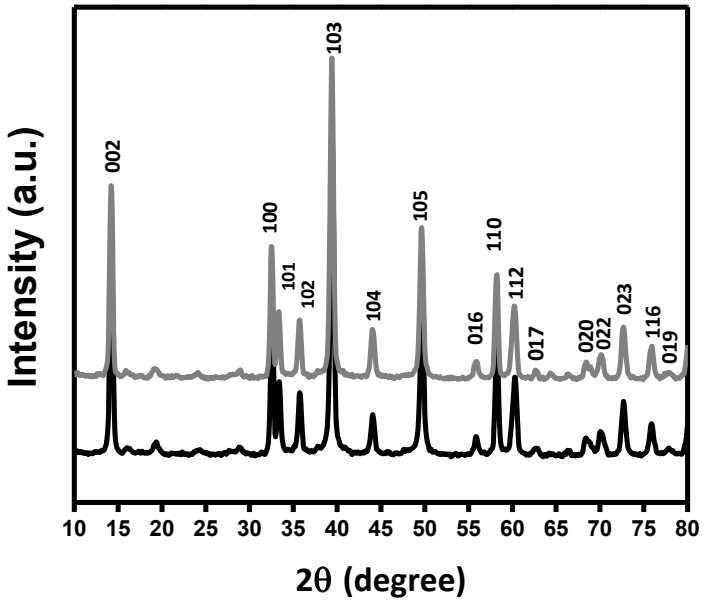

Figure 1. XRPD pattern of as-received MoS_2 NP (gray line) and MPs (black line) (indexed with ICDD Ref pattern# 98-002-4000).

3.2. Morphology analysis

Figure 2(a) and (b) display SEM micrographs of MoS_2 NPs and MPs, respectively. There is a wide range of morphologies and broad size distribution for MoS_2 NPs, making it relatively difficult to assess an average particle size from the micrograph. A rough estimate for the average primary particle size of MoS_2 NP is ~90 nm. From the micrographs of MoS_2 MPs an average primary particle size of around ~1.2 µm can be estimated. TEM micrographs of MoS_2 NPs and MoS_2 MPs are displayed in Figure 2(c) and (d), respectively, where the layered structure can be observed for both MoS_2 NPs and MPs.

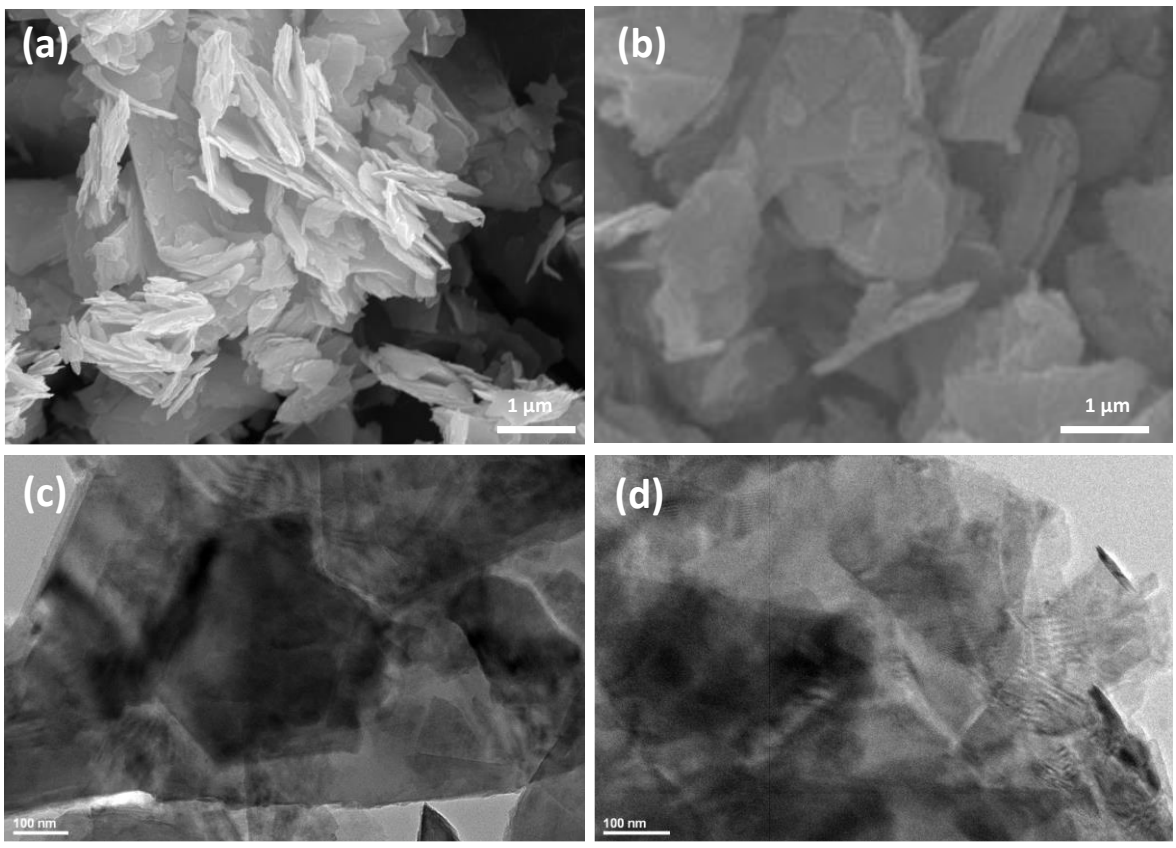

Figure 2. SEM and TEM micrographs of as-received particles; (a, c) MoS_2 NPs and (b, d) MoS_2 MPs.

3.3. Dynamic Light Scattering (DLS) Analysis

DLS analysis was performed for MoS_2 NFs and MFs in W and W/EG base liquids to analyze the dispersed size of NPs and MPs within the results are presented in Figure 3 (a). The size distribution of MoS_2 MPs and NPs in W medium was estimated in the range of 1800-4900 nm and 1050-1860 nm with an average hydrodynamic size of 3120 nm and 1400 nm, respectively. DLS analysis was also performed in W/EG medium for MoS_2 MFs and NFs. The estimated sizes ranges were 140-5900 nm and 100-795 nm with an average solvodynamic size of 576 and 340 nm, respectively. A comparison between DLS estimates in W and W/EG media exhibits that the narrowest particle size distribution was obtained for MoS_2 NP in water media though skewed towards larger sizes. Furthermore, both NP and MP exhibit smaller average solvodynamic size in W/EG base liquid compared to the W medium. This may indicate a better dispersion of MoS_2 particles in W/EG medium than W alone. Such a significant discrepancy between the primary particle size estimated

from SEM and solvodynamic size measured with DLS can be ascribed to the agglomeration of the MoS_2 NPs and MPs in W. EG can coordinate with the nanoparticle surface, thus improving their dispersion quality, as observed by comparing the portion of the DLS size distribution in < 1000 nm range in W vs W/EG media. Surface properties are important characteristics that are liable to play important role in thermo-physical properties of NFs/MFs [45]. For this purpose, in order to understand the surface chemistry of particles, FTIR analysis was carried out on "as-received" MoS_2 NP and MoS_2 MP. The results of FTIR spectra are displayed in Figure 3 (b). Almost identical surface characteristics were observed for both as-received MoS_2 NPs and MPs. The absorption band at 1120 cm^{-1} is ascribed to the typical Mo-S stretching vibration. MoS_2 MP spectrum showed residual organic components with absorption bands centered at 2920 cm^{-1} and 2856 cm^{-1} that can be assigned to stretching vibration of C-H. It is well known that MoS_2 has a high surface activity, and can absorb various gaseous species from air, which is the reason it is used as a catalyst in some applications. Absorption between 4000 cm^{-1} to 3400 cm^{-1} and 2000 cm^{-1} to 1300 cm^{-1} results from the characteristic adsorbed water vapor (H_2O) on the MoS_2 surface. In addition to water vapor, the absorption of carbon dioxide (CO_2) may be observed near 2350 cm^{-1} and 670 cm^{-1} due to adsorbed CO_2 species on the MoS_2 surface. As-received MoS_2 MPs do not exhibit a clean surface, which may be attributed to some surface exposed organic layers or contamination during the synthesis process.

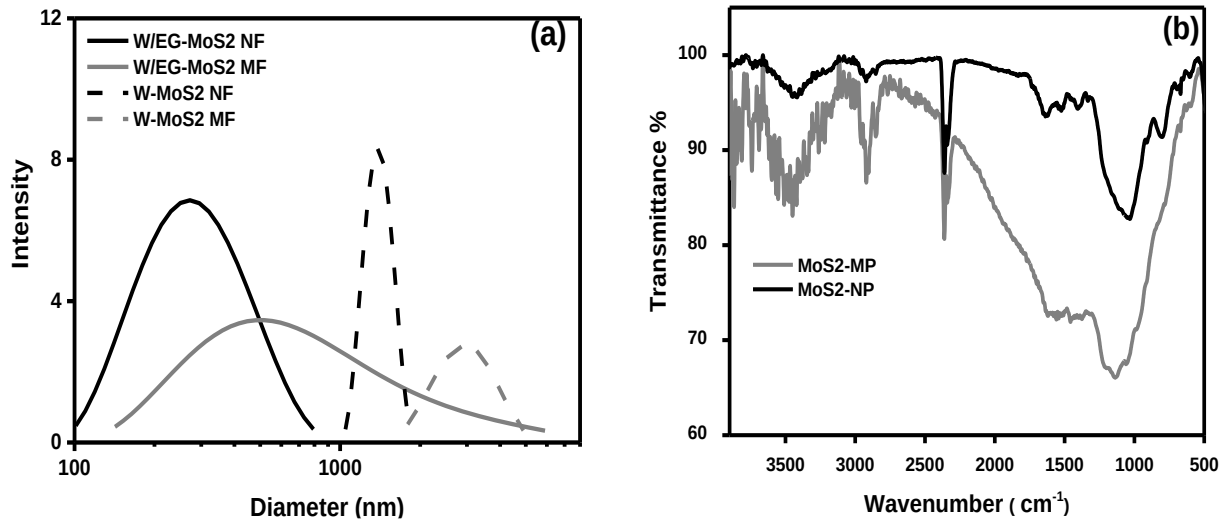

Figure 3. (a) Particle size distribution of MoS_2 in W and W/EG base liquids measured by DLS. (b) FTIR spectra of as-received MoS_2 NPs (gray line) and MPs (black line).

3.4. Thermo-physical properties evaluation of MoS_2 NFs/MFs

Two major thermophysical properties, as the TC and viscosity NFs and MFs, are influenced by the addition of NPs and MPs into the base fluids. Effective NFs-MFs for heat transfer applications require enhanced TC with minimal viscosity increase [46]. Hence, formulation of NFs and MFs with the highest possible TC enhancement with a minimal impact on viscosity may result in the desired goal. TC and viscosity were measured to evaluate the heat exchange performance of the fabricated MoS_2 NFs and MFs, as listed in Table 1. The results for relative TC of samples [(K_{nf} /K_{bl}-1)*100] at 20 °C are presented in Figure 4(a) where K_{nf} stand for TC of NFs-MFs and and K_{bl} for the base liquid. For all water and W/EG based NFs and MFs, the TC enhanced with increasing MoS_2 NPs and MPs loadings. As Figure 4 (a) displays the NFs with both W and W/EG base liquids

exhibit higher TC improvement compared to the MFs with the same NP-MP loading. It displays the positive impact of utilizing nanomaterials as compared to micron sized particles. It should be noticed that since particle size distribution is broad, the results might not be as conclusive as it should be. There is no strong theory/model to explain this matter yet; however, it may be attributed to the higher

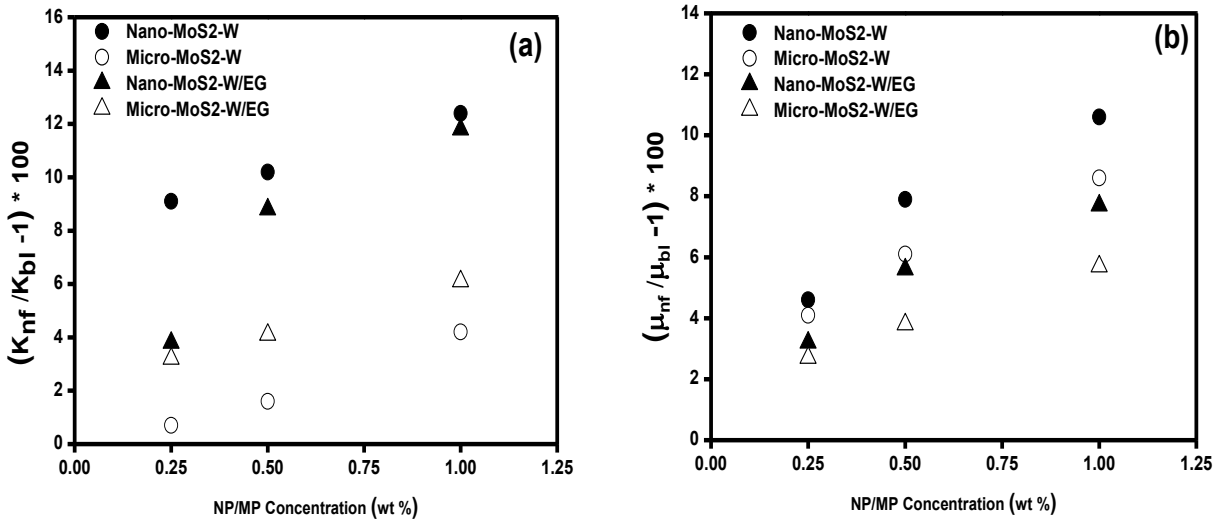

Figure 4. (a) TC enhancement vs NP/MP concentration of water and W/EG based MoS_2 NFs and MFs with various loading of MoS_2 NP and MP and (b) increase in viscosity vs NP/MP concentration of water and W/EG based MoS_2 NFs and MFs with various loading of MoS_2 NP and MP (0.25, 0.5 and 1wt%); all measured at 20 ^oC. Error bars ± 2% for TC and ± 4% for are not shown as they are within the data marker size.

surface/interfacial area of MoS_2 NPs as well as their Brownian motion compared to MoS_2 MPs. Furthermore, based on Figure 4(a), W based NFs show higher TC improvement compared to the W/EG based NFs, which suggests that W based suspensions containing MoS_2 NPs are more favorable for use as heat transfer fluids than the similar samples with W/EG base liquid. On the other hand, compared to the W based MFs, W/EG based MFs displayed higher TC enhancement, which revealed W/EG based suspensions containing MoS_2 MPs are more advantageous for use as heat transfer fluids than similar W based suspensions. Therefore, as the base liquid effect, W based NFs exhibited higher efficiencies as heat transfer fluids than the similar suspensions with W/EG base liquid, while W/EG based MFs showed better performance as heat transfer fluids than W based suspensions. Rheological behavior of suspensions was evaluated by measuring the viscosity of NFs and MFs. All MoS_2 NFs and MFs exhibited Newtonian behavior in the test temperature region. The values of increase in viscosity defined as $[(\mu_{nf} / \mu_{bl})-1 *100]$ for the same NP-MP content and temperature are shown in Figure 4(b). μ_{nf} stands for the viscosity of NFs-MFs, μ_{bl} for the base liquid. MoS_2 NFs with both W and W/EG base liquids displayed higher rise in viscosity compared to MoS_2 MFs, which may be ascribed to the large contact/interface area between MoS_2 NPs and W and W/EG as base liquids. Except for water based MFs, for all MoS_2 NFs and MFs, the magnitude of TC enhancement was higher than increase in viscosity. The reverse trend was observed for water based MFs, i.e. the TC enhancement was lower than the viscosity increases. A maximum TC enhancement of 12.4 % with 10.6 % viscosity increase was achieved for W based MoS_2 NFs with 1wt % MoS_2 NPs at 20 ^oC. It should be mentioned that the error was in the range of ± 2% for TC and ± 4% for viscosity measurements, respectively. It shows that this NF system

may have a potential for use in some heat transfer applications where there is no issues related to pressure drops. However, there are no suitable theoretical models reported in the literature that can be directly applied to compare our experimental results. Maxwell's effective medium theory [47] as well as Einstein's law of viscosity [48] are the two common models used to predict TC and viscosity of NFs and MFs, respectively. Although they are suitable for particles of spherical shape, these models were applied for our NFs and MFs systems in order to get an approximate predicted theoretical value. The results reveal that both Maxwell effective medium theory and Einstein law of viscosity underestimate the TC and viscosity of NFs/MFs changes, respectively. Although there are some reports in the literature reporting models and mechanisms to predict the viscosity and TC of NFs, respectively [49-50], due to different conditions including dissimilar morphologies, those models and mechanisms are not applicable to our presented NFs systems containing MoS_2 particles. The problem is common to all NF-MF formulations prepared by the two-step method, especially if the particles are synthesized by high temperature gas-phase synthesis. This reveals the serious need to develop specific models applicable for these kinds of NF and MF systems. Zeng et al [39] reported TC of engine oil-based NFs containing 1wt % 50 and 100 nm stearic acid capped MoS_2 NPs, where a TC enhancement of ~17.5 % at 40 °C and 38.7 % at 180 °C have been achieved. Furthermore, their TC enhancement for 1wt % MoS_2 NF at 40 °C is slightly higher than the value obtained at 20 °C for similar NP loading. This minor difference could come from the presence of stearic acid as surface modifier, base

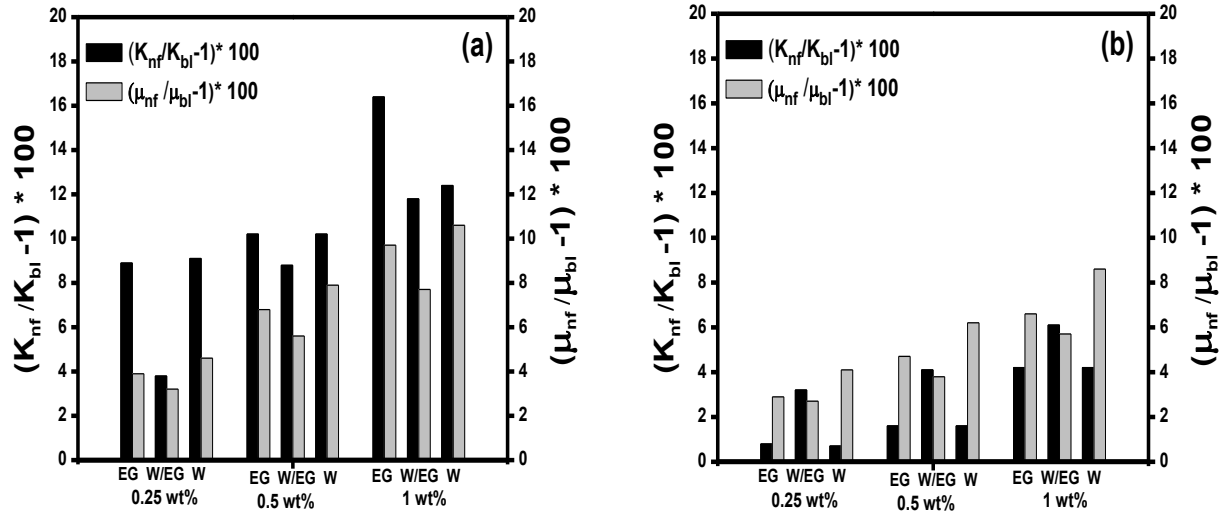

Figure 5. Comparison of TC enhancement (black bars) with increase in viscosity (grey bars) for (a) NFs in W and W/EG containing 0.25, 0.5 and 1 wt% MoS_2 NPs; (b) MoS_2 MFs in W and W/EG containing 0.25, 0.5 and 1 wt% MoS_2 MPs; all measured at 20 °C.

liquids, different test temperatures as well as dissimilar TC measurement method (TPS vs laser flash method). Additionally, different source of MoS_2 particles with varied morphologies including size and shapes, presence of impurities and different surface chemistry may be some other possible reasons for this difference. In another investigation, Su et al [40] reported < 1 % TC enhancement of NFs with both oil (LB2000) and polyhydric alcohol (PriECO6000) with NP loading of 0.5 wt %, which is much lower than the TC values measured for NFs and MFs with the same particle loading. The differences could be attributed to the particle morphology including shape and size and purity of base liquids as well as the surface chemistry of the particles. For an easier comparison, the values of TC enhancement and increase in viscosity of MoS_2 NFs/MFs with different base liquids,

W, W/EG and EG with different MoS_2NP/MPs loading are summarized in Figure 5. Our results on W and W/EG base liquids are slightly lower when compared to EG based NFs and MFs reported earlier. This reveals the importance of base liquid effect on the thermophysical properties of suspensions.

4. Conclusions

We fabricated and studied the physicochemical and thermophysical properties of W and W/EG based NFs and MFs containing MoS_2 NPs and MPs as potential heat transfer fluids. NFs and MFs were fabricated by dispersing MoS_2 NPs and MPs with varying particle loading (0.25, 0.5 and 1 wt %) in W and W/EG base liquids. SEM analysis revealed an average primary particle size of ~90 nm for MoS_2 NPs and ~1.2 μm for MoS_2 MPs, respectively. Due to the presence of MoS_2 NPs-MPs, TC enhancement over the base liquids was achieved for all MoS_2 NFs and MFs. Their rheological behavior was evaluated by measuring the viscosity, revealing Newtonian behavior in the tested temperature range and showing the increasing viscosity with increased MoS_2 NP-MP loading. Our investigation on the effect of particle size on TC property displayed that MoS_2 NPs (MoS_2 NFs) with both W and W/EG base liquids exhibited higher TC enhancement compared to the dispersions containing MoS_2 MPs (MoS_2 MFs). Moreover, suspensions containing MoS_2 NPs (MoS_2 NFs) in both W and W/EG base liquids, showed higher viscosity increase compared to the suspensions containing MoS_2 MPs (MoS_2 MFs). As the base liquid effect, W based NFs containing MoS_2 NPs exhibited higher heat transfer efficiencies as heat transfer fluids than similar suspensions with W/EG base liquid. On the other hand, W/EG based MFs containing MoS_2 MPs showed improved performance as heat transfer fluids compared to similar suspensions with W base liquid. Among all MoS_2 NFs and MFs in water or W/EG based liquids, water based MoS_2 NF containing 1 wt % MoS_2 NP displayed the most promising results with 12.4 % TC enhancement and ~10.6% viscosity increase at 20 ^oC (base liquid effect). It indicates the potential of this kind of NF for heat transfer applications. Maxwell's effective medium theory as well as Einstein's law of viscosity underestimated the TC and viscosity of NFs/MFs, respectively. It implies the need for develop appropriate models to estimate those properties. Despite the fact that in this work the effect of base liquid and particle size on the thermo-physical characteristics of MoS_2 MFs and NFs were studied, there are still serious scientific gaps which can be filled up with some future works. Study on the effect of broad range of particle concentration (with the use of surfactants/dispersants) on thermo-physical properties of MoS_2 NFs and MFs containing MoS_2 NPs/ MPs and developing proper models for prediction of TC and viscosity of those suspensions are among the work we plan to undertake.

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