Thermo-physical evaluation of dielectric mineral oil-based nitride and oxide nanofluids for thermal transport applications

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Abstract
The thermal, physical and morphological characteristics of dielectric insulating oil - based nanofluids were investigated. The nanofluids were produced by the two-step method, homogeneously dispersing AlN and TiO2 nanoparticles within insulating mineral oil (MO). The filler fraction of the nanofluids were 0.01, 0.10 and 0.50wt.%. Thermo-physical properties such as thermal conductivity and viscosity were measured. A comparison of two produced nanofluids: non-modified surface nanoparticles, and modified nanoparticles with oleic acid (OA) is presented and shown to improve stability while preserving thermal properties.

Keywords : Nanofluids, Dielectric mineral oil, Thermal conductivity, Viscosity

1. Introduction

Nanofluids are highly effective heat transfer media attributed to its anomalous high thermal conductivity. Solid structures possess high thermal conductivity, compared to conventional fluids, by several orders of magnitude. The incorporation of these reinforcing structures within heat transfer fluids results in higher thermal conductive mixtures that could improve performance and efficiency on various systems (Jaime Taha-Tijerina et al., 2012). However, care must be taken to maintain undesirable properties, such as viscosity and electrical conductivity without significant increase (Jaime Taha-Tijerina et al., 2012; Jaime Taha-Tijerina, Sakhavand, Kochandra, Ajayan Pulickel, and Shahsavari, 2017).

Studied nanofluids are mainly produced within water, ethylene-glycol (EG), and other oils and mixture-based solutions. Nevertheless, little research has been conducted on dielectric fluids and other lubricants for thermal management applications (Du and Li, 2014; J. Taha-Tijerina et al., 2013; Jaime Taha-Tijerina et al., 2012; Taha, Narayanan, Ajayan, Contreras, and Rodriguez, 2015; Yuefan Du et al., 2012). On specific fields for instance, in high voltage power transmission systems there is a need to dissipate the generated heat from core and windings in an efficient cost-effective manner. Dielectric mineral oil (MO) acts as a coolant, also playing a critical role as dielectric insulation. The heat in these devices is mainly transferred through convective circulation; therefore, large increment in MO viscosity should be avoided to interfere with inner mechanisms on the apparatus. Heat transfer enhancement in such systems is essential from the perspective of energy savings.

Nanofluids could be used for numerous fields and engineering applications such as medical, energy storage, transportation, microelectronics, reducing pollution, space and defense, tools wear and friction reduction, power transmission systems and nuclear systems cooling, etc. (J.E. Contreras, Rodriguez, and Taha-Tijerina, 2017; José E. Contreras, Rodriguez, and Taha-Tijerina, 2018; M. Mohan et al., 2013; Pena-Paras et al., 2018; Jaime Taha-Tijerina et al., 2019; US20140077138A1, 2014; Jose Taha-Tijerina et al., 2018; Jaime Taha-Tijerina, 2018).

Analytical and experimental research on convective heat dissipation of nanofluids were conducted by several research groups (Ma, Kumar, Kuchibhotla, and Banerjee, 2018; Mohan et al., 2013; J. J. Taha-Tijerina et al., 2014; Zheng, Tan, Zhang, Wang, and Meng, 2017). Significant improvements in heat transfer were observed for diverse nanofluid
systems. Furthermore, thermal conductivity enhancements showed temperature-dependence behavior, increasing the thermal performance as temperature is raised, which makes the nanofluids more suitable for application at elevated temperatures (Du and Li, 2014; Jiang et al., 2014; Jaime Taha-Tijerina et al., 2012; Wu, Zhu, Wang, and Liu, 2009).

Aluminum nitrides (AlN) and Titanium oxides (TiO$_2$) are useful nanoparticles applied in many fields due to their heat exchange characteristics. For instance, Hu et al. (Hu, Shan, Yu, and Chen, 2008) investigated the thermal transport performance of AlN/ethanol system, results at room temperature showed a considerable increase of 20% in thermal transport with 4.0vol.% of AlN. Moreover, in this investigation it was also identified a strong temperature dependence of the thermal conductivity behavior. Yu et al. (Yu, Xie, Li, and Chen, 2011) investigated thermal conductivity behavior of AlN reinforcing two conventional fluids, ethylene glycol (EG) and propylene glycol (PG). It was found an improvement in thermal conductivity of 39 and 40% for EG and PG, respectively, having the same particle size (average ~ 160nm) and nanoparticles concentration. On investigations by Choi et al. (Choi, Yoo, and Oh, 2008) the effect of AlN nanopowders dispersed within insulating oil with oleic acid (OA) as surfactant was analyzed. It must be noted that in some cases, the use of surfactants might decrease the thermal conductivity performance of nanofluids, since surfactants could introduce defects at the interfaces (Xie and Chen, 2011). Nevertheless, an enhancement of 8% in thermal conductivity at 0.50vol% of AlN nanofluid was shown, furthermore an enhancement of 20% of the overall heat transfer capacity was achieved.

On the other hand, Ghadimi et al. (Ghadimi and Metselaar, 2013) investigated the stability of TiO$_2$ nanosuspensions, comparing the effect of surfactant addition and sonicating processing. The highest performance of thermal conductivity was with 3 h. of water bath sonicating and addition of 0.1wt.% or surfactant, which also reflected as the most stable suspension. It was observed that longer duration of sonicating process will cause agglomeration of the reinforced nanostructures.

In this research, AlN and TiO$_2$ nanoparticles are homogeneously dispersed within MO following a two-step method. To avoid high increments in viscosity and due to previous work (Jaime Taha-Tijerina et al., 2012), the chosen filler fractions were 0.01, 0.10 and 0.50wt.%. Additionally, the use of oleic acid (OA) as a surface modifying agent for the nanoparticles was studied. The nanofluids were thermo-physically characterized by measuring thermal conductivity and dynamic viscosity. Thermal conductivity was measured using a KD2 Pro device (Decagon Inc.); temperature-dependent measurements were performed using a thermal bath, and samples were thermally equilibrated before each measurement. Viscosity was measured with a Brookfield DV-E rotating spindle viscometer. For the morphological characterization, dry samples of the dispersions were analyzed by scanning electron microscopy (SEM).

2. Experimental Details

2.1. Nanofluids

For nanofluids preparation, the two-step method is the most used due to its low cost, and compatibility with many nanoparticles. Dry nanopowders of AlN (< 40 nm) and TiO$_2$ (10 – 30nm) were obtained from SkySpring Nanomaterials, Inc. Dispersions were done by homogeneously dispersing the dry nanopowders within MO, extensive water bath sonication (4 - 6 hrs.) was performed under controlled temperature (< 35°C), resulting in stable mixtures with no apparent sedimentation for a few days (~ 4 - 5 days).

2.2. Nanoparticle Surface modification technique with OA

Previous investigations revealed that OA treatment on nanoparticles promoted excellent dispersion among the working fluid. OA has also been used by researchers to modify (or coat) the nanoparticles surfaces and achieve stable oil-based nanofluids (Parametthanuwat and Rittidech, 2013; Primo, Garcia, and Albarracín, 2018). Murshed et al. (Murshed, Leong, and Yang, 2005) observed that low concentration (≤ 0.02vol.%) of OA or a cationic surfactant, as dispersants could greatly improve the dispersion stability of TiO$_2$ nanofluids, without reducing the thermal conductivity.

In our research a surface modification technique was performed on the nanoparticles. Nanopowders were first dispersed within OA at a 20wt.% and sonicated for 45 minutes. The dispersion was then centrifuged to obtain supernatant and separation of nanoparticles from the excessive OA. The remaining nanoparticles, still wet in OA, were rinsed with ethanol and centrifuged 3 more times, removing the excessive top layer of liquid in every step. The product was taken out from the centrifugation tubes and left to dry in open 40ml vials inside of a dust-controlled chamber for one week.
The result was a solid mass of weakly clogged powder that was grinded, re-dispersed within MO at various concentrations, and finally extensively sonicated to assure homogeneous dispersion of the nanoparticles. Figure 1 shows a comparison between nanofluids with non-modified and modified (surface treatment) nanoparticles after one week of being produced.

![Fig. 1](image)

**Fig. 1** Nanofluids a) AlN at 0.50wt.%, b) TiO2 at 0.50wt.%, without OA treated nanoparticles (black caps) and with OA treated nanoparticles (white caps) after four weeks of being produced.

The produced nanofluids with the modified nanoparticles are clearly more stable and resistant to sedimentation. It is observed that this improvement is more significant in the AlN-nanofluid. The reduction in sedimentation rates of nanoparticles is not only attributed to the surface modification, but also due to a density gradient created during centrifugation. The heavier (and largest) nanoparticles and agglomerates are left behind in the centrifugation tubes, while only the smallest particles are used for the further production of nanofluids.

### 2.3. Nanostructures morphological characterization

Nanoparticles morphology and dispersion quality of the nanofluids were investigated by using a JEOL JSM-6010PLUS/LA system. It was observed that increasing the filler fraction of either AlN or TiO2 nanoparticles without functionalization, the size of the agglomerates also increased. However, when the AlN nanoparticles were functionalized with OA as a surface modifying agent, agglomeration did not occur in the same way, even at the highest concentration (0.50wt.%) the agglomeration tends to be smaller and less spherical for the functionalized nanoparticles.

For the suspended particles analysis, a high Vacuum Tescan Vega3 SEM with encapsulated liquid analysis (QX-102, Quantomix Ltd.) was used. The liquid sample preparation consists of 15 µl taken from the supernatant placed into the sample holder for liquids and then sealed according to the procedure. The analysis of samples was performed using an acceleration voltage of 20kV and backscattered electron detector (BEC).

It was observed from the AlN nanofluids evaluation that OA functionalization allowed greater number of suspended nanoparticles, producing more uniformity. Figure 2 shows the 0.50wt.% AlN nanofluid with and without OA functionalization. Figures 2a and 2b show submicron particles with irregular sizes and morphologies. The average size of agglomerates of particles measured for of AlN treated with OA is 476nm ± 120nm (Figure 2a) and for AlN without treatment corresponded to 515nm ± 108nm (Figure 2b). From the AlN nanofluids analysis, it can be concluded that the nanofluid with OA functionalization prevent agglomeration allowing a greater number of nanoparticles being stable in the suspension and reducing the standard deviation of the particle size which produced more uniformity.

Similarly, Fig. 3 shows the 0.50wt.% TiO2 nanofluid with and without OA functionalization. It was observed that unfunctionalized nanofluids presents high. It is observed in Fig. 3b that the OA-treated sample consists of larger submicron particles with irregular morphology and larger sizes. The particles were measured in a range of 450 nm to 714 nm, with and average particle size of 452nm ± 75m. The increment of average size can be attributed to the micelle formed around the surface of the AlN and TiO2 nanoparticles, by the OA molecules.
2.4. Thermal conductivity characterization

Thermal conductivity measurements on AlN and TiO₂ nanofluids at various filler fractions were carried out according to the transient hot-wire (THW) technique, with a KD2 Pro device. Temperature-dependent measurements were performed using a thermal water bath. Samples were thermally equilibrated for at least 10 minutes before each measurement. The measured values are compared with the base fluid (MO) thermal conductivity (k₀). An additional validation test was performed on EG, resulting in a maximum deviation from reported values of up to 2% for temperature of 323K. A minimum of 10 measurements were taken for each set of experiments to report averaged values with standard deviation as error bars.

2.5. Viscosity characterization

Viscosity was measured with a Brookfield DV-E rotating spindle viscometer. A temperature control enclosure for small samples (~ 16 ml) and the S-18 spindle were used. Precise temperature in the samples was achieved by recirculating water from a constant temperature bath. This temperature was monitored with an accuracy of 0.1K.

3. Results and discussions

THW technique is used with KD2 probe device since it is a very common methodology to evaluate fluids thermal conductivity, and it is easy to apply. While results obtained with it are more reliable at room and lower temperatures, some authors confirmed accurate results were obtained also at higher temperatures (up to 323K) (Das, Putra, Thiesen,
and Roetzel, 2003; LotfizadehDehkordi et al., 2013; Jaime Taha-Tijerina et al., 2012; Yang and Han, 2006). According to our experiments, thermal conductivity above 333K have low reproducibility and very high variation, due to unstable fluid, i.e. molecules excitation. These large variations in data between replicated experiments lead to uncertainty. Precautions must be taken for the use of reported high temperature coefficients, and therefore, even though coefficients were obtained up to 353K, here are reported only up to 333K.

Figure 4 shows the temperature-dependent thermal conductivities of both AlN/MO and TiO2/MO nanofluids at various filler fractions and temperatures. The nanofluids showed a temperature-dependent variation, indicating the role of nanoparticles in thermal conductivity (LotfizadehDehkordi et al., 2013; Jaime Taha-Tijerina et al., 2012). Effective thermal conductivities ($k_{eff}$) of nanofluids increased with temperature (measurements were made from room temperature ~298K up to 333K), indicating the role of Brownian motion on measured thermal conductivities. The thermal conductivity of the nanofluids showed enhancements, compared to base fluid, up to 16% and 11% for TiO2 and AlN, respectively. (Fig. 4). Improvement was found to be higher for the nanofluids with the highest fractions of nanoparticles.

Low mass fractions of nanoparticles were chosen in order to avoid significant enhancements in rheological properties. High viscous fluids result in a lower convective heat transfer performance, particularly when in natural convection. Dynamic viscosity measurements showed a reduction compared to base fluids for the two lowest concentrated nanofluids (0.01 and 0.10wt.%), and an increase of up to 22% for the highest concentrated nanofluid (0.50wt.%), as seen in Fig. 5.
Several models can be found in the literature to predict the viscosity of nanofluids for different concentrations of nanoparticles (Azmi, Sharma, Mamat, Najafi, and Mohamad, 2016; S. M. Sohel Murshed and Estellé, 2017). These models have evolved from the classical model of Einstein to more complicate ones that consider the effect on the viscosity due the Brownian motion or the interaction of the particles. However, these models need to be adjusted by correction parameters that are determined experimentally. Therefore, these models are limited to predict satisfactorily the viscosity of the nanofluids for a specific range of nanoparticles, base fluid, temperatures and operating conditions.

Most of the models and experiment reported in the literature coincide that there is an increase of the thermal conductivity in a nanofluid due to the addition of nanoparticles in a fluid base. However, more research is needed to explain the reasons of this important enhancement in the thermal conductivity of the nanofluid. Some of the theories indicate that the increase of the thermal conductivity of the nanofluid can be attributed to a combination of different mechanisms such as the Brownian motion, thermal diffusion of the nanoparticles and the nano-convection and micro-convection (Azmi et al., 2016).

Several models have been developed to predict the effective thermal conductivity of the nanofluids. The models have evolved from the theoretical model of Maxwell to more sophisticated models that consider the shape, size and concentration of the nanoparticles, the Brownian motion, the temperature of the fluid and the chemical surface interaction of the nanoparticles and the fluid (Azmi et al., 2016). However, experimental information and semi-empirical factors are required in order that these models provide good results.

Figure 6 shows viscosity measurements results of MO and high concentrated (0.50wt.%) AlN nanofluid with and without OA. It can be seen that OA functionalization of nanofluids reduces their viscosity.

![Dynamic Viscosity of MO and AlN Nanofluid](image)

**Fig. 6** Measured dynamic viscosity of MO, 0.50wt.% AlN nanofluid and OA treated 0.50wt.% AlN nanofluid at various temperatures.

The addition of OA in the preparation of the nanofluid helps to prevent the agglomeration of the nanoparticles increasing its stability. The nanoparticle surface is coated or modified in order that when a shear stress is applied to a nanofluid, the bond between the nanoparticles can be broken more easily, reducing the viscosity of this fluid compared with the one without OA treatment (Ghasemi, Fazlali, and Mohammadi, 2018). A nanofluid without a surfactant such as OA, there is a stronger bond between the nanoparticles and a higher shear rate is needed to break these bonds, resulting in a higher value of viscosity.

6. Conclusions

The produced nanofluids have the potential to be used as dielectric insulation material for high voltage power transmission systems, such as electrical transformers.

The surface modification technique for the AlN and TiO₂ nanoparticles resulted successful for improving stability of the highest concentrated nanofluids without affecting thermal conductivity. The whole process of creating an OA layer...
in the nanostructures made a desired effect of homogeneous dispersion and less compact agglomerates, therefore, less sedimentation. This improvement was more notorious for AlN nanoparticles. For instance, viscosities of the 0.01wt.% nanofluids were lower than that of MO and even at the highest concentrations of nanoparticles viscosity enhancement was quite low (< 10%).

High thermal conductivities were obtained from the nanofluids compared to MO performance. Despite of the lack of dielectric tests performed in this work, enhancements in electrical conductivity of the oil can be discarded owing to the choice of low mass fractions of ceramic and oxide nanoparticles. Even though, the filler fraction of nanoparticles dispersed within MO were low compared to conventionally studied nanofluids, the enhancement in thermal conductivity was significant and prominent at higher temperatures, ~12% and 15% for the 0.50wt.% AlN and TiO₂ nanofluids, respectively.

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