Fabrication and thermo-physical properties characterization of ethylene glycol—MoS$_2$ heat exchange fluids

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ARTICLE INFO

ABSTRACT

This study reports on the fabrication and thermo-physical properties evaluation of ethylene glycol (EG) based heat exchange fluids containing molybdenum disulfide nanoparticles (MoS$_2$ NPs) and micrometer sized particles (MPs). For this purpose, MoS$_2$ NPs and MPs (with average size of 90 nm and 1.2 μm; respectively) were dispersed and stabilized in EG with particle loading of 0.25, 0.5, 1 wt%. To study the real effect of MoS$_2$ NP/MP the use of surfactants was avoided and ultrasonic agitation was used for dispersion and preparation of stable MoS$_2$ NFs/ MFs. The objectives were investigation of impact of MoS$_2$ particle size (including NP/MP) and particle loading on thermo-physical properties of EG based MoS$_2$ NFs/MFs including thermal conductivity (TC) and viscosity of NFs/MFs at 20 °C. All suspensions (NFs/MFs) exhibited a higher TC than the EG as base liquid and NFs showed higher TC enhancement values than the MFs. A TC enhancement of 16.4% was observed for NFs containing 1 wt % MoS$_2$ NPs while the maximum increase in viscosity of 9.7% was obtained for the same NF at 20 °C. It indicates this NF system may have some potential to be utilized in heat transfer applications.

1. Introduction

Many research have been directed in the formulation of heat transfer fluids over the previous several decades resulting in the development of currently utilized heat transfer enhancement techniques. Conventional heat transfer fluids such as water, ethylene glycol (EG) and mixtures of them play essential roles in many cooling applications. Traditional heat transfer fluids however suffer from inherently poor thermal characteristics due to low thermal conductivity (TC), which is one of the essential limits for extensive heat transfer applications. Since solid particles have higher TC compared to the conventional heat transfer fluids, dispersion of suspended solid particles may be a reasonable choice to improve heat transfer by the suspensions. Suspension containing micrometer sized particle (MPs) known as microfluids (MFs) have already been demonstrated to improve thermal characteristics of base liquids [1]. However, micrometer sized inclusions are difficult to be kept in suspension limiting its widespread application. Nanofluids (NFs), proposed by Choi [2], in retrospect, are more stable in suspension and that is the reason why this has attracted huge attention for

enhancing thermal characteristics of conventional heat transfer fluids such as water and EG. NFs are colloidal suspensions containing suspended solid nanoparticles (NPs) or other types of nanostructured materials in a base liquid with sizes typically ranging between 1 and 100 nm (at least in one direction). Additional energy transport due to the motions of nanoparticles induced by van der Waals forces, electrostatic forces due to the electric double layer at the surface, stochastic forces and the hydrodynamic force. Stochastic force that gives rise to the Brownian motion and electrostatic forces are typically significant only for small particles, whereas the van der Waals force is high when the suspension is concentrated. NFs have been applied for various heat transfer applications such as in solar collectors, microchannels, automobiles, thermal energy storage, heat exchangers, heat sinks, microelectronics and energy supply and power generation [3–10]. Preparation of stable suspensions containing NPs and MPs (NFs/MFs) is a key engineering step in using these fluids to enhance the TC of conventional heat transfer fluids. Two major techniques are commonly utilized to fabricate NFs; (i) the one-step preparation method [11–12] representing the direct formation of NPs inside the base liquids, and (ii) the two-step

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method [12–14] where pre-synthesized NPs are dispersed in the base liquid. Due to the large scale availability of commercial NPs and MPs, the two-step preparation technique is the most commonly used method. However, on the other hand, presence of some unavoidable impurities (as undesired or secondary phases) in commercial NPs may influence the thermo-physical properties of NFs. In either technique, the fabrication of a uniformly dispersed NP in the base liquid is vital for obtaining desired characteristics of the NFs. So far various nanostructured materials including metallic compounds as well as many ceramic materials including oxides such as Al_2O_3 [15], TiO_2 [16], CeO_2 [17], mesoporous SiO_2 [18], sulfides like MoS_2 [19–20] and carbides such as SiC [21–23] have been used to fabricate NFs for heat transfer applications. Among sulfide nanostructured materials, molybdenum disulfide (MoS_2) is one of the promising material for preparing efficient NFs for cooling purposes. MoS_2 is a kind of layer structured material with demonstrated applications in lubricants and as catalysts for several years due to its unique properties like photocorrosion resistance, anisotropy and chemical inertness [24–26]. Several factors such as composition and loading of NP/MP, stability of suspension, base liquid composition, NF fabrication method and surface modifier may influence the thermo-physical properties of NFs including TC and viscosity [27–28]. Among all factors, although the size of NPs/MPs is probably the most important, affecting TC and viscosity of NFs, which has not been well studied yet. Not only the number of publications studying the effect of particle size on thermo-physical properties of NFs/MFs (including TC and viscosity) is limited, but also there are discrepancies in the reported works. Literature survey revealed that some investigations show TC and viscosity of NFs are increased when smaller NPs is used [29], while others reported that NFs thermos-physical properties improve with increasing particle size [30]. To have a better understanding, we performed a systematic study to investigate the effect of MoS_2 size including NP and MP on thermo-physical properties of EG based MoS_2 NFs and MFs. EG was selected as base liquid since it is one of the most commonly used heat transfer cooling liquids in industries. The MoS_2 nanostructured materials have already been used to improve the lubrication properties of conventional lubricants [31–35]. Afterwards some attempts were carried out to use these nanostructured materials for enhancing thermal characteristics of conventional heat transfer fluids. Recently Zeng et al. [19] reported the application of MoS_2 NF for heat transfer applications where engine oil based (B350) NFs with MoS_2 NPs capped by stearic acid as surface modifier were used as the inclusions. The NPs were spherical in shape with diameter ranging between 50 and 100 nm and they studied the TC of NFs without reporting any result on viscosity of NFs. Su et al. [20] fabricated MoS_2 water-based and oil-based NFs with different NP loading (ranging from 0.01 to 0.5 wt%), and investigated their stability and subsequent TC (using transient hot-wire method at room temperature). They also did not present any results for viscosity of NFs. To fabricate NFs by two step method, Su et al. used commercial MoS_2 NPs with average diameter of 30 nm and pure water, LB2000 vegetable lubricant and PriECO6000 unsaturated polyhydric alcohol ester as base liquids. To the best of our knowledge there is no studies reporting the effect of particle size (MP vs NP) of MoS_2 NPs/MPs on thermo-physical properties when EG is used as base liquid. There is no data in the literature reporting the use of EG base liquid in spite of the fact that it is one of the most commonly used heat transfer fluid with MoS_2 inclusions. We report the fabrication of MoS_2 NFs/MFs by dispersing MoS_2 NPs (with average size of 90 nm) and MPs (with average size of 1.2 μm) at particle loading of (0.25, 0.5 and 1 wt%) in EG base liquid. For the first time, to the best of our knowledge, we report the effect of MoS_2 NPs and MoS_2 MPs (particle size effect) on thermo-physical properties of EG based NFs/MFs including TC and viscosity.

Table 1
EG based MoS_2 NFs evaluated for TC and viscosity properties.

Sample	NP/MP	Base liquid	NPs/MPs loading (wt%)
Nano-MoS_2-EG-0.25 wt%	MoS_2 NP	EG	0.25
Nano-MoS_2-EG-0.5 wt%	MoS_2 NP	EG	0.5
Nano-MoS_2-EG-1 wt%	MoS_2 NP	EG	1
Micro-MoS_2-EG-0.25 wt%	MoS_2 MP	EG	0.25
Micro-MoS_2-EG-0.5 wt%	MoS_2 MP	EG	0.5
Micro-MoS_2-EG-1 wt%	MoS_2 MP	EG	1

2. Experimental

2.1. Materials and methods

Molybdenum disulfide nanoparticles/microparticles (MoS_2 NPs/MPs) and Ethylene glycol (EG) were acquired from Sigma Aldrich (Germany). All the reagents were used as received, without any further purification.

2.2. Fabrication of EG based MoS_2 NF/MF via two-step method

MoS_2 NFs and MFs were prepared by adding a known weight of MoS_2 NPs and MPs in EG as the base liquid and ultrasonic mixing of the suspension was carried out for 20 min. The use of dispersant/additive was avoided to study the actual impact of MoS_2 NPs/MPs. A series of stable MoS_2 NFs and MFs with concentrations of 0.25 wt%, 0.5 wt% and 1 wt% were obtained as summarized in Table 1. All suspensions were found to be stable for at least 96 h without any visual signs of settling down.

2.3. Characterization techniques

Microstructure and morphology of MoS_2 NPs and MPs were evaluated by using Scanning Electron Microscopy (SEM; FEG-HR Zeiss-Ultra 55). Transmission Electron Microscopy (TEM) analysis of the particles were performed using JEOL 2100 at 200 kV acceleration voltage. Nicolet Avatar IR 360 spectrophotometer, in the range of 500–4000 cm^{-1} was used for Fourier Transform Infrared Spectroscopy (FTIR). Powder X-ray diffraction (XRD) was performed in a Philips X'pert pro Diffractometer with Cu Kα source (λ = 1.5418 Å). Average solvodynamic particle size distribution of MoS_2 NPs/MPs was evaluated by Beckmann-Coulter Delsa Nano C system. TC of NFs were measured by using TPS 2500 instrument (HotDisk model 2500), which works based on the Transient Plane Source (TPS) method [36]. Finally, viscosity of NFs was determined by using a DV-II + Pro-Brookfield viscometer.

3. Results and discussion

3.1. X-ray powder diffraction (XRPD)

In order to identify the crystal structure of MoS_2 NPs and MPs, X-ray powder diffraction (XRPD) was carried out. Fig. 1 shows the XRPD pattern of MoS_2 NP and MoS_2 MPs which are almost identical and all major diffraction peaks were indexed for the hexagonal phase of MoS_2, Molydenite (ICDD Ref pattern# 98-002-4000) as marked by the Miller indices.

3.2. Morphology analysis

SEM micrographs of MoS_2 NPs and MoS_2 MPs are shown in Fig. 2(a) and (b), respectively. There is a wide range of morphologies and wide size dispersion for MoS_2 NPs, making it relatively difficult to estimate the size of NPs from the micrograph; nevertheless, a rough estimate revealed the average primary particle size as 90 nm for MoS_2 NP.
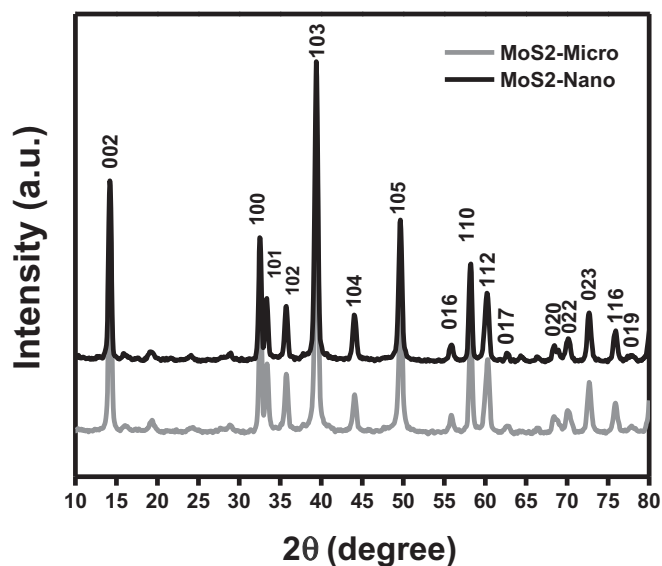
Fig. 1. XRPD pattern of as-received MoS_2 NP and MPs (indexed with ICDD Ref pattern# 98-002-4000).

Similarly, micrographs of MoS_2 MPs show average primary particle sizes of around 1.2 μm. Fig. 2(c) and (d) display the TEM micrographs of MoS_2 NPs and MoS_2 MPs, respectively, where the layered structured shape can be observed for both MoS_2 NPs and MoS_2 MPs.

3.3. Dynamic light scattering (DLS) and Fourier transform infrared spectroscopy (FTIR) analyses

To understand the influence of effective size of dispersed NPs in the liquid media, DLS analysis was carried out to estimate the dispersed size of MoS_2 NPs and MoS_2 MP in EG. The solvodynamic size measured by DLS is defined as "the size of a hypothetical hard sphere that diffuses in the same fashion as that of the particle being measured". The diameter calculated from the diffusional properties of the particle will be indicative of the apparent size of the dynamic solvated particle, as well as agglomerate/aggregates. The results are displayed in Fig. 3(a) where a wide range of particle size distribution (240–2400 nm) with an average peak value of ~950 nm was obtained for MoS_2 NP particles in EG base liquid. For MoS_2 MP, a much wider range of size between 1.8 and 5.0 μm, with an average peak value of ~3 μm was estimated. In both the cases the estimated size is that of the equivalent hard sphere for the anisotropic MoS_2 particles, indicating aggregation/agglomeration of particles in EG base liquid. A comparison between SEM, TEM and DLS results for MoS_2 NP and MoS_2 MP displays that the primary particle size obtained from SEM and TEM micrographs is less than the predicted sizes from DLS method in EG medium. This difference may be due to the aggregation/agglomeration of MoS_2 NP and MP in the EG, besides the adsorbed liquid layer on surface of particles. In order to study the surface characteristics of MoS_2 NP and MoS_2 MP, FTIR analysis was carried out. FTIR spectra for both "as-received" MoS_2 NP and MP are shown in Fig. 3(b). Both NPs and MPs displayed almost identical surface characteristics. The band at $1120\ cm^{-1}$ is assigned to characteristic stretching vibration of Mo—S. Compared to MoS_2 NP spectrum, MP showed organic components with the peaks centered at 2920 and $2856\ cm^{-1}$ that can be attributed to stretching vibration of C—H indicating that the MPs do not have a very clean surface, which may be some contamination or surface exposed organic layers during their fabrication.

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

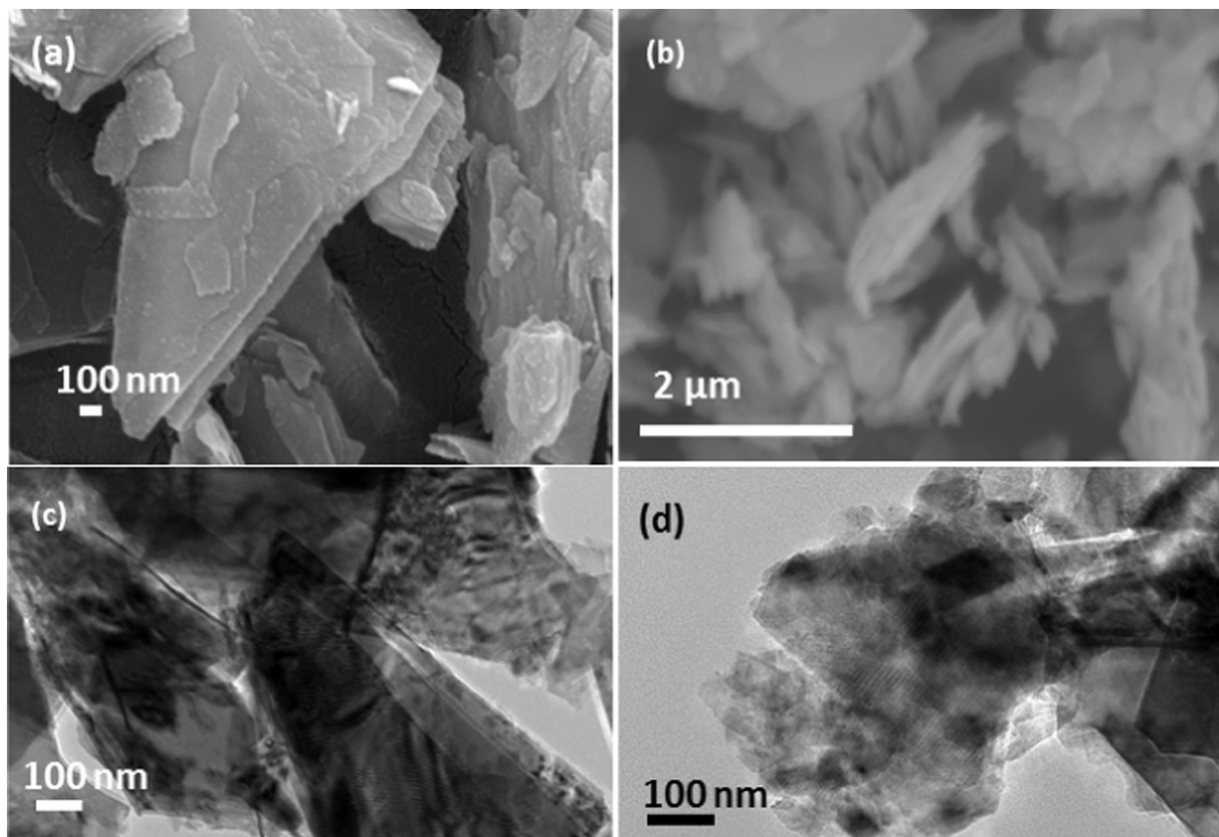TC and viscosity are two major thermo-physical characteristics

Fig. 2. SEM micrographs of (a) MoS_2 NPs and (b) MoS_2 MPs. TEM micrographs of (c) MoS_2 NPs and (d) MoS_2 MPs.
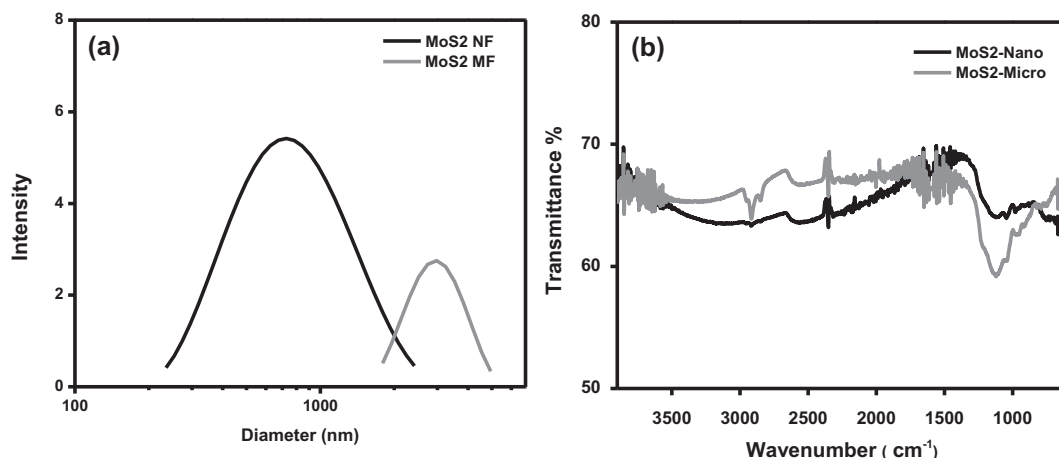
Fig. 3. (a) Particle size distribution of MoS_2 in EG base liquid measured by DLS. (b) FTIR spectra of as-received MoS_2 NPs and MPs.
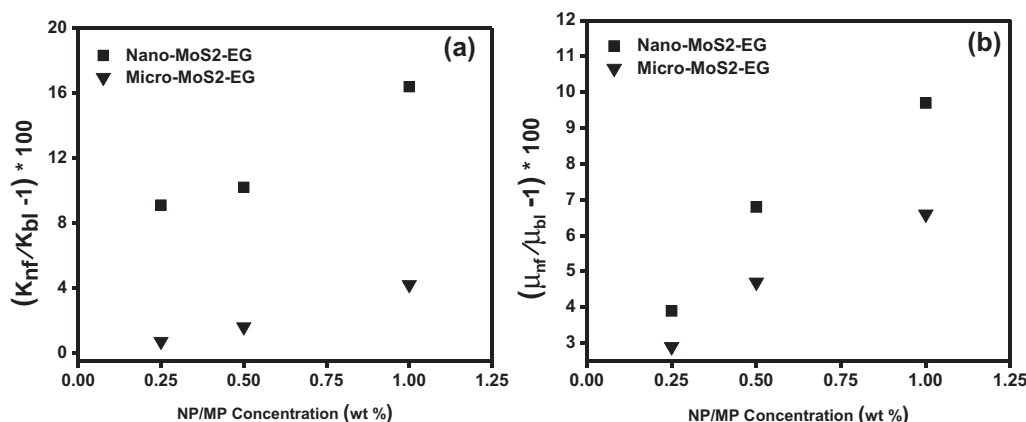

Fig. 4. (a) TC enhancement of MoS_2 NFs and MFs with various loading of MoS_2 NP and MP and (b) increase in viscosity of MoS_2 NFs and MFs with various concentration of MoS_2 NP and MP (at 20 °C).

related to the performance of suspensions as heat transfer fluids. Therefore, these two tests were carried out to evaluate the capability of our fabricated NFs and MFs for use in some heat transfer applications. To ensure having an efficient NF for heat transfer applications, preparation of NF/MF with higher TC enhancement and minimal increase in viscosity (over the base liquid) play an important role [37–38]. TC and viscosity properties were measured at 20 °C. Fig. 4(a) shows the TC enhancement values vs MoS_2 NP loading in the relevant base liquid defined by $[(K_{nf}/K_{bl}) - 1 * 100]$, at 20 °C. K_{nf} and K_{bl} stand for TC of NFs and TC of base liquid, respectively. All NFs (except Micro-MoS_2-EG-1 wt %) displayed higher TC values (K_{nf}) than EG as base liquid indicating enhancement of TC due to the presence of MoS_2 NP. We also measured the viscosity of NFs and its values of increase defined as $[(\mu_{nf}/\mu_{bl}) - 1 * 100]$ at the same particle loadings and temperature and the results are displayed in Fig. 4(b). μ_{nf} and μ_{bl} are the viscosity of NFs/MFs and viscosity of base liquid, respectively. All MoS_2 NFs and MoS_2 MFs exhibited Newtonian behavior at the tested temperature region. Furthermore, all NFs/MFs exhibited higher viscosity values (μ_{nf}) than EG base liquid. NFs containing MoS_2 NPs showed higher TC enhancement compared to the MFs containing MoS_2 MPs for the same particle loading. Both NFs and MFs showed Newtonian behavior in their viscosity, while NP addition in NFs lead to increased viscosity of the fluids in comparison to the MP inclusions in MFs. This can be attributed to arise from the large interface/contact area between MoS_2 NPs and EG base liquid. Among all NFs and MFs containing MoS_2 NPs and MPs, the most promising results were observed for NF containing 1 wt% MoS_2 NP (Nano-MoS_2-EG-1 wt%) with reasonable TC enhancement of 16.4% but with a controlled increase in viscosity by about 9.7%. It may indicate the favorable characteristics of this NF system with capability for use in some heat transfer application. Due to the morphology of MoS_2

NP and MPs used in this work, there are no proper theoretical models/equations to compare our obtained experimental results with the predicted values from the models. Maxwell's effective medium theory [39] as well as Einstein law of viscosity [40] are common models/equations to predict the TC and viscosity of NFs and MFs, respectively. Although they are appropriate for particles with spherical shape, to have an idea about some predicted values we applied them in our NF and MFs systems. The results showed that both Maxwell effective medium theory and Einstein law of viscosity underestimate the TC and viscosity of NFs/MFs, respectively. As mentioned earlier there is no study in the literature reporting on thermo-physical properties of MoS_2 particles with EG as base liquid for heat transfer applications. For this reason, a direct comparison between our findings and the relevant results in the literature is challenging. Zeng et al. [19] studied TC of engine oil based (B350) NFs containing 50 and 100 nm sized MoS_2 NPs capped by stearic acid using an instrument based on laser flash. They reported a TC enhancement of ~17.5% and 38.7% for a NF containing 1 wt% MoS_2 NPs at 40 °C and 180 °C, respectively. Moreover, their TC enhancement value for NF containing 1 wt% MoS_2 NP at 40 °C is a fraction higher than our observed value at 20 °C for the same NP loading. This slight difference could arise due to the difference in base liquids, presence of stearic acid as surface modifier, different TC measurement method (TPS vs laser flash method) as well as different test temperature. Moreover, different composition of MoS_2 particles with varied morphologies including size and shapes, dissimilar surface chemistry and presence of impurities can be some possible reasons for this difference. In another work, Su et al. [20] showed TC enhancement of < 1% for NFs with both LB2000 oil and PriECO6000 polyhydric alcohol at NP loading of 0.5 wt%. Their reported values are much lower than the values we obtained for our fabricated NFs and MFs at the same MoS_2 NP and MP

loading. The possible reasons maybe again be related to the different morphology including size and shape with different base liquid, surface chemistry as well as purity of particles. Thus this study demonstrates that MoS_2 NF can serve as efficient heat transfer fluids for various applications.

4. Conclusions

We presented fabrication and evaluation of highly stable MoS_2 NFs and MoS_2 MFs for heat transfer applications using a two-step preparation method. The NFs/MFs were fabricated by dispersing MoS_2 NPs and MoS_2 MPs at various particles loading of 0.25, 0.5 and 1 wt% in EG base liquid. From SEM analysis an average primary particle size of around 90 nm and 1.2 μm was observed for MoS_2 NPs and MPs, respectively. Thermo-physical characteristics of NFs and MFs including TC and viscosity properties were investigated at 20 °C. For all MoS_2 NFs and MoS_2 MFs enhancement of TC over the EG as base liquid was obtained due to the presence of MoS_2 NPs and MoS_2 MPs. All MoS_2 NFs and MoS_2 MFs displayed Newtonian behavior at the tested temperature region and the viscosity of NFs/MFs increased with increasing loading of MoS_2 NP and MP. Test results on the effect of particle size displayed that MoS_2 NFs containing MoS_2 NPs showed higher TC enhancement compared to the suspensions with MoS_2 MFs containing MoS_2 MPs. Among all EG based MoS_2 NFs and MFs MoS_2, NF containing 1 wt% MoS_2 NP's displayed most promising results with 16.4% TC enhancement with only 9.7% increase in viscosity (at 20 °C). It shows the potential of using this kind of NF in some heat transfer applications.

Acknowledgments

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