



Experimental investigation on the effect of SiO₂ secondary phase on thermo-physical properties of SiC nanofluids



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ABSTRACT

Nanofluids (NFs), wherein solid nanoparticles (NPs) are dispersed in traditional heat exchange fluids, are recognized for improving the performance of traditional fluids by enhancing their thermal conductivity (TC). The presence of impurities or undesired phases in commercial NPs may influence the thermo-physical properties of NFs including TC and viscosity, which makes it difficult to understand the real effect of NPs on heat transport characteristics of NFs. Moreover, the presence of these impurities in commercial NPs is unavoidable and their removal from commercial NPs with no negative impact on composition of NPs is challenging. To study the impact of impurities on thermo-physical properties of NFs a systematic experimental work was performed using commercial α -SiC and SiO₂ NPs as the secondary phase as it commonly co-exists in commercial SiC batches. For this purpose, a series of NFs containing 9 wt% of α -SiC/SiO₂ NP mixture with different content of SiO₂ NPs from 5 to 50% were fabricated and investigated. The results show that as the undesired impurity phase (SiO₂) increases, TC of NFs decreases slightly while viscosity increases dramatically. This may be a sound path to tuning the viscosity of the NFs while the achieved high TC is mildly influenced by the secondary phase.

1. Introduction

Poor thermal characteristic of traditional heat exchange fluids is an obstacle for enhancing the efficiency of heat transfer. The past decade has witnessed extensive research on new technologies for the improvement of the current traditional heat transfer fluids. Nanofluids (NFs) are new type of fluids [1], which are defined as nanotechnology based dispersions wherein solid nanoparticles (NPs) or other types of nanostructured materials are dispersed in base liquids. It has been reported that addition of NPs into the conventional base liquids may improve their thermal conductivity (TC). This characteristic makes NFs attractive for use in some energy related applications such as micro-channels, solar collectors, automobiles, air conditioning and power generation [2–6]. Many choices of traditional heat transfer fluids are used as base liquids to prepare NFs. Water and ethylene glycol (EG) are the most commonly used fluids because of their thermophysical properties as well as reasonable price. Nevertheless, both have drawbacks: Water has relatively high TC value, however it boils at extreme temperatures. On the other hand, although EG has a wider operating

temperature range, its TC is low. Therefore a mixture of water and EG is used to preserve not only the favorable cooling ability of water, but also the wider temperature range for operation of EG for heat transfer applications [7]. For this reason study of NFs with W/EG base liquid has attracted considerable attention [8–16]. Desired NFs for heat transfer application should demonstrate a high TC with minimal increase in viscosity due to addition of NPs [7]. Mainly two major techniques as one-step and two-step preparation methods are utilized for fabrication of NFs. In the one-step method [17–19], NPs are directly synthesized in base liquid, resulting in the formation of NFs in a combined process. In the two-step fabrication method; as most the commonly used technique [20–23], at first NPs are first synthesized, followed by their dispersion and stabilization in the base liquid. There is an advantage for this preparation method which is the large scale availability of NPs from different commercial sources and various suppliers. It means NFs in large volume can be prepared by this process while it is challenging in one-step method. However, due to the large-scale production of commercial NPs some factors such as presence of impurities or undesired phase on commercial NPs is unavoidable. It may result in influencing

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the thermo-physical properties of NFs including TC and viscosity. In other words, the presence of impurity/undesired phase does not reflect the real heat effect of NP composition on transport property yet its presence in the commercial NPs is unavoidable. Moreover, their removal from the commercial NPs with no negative impact on composition of NPs is challenging. Although a large volume of investigations has been carried out to study the impact of different factors on thermo-physical properties of NFs such as NP concentration [24], size [25–27], crystal structure [28], base liquid [29–30], pH of suspension [31], stability [32], temperature [33], surface modification and surfactant [34–35] and preparation method [19,36]. To the best of our knowledge the effect of impurities/secondary phase on thermo-physical properties of NFs has not been studied in detail yet. Moreover, other works reporting on using commercial SiC particles (or suspensions containing commercial SiC particles) [37–42] have not addressed this gap. Therefore, there is a serious need to study the real contribution of NPs (or the impact of impurities) on the thermo-physical properties of NFs. In our recent studies [28–29] using commercial α -SiC NPs we identified the presence of SiO₂ as the secondary (undesired) phase. Extending these studies, which revealed that the impurities may affect the thermo-physical properties of NFs ([28–29]), we aimed at performing a systematic work to study the effect of SiO₂ NP as the secondary phase on the thermo-physical properties of α -SiC-W/EG NFs. Several NFs samples were fabricated by stabilizing 9 wt% of α -SiC/SiO₂ NP mixture with different ratios of SiO₂/SiC NP. Our findings on the effect of SiO₂ NPs on thermo-physical properties of fabricated SiC NFs are presented in detail.

2. Experimental

2.1. Materials and Methods

Alpha Silicon Carbide (α -SiC) particles was purchased from Superior Graphite; USA. Silica (SiO₂) NP, Ethylene glycol (EG, 99.8%) and ammonium hydroxide (NH₄OH, 28% NH₃ in H₂O) were acquired from Sigma Aldrich; Germany. All reagents were used as received, without any purification.

2.2. Fabrication of α -SiC /SiO₂ NF

A series of W/EG based NFs were fabricated by dispersing a known weight of α -SiC/SiO₂ NP mixture with different ratios of SiO₂/SiC NP, where amount of SiO₂ NP varied from 5 to 50%, via two-step method. In order to achieve stable NFs containing 9 wt% α -SiC/ SiO₂ NP mixture the suspensions were sonicated for 15 min at controlled temperature and the pH of samples was adjusted to \sim 9.5 using NH₄OH. All NFs (Table 1) showed desired stability without any precipitation within a week. The use of any surface modifier or surfactant was avoided to study the real contribution of α -SiC/SiO₂ NP mixture on the thermo-physical properties of NFs. Finally, the fabricated NFs were evaluated for thermo-physical properties including TC and viscosity.

Table 1
NFs containing SiC/SiO₂ NP mixture tested for thermo-physical properties.

Sample ID	NP Composition (wt%)	Base Liquid (50:50) by wt%	NP loading (wt%)	pH
SS0	α -SiC/SiO ₂ (100:0)	W/EG	9	\sim 9.5
SS1	α -SiC/SiO ₂ (95:05)	W/EG	9	\sim 9.5
SS2	α -SiC/SiO ₂ (90:10)	W/EG	9	\sim 9.5
SS3	α -SiC/SiO ₂ (80:20)	W/EG	9	\sim 9.5
SS4	α -SiC/SiO ₂ (70:30)	W/EG	9	\sim 9.5
SS5	α -SiC/SiO ₂ (60:40)	W/EG	9	\sim 9.5
SS6	α -SiC/SiO ₂ (50:50)	W/EG	9	\sim 9.5

2.3. Characterization Techniques

Microstructure and morphology of α -SiC and SiO₂ NPs were evaluated by using Scanning Electron Microscopy (SEM; FEG-HR Zeiss-Ultra 55). Transmission Electron Microscopy (TEM) analysis of the particles were carried out 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 (FT-IR) analysis of solid particles and solid/liquid samples. Zeta potential analysis of α -SiC and SiO₂ NPs was done to assess the stability region of NFs; average solvodynamic particle size distribution of particles was estimated by Beckmann-Coulter Delsa Nano C system. TC of NFs was measured by using TPS 2500 instrument (HotDisk model 2500), which works based on the Transient Plane Source (TPS) method. To check the validity of the TPS instrument, a standard source for thermodynamic properties of water (IAPWS reference) [43] was used. Compared to the reference the accuracy of measurement for distilled water was observed within 2% [43]. Finally, viscosity of NFs was measured using a DV-II + Pro- Brookfield viscometer.

3. Results and Discussion

3.1. X-Ray Diffraction (XRD) Analyses

In order to identify the crystal structure of SiC NPs and SiO₂ NP, X-ray powder diffraction (XRPD) analysis was performed. The result for SiC NPs has been reported in our recent work [28]. Based on that hexagonal crystal structure was observed and all obtained peaks were indexed with the JCPDS # 01–073-1663 for hexagonal SiC phase. XRPD analysis showed amorphous structure for SiO₂ NPs.

3.2. Morphology Analysis

Detailed microstructural analysis of α -SiC was performed earlier where α -SiC particles have shown a wide size distribution with primary particle size of 115 ± 35 [28], a typical micrograph is presented in Fig. 1(a). SiO₂ NP (Fig. 1b)) seem to be agglomerated and have very small size, however,

it is hard to perform any size estimate from SEM micrographs. TEM analysis was performed to estimate average primary particle size. TEM micrographs of α -SiC and SiO₂ particles are presented in Fig. 1(d) and (e), respectively. TEM analysis confirms hexagonal morphology for α -SiC particles while spherical morphology with estimated primary particle size in the range of 15 ± 5 nm is obtained for SiO₂ NPs. Selected area electron diffraction (SAED) pattern, presented in inset images in Fig. 1(d) and (e) was indexed for hexagonal crystal structure α -SiC (ICDD no: 01–073-1663) and showed amorphous structure for SiO₂ NP, respectively; in agreement with the result of XRD analysis [28]. Fig. 1(c) displays α -SiC/SiO₂ NP mixture sample, where two different size populations are observed, smaller particles representing SiO₂ and larger ones SiC.

3.3. Dynamic Light Scattering (DLS) Analysis

To study the impact of effective size of dispersed NPs in W/EG media medium, DLS analysis was carried out to estimate the dispersed size of both α -SiC and SiO₂ NPs in liquid medium. The results are shown in Fig. 2(a) for both α -SiC and SiO₂ NP in W/EG base liquid. A particle size distribution between 60 nm and 2000 nm with an average peak value of 520 nm for α -SiC NPs, and particle size distribution between 40 and 450 nm with an average peak value of around 160 nm was measured for SiO₂ NPs in W/EG medium, respectively. In other words, narrower NPs size distribution with a smaller average dispersed size is estimated for SiO₂ NPs. Comparison between TEM/SEM analysis (Fig. 1) and DLS results shows the estimated sizes in W/EG medium is larger than the primary particle size obtained from SEM and TEM

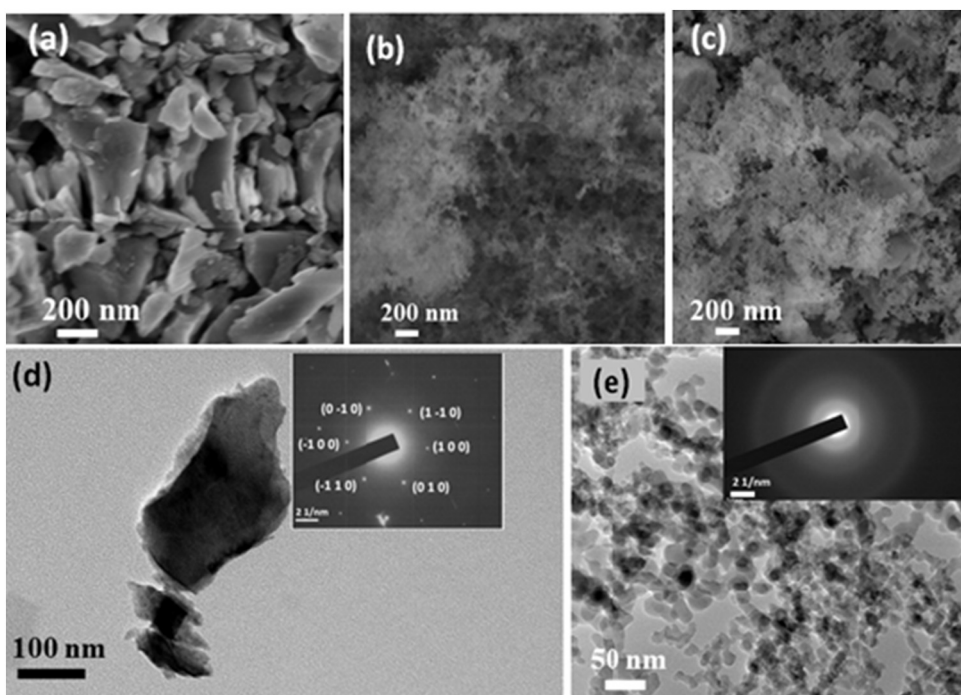


Fig. 1. SEM micrographs of: (a) α -SiC, (b) SiO_2 NPs and (c) α -SiC/ SiO_2 NP mixture; TEM micrographs of: (d) α -SiC NPs and (e) SiO_2 NPs (inset: SAED pattern of α -SiC and SiO_2 NPs).

micrographs. This indicates agglomeration of α -SiC and SiO_2 NP in base liquid has taken place, which is one of the most important factors influencing the stability of NF. Previous studies [44] showed that for fabrication of stable suspensions, pH value of the NF play a key role. To identify the optimum values of pH, zeta potential analysis was performed for both the α -SiC and SiO_2 NPs, in the pH region from 2 to 10. The results are presented in Fig. 2(b), revealing that the highest negatively charged NPs was obtained at pH of ~ 9.5 . Therefore, the pH of the NF samples was adjusted to 9.5.

3.4. Fourier transform infrared spectroscopy (FT-IR) analysis

Since the study of surface characteristics of NPs play important role in suspension characteristics, FT-IR analyses for “as-received” α -SiC, SiO_2 and α -SiC/ SiO_2 NP mixture was carried out. The results are presented in Fig. 3 where the absorption band from 860 cm^{-1} to 760 cm^{-1} is assigned to the Si-C bond. For α -SiC particles, the band at 1200 cm^{-1} as well as the absorption between 1100 cm^{-1} and 1000 cm^{-1} are attributed to the Si-C, Si-O-Si and/or Si-O-C, respectively. Moreover, in the same particle, there are two bands between 1525 and 1620 cm^{-1} , assigned to the C = O groups, which may originate from the residual chemicals or un-reacted precursor residues from the synthesis. The analysis on SiO_2 NP showed typical absorption

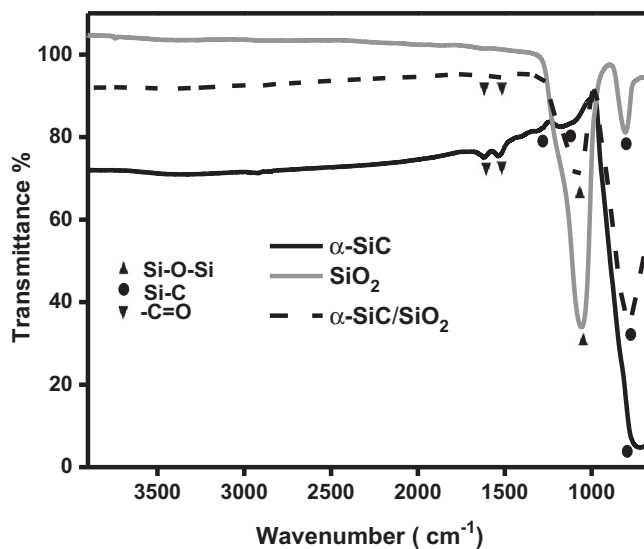


Fig. 3. FT-IR analysis for α -SiC NPs, α -SiC / SiO_2 NP mixture and SiO_2 NPs.

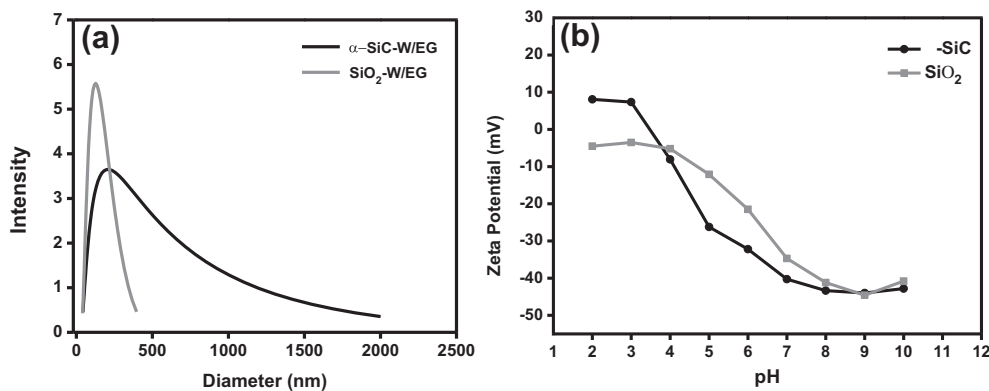


Fig. 2. (a) NP size distribution via DLS for α -SiC and SiO_2 NPs in W/EG base liquid and (b) Zeta potential as a function of pH for α -SiC and SiO_2 NPs.

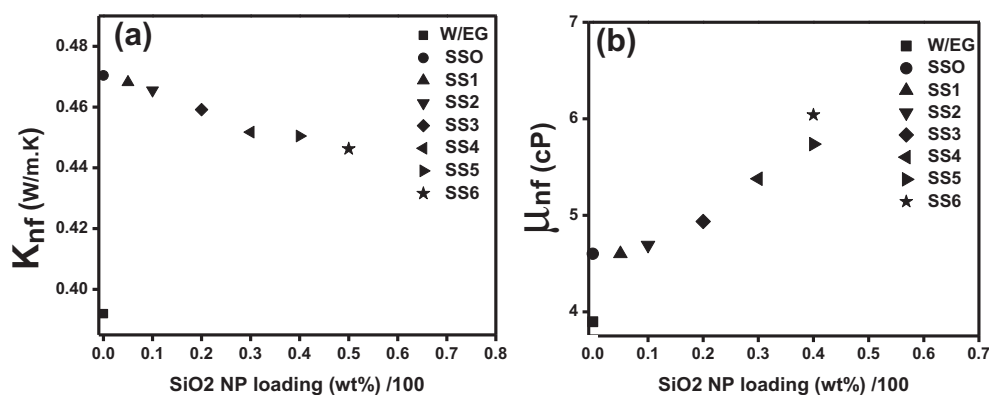


Fig. 4. Thermo-physical properties of W/EG based NFs containing α -SiC/SiO₂ NP mixture vs SiO₂ NP loading: (a) Absolute TC, K_{nf} , (b) absolute viscosity, μ_{nf} (all results are recorded at 20 °C).

bands for OH-terminated SiO₂ as seen from the observed Si–O–Si bands at around 850 cm⁻¹ and 1060 cm⁻¹. To analyze the composite, some NF containing composite α -SiC/SiO₂ NP was dried and then FT-IR analysis was carried out. In the spectrum for the composite, the bands at 850 and 1060 cm⁻¹ are assigned to Si–O–Si and the two bands from 1525 cm⁻¹ to 1620 cm⁻¹ are attributed to the C = O groups, revealing the co-existence of the two phases.

3.5. 3.6. Thermo-physical properties of NFs

TC and viscosity are two significant properties relating to the performance of NFs as heat transfer fluids. Both properties display the same importance in designing NFs for heat transfer applications. In fact fabrication of NFs with higher relative TC and minimal increase in viscosity is desired. In this case one may expect an efficient NF for heat transfer applications. Fig. 4(a) shows the absolute TC values of NFs containing α -SiC/SiO₂ NP mixture vs SiO₂ NP loading. All NFs exhibited higher TC values (K_{nf}) than the base liquid W/EG showing TC enhancement due to the presence of α -SiC/SiO₂ NP mixture. Moreover, as SiO₂ NP loading increases TC value decreases, which is expected due to the fact that SiO₂ has lower thermal conductivity as compared to SiC. The viscosity of NFs was measured at 20 °C and the results are presented in Fig. 4(b). NFs displayed Newtonian behavior revealing the viscosity is independent of shear rate. Moreover, all suspensions showed higher viscosity values (μ_{nf}) than the base liquid W/EG. The results also showed that as SiO₂ NP loading increases, the viscosity value increases. To have a better comparison on thermo-physical properties of NFs, all TC enhancement values defined by $[(K_{nf}/K_{bl})-1] * 100$ and increase in viscosity values defined as $[(\mu_{nf}/\mu_{bl})-1] * 100$ are displayed in Fig. 5. K_{nf} , K_{bl} , μ_{nf} and μ_{bl} stand for TC of NFs, TC of base

liquid, viscosity of NFs and viscosity of base liquids, respectively. The maximum and minimum TC enhancement of 20% and ~14% as well as maximum and minimum increase in viscosity of ~54.5% and ~14% were observed for SS6 and SSO NF systems, respectively. This indicates an important finding of this work: the higher the SiO₂ NP loading, the higher.

the increase in viscosity and the lower the TC, where the impact of increased SiO₂ content is much lower on the TC than the viscosity. In other words, this particular composition with presence of SiO₂ NP as undesired phase, offers NF with tunable thermo-physical properties wherein TC and viscosity values can be tailored by SiO₂ NP as the secondary phase. It indicates the favorable characteristics of this NF system with capability for use in practical heat transfer application where NFs with tunable thermo-physical properties are needed. These kind of NFs may find use in some heat transfer applications such as microelectromechanical system (MEMS) and nanoelectromechanical system (NEMS) based devices where a desired level of TC enhancement are required [45]. Since there is no report in the literature on similar NF systems presented in this study, a direct comparison between the presented results and other published work is not possible. A comparison shows that the TC enhancement value for the worst case in this study (sample SS6 containing 50% secondary phase; SiO₂ NP) is around two times greater than the value reported by Lee et al. [37] for a water based SiC NF at nearly the same SiC NP loading. It indicates that our fabricated NF is more beneficial for heat transfer application. The reason is not clear but besides NP composition, different base liquids (base liquid effect) may be the dominant cause. Li and Zuo [41].

fabricated W/EG based (60:40 volume ratio) SiC NFs containing 1 vol% particle (using PVP as surface modifier) and reported a TC enhancement of 33.84% at 20 °C. The difference may be related to the different ratio of water/EG mixture, presence of PVP, different TC measurement method (Transient Hot Wire method) or different crystal structure or composition of SiC particles with different sizes and purities. Timofeeva et al. [42] reported on W/EG based SiC NFs containing 13 wt% α -SiC NPs loading with ~15% TC enhancement and 35% increase in viscosity. Although SS6 sample in this study (as the worst case) showed almost the same TC enhancement, exhibited ~20% more increase in viscosity. It shows how the SiO₂ NP loading as an undesired phase or impurity can dramatically alter the viscosity of these kinds of NFs systems, while mildly influencing the TC. In other words, these results revealed how SiO₂ NP loading can effectively contribute for tailoring the thermo-physical properties of NFs where particular NF with tunable thermo-physical properties is needed.

4. Conclusions

We presented on the fabrication and evaluation of highly stable NFs fabricated using α -SiC/SiO₂ NP mixture for heat transfer applications, which were fabricated by stabilizing SiC/SiO₂ NP mixture in W/EG media, using colloidal stabilization strategies. A detailed physico-

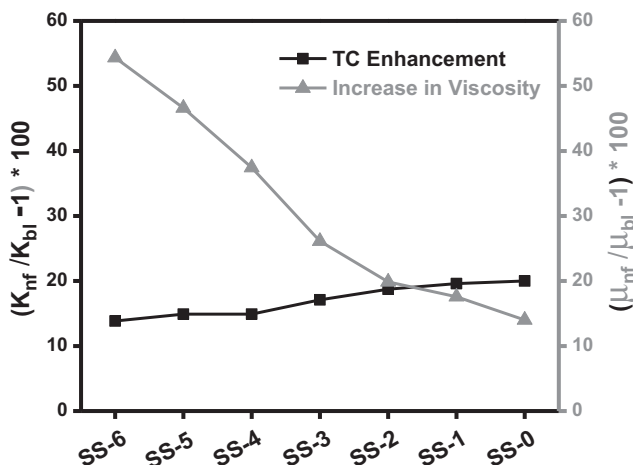


Fig. 5. TC enhancement vs. increase in viscosity values for W/EG based NFs containing α -SiC/SiO₂ NP mixture (all results are recorded at 20 °C).

chemical evaluation showed different characteristics of SiC/SiO₂ NP mixture, including, primary/dispersed particle size and surface chemistry. Thermo-physical properties of NFs including TC and viscosity were measured at 9 wt% NP mixture loading. TC enhancements as well as viscosity increase of the NFs over the base liquid, due to the presence of SiC/SiO₂ NP mixture were observed. As the SiO₂ NP loading increased the viscosity of NFs increased dramatically while the TC changed moderately. It displays effective contribution of undesired SiO₂ NPs to viscosity in order to fabricate a SiC NF with tunable thermo-physical properties. Among all the samples, NF containing α -SiC (free of SiO₂ NP) exhibited the highest TC enhancement by 20%, while only 14% increase in viscosity, while the NF with highest SiO₂ content displayed 14% TC enhancement with 54% increase in viscosity. It displays promising characteristics of this NF system with capability for use in practical heat transfer application where a NF with tunable thermo-physical properties is needed; for instance, some technological applications such as nanoelectromechanical system (NEMS) and microelectromechanical system (MEMS) based devices.

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