



Effect of oxide nanoparticles on the thermal, rheological and tribological behaviours of refrigerant compressor oil: An experimental investigation



S.S. Sanukrishna^{a,b}, S. Vishnu^b, T.S. Krishnakumar^{a,b}, M. Jose Prakash^{b,*}

^a University of Kerala, Kerala, India, 695034

^b Department of Mechanical Engineering, TKM College of Engineering, Kollam, 691005, Kerala, India

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ABSTRACT

Oxide based nanolubricants are prepared by dispersing TiO₂, SiO₂ and Al₂O₃ nanoparticles into synthetic refrigerant compressor oil (Polyalkylene glycol) using two-step method. Thermal conductivity of nanolubricants for 0.07 to 0.6% volume fractions in the temperature range 20 °C–50 °C and rheological properties in the range 20 °C–80 °C have been investigated. The tribological characterization is performed with a four ball tribo-tester. The results show that as the volume fraction increases thermal conductivity and viscosity of nanolubricants increase. The maximum increase in thermal conductivity ratio for TiO₂, SiO₂ and Al₂O₃ nanolubricants are 1.38, 1.31 and 1.48, respectively and corresponding increase in viscosity ratios are 5.64, 10.34 and 9.71, respectively. Rheological studies show that, unlike pure lubricant, nanolubricants exhibit non-Newtonian shear thinning behaviour. The tribological test results disclose that, lubricant appended with TiO₂ and SiO₂ have excellent friction reduction and anti-wear properties compared to pure lubricant.

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Étude expérimentale sur l'effet des nanoparticules d'oxyde sur le comportement thermique, rhéologique et tribologique de l'huile du compresseur frigorifique

Mots-clés: Nanolubrifiant; Conductivité thermique; Rhéologie; Tribologie; Coefficient de frottement

1. Introduction

The enhancement of thermal conductivity of fluids by the dispersion and stabilization of solid particles was established by Maxwell (Levin and Miller, 1981a, b) more than a century ago. The nanofluids are proposed as the next generation heat transfer media due to the fact that their thermal transport properties are significantly higher than those of the base liquids (Eastman et al., 1997). Nanolubricant is a new class of lubricant produced by dispersing nano-sized particles of metals, metal oxides, carbon and its allotropes into conventional lubricating oils. Addition of chemical compounds, commonly known as additives, has

been a well-known practice to enhance the thermal and tribological characteristics of coolants and lubricants (Bobbo et al., 2010). Fine solid metal particles have also been employed as anti-friction and anti-wear additives to oils for enhancing their thermophysical and tribological properties. The development of nanotechnology has opened up new avenues in diverse fields including lubrication (Celen et al., 2014). Few studies are reported in the literature regarding the thermophysical, rheological and tribological characteristics of nanolubricants. According to the investigations that have been conducted, presence of nanoparticles in oils may enhance their lubricating properties compared to base fluid; this would, in turn, result in increase in the durability of the components (Kole and Dey, 2013; Akhavan-Behabadi et al., 2015; Esfe et al., 2014; Rajendhran et al., 2018). Thermal conductivity and viscosity are the thermophysical characteristics of nanofluids that have been studied by many investigators and shown that, the thermal conduc-

* Corresponding author.

E-mail address: josetkm@tkmce.ac.in (M. Jose Prakash).

Nomenclature

English symbols

m	mass of nanoparticles [g]
k	thermal conductivity [$\text{Wm}^{-1}\text{K}^{-1}$]
T	temperature [$^{\circ}\text{C}$]
C_p	specific heat [$\text{kJ kg}^{-1}\text{K}^{-1}$]

Greek symbols

φ	volume fraction [%]
μ	dynamic viscosity [cP]
ρ	density [kg m^{-3}]
τ	shear stress (dyne $(\text{cm})^{-1}$)
γ	shear rate (s^{-1})

Subscripts

bf	base fluid
eff	effective
np	nanoparticle

Abbreviations

COP	coefficient of performance
HFC	hydrofluorocarbon
PAG	polyalkylene glycol
TEM	transmission electron microscope
SEM	scanning electron microscope
EER	energy efficiency ratio

tivity increase with increase in volume fraction and temperature (Duangthongsuk and Wongwises, 2009; Buonomo et al., 2015). Viscosity plays an important role in the rheological behaviour of fluids (Afrand et al., 2016; Akbari et al., 2017). Temperature, nanoparticles loading and particle size can affect viscosity, thus the rheological behaviour of nanofluids. The viscosity of nanofluids increases with increase in volume fraction and particle size and a reverse trend is observed with increase in temperature (Esfe and Saedodin, 2014; Serebryakova et al., 2015; Bobbo et al., 2012). Rheological behaviour of nanofluids is quite different from each other and a specific behaviour cannot be established for all nanofluids. Viscosity of Ag-heat transfer oil was studied by Aberoumand et al. (2016) and their results indicated that the base fluid which exhibits Newtonian behaviour changes in to non-Newtonian while adding even small amounts of nanoparticles. Addition of nanoparticles in refrigeration systems results in remarkable improvement in thermophysical, and heat transfer capabilities which in turn enhances the efficiency and reliability (Sanukrishna and Prakash, 2017; Sozen et al., 2014). In refrigeration systems when the refrigerant is circulated through the compressor it carries traces of nanolubricant so that the other parts of the system will have nanolubricant - refrigerant mixture. Kedzierski (2001,2014) shown that lubricant viscosity significantly influences the boiling characteristics of refrigerant/lubricant mixtures. There are also some studies in the literature reporting the performance improvement of refrigeration systems that use nanolubricants (Subramani and Prakash, 2011; Kumar and Elansezhian, 2012; Sharif et al., 2017). Studies show that addition of Al_2O_3 in compressor oil increases the freezing capacity and COP. Wang et al. (2010) showed that the Energy Efficiency Ratio(EER)of residential air conditioners can be increased by 6% if the polyolester oil is replaced with NiFe_2O_4 -nanolubricant. In another study, Sabareesh et al. (2012) explored the effect of dispersing low concentration of TiO_2 nanoparticles in the mineral oil based lubricant. They investigated the effect of nanoparticles on viscosity, lubrication qualities, and the performance of refrigeration systems utilizing R12 (Dichlorodifluoromethane) as the working fluid and reported that average heat transfer increases by 3.6%,

Table 1

Properties of nanoparticles.

Property	SiO_2	Al_2O_3	TiO_2
Average particle size, nm	20	13	21
Density, $\text{g}(\text{cm})^{-3}$	2.6	3.8	4.26
Purity,%	99.8	99.9	99.5
Molecular Wt., $\text{g}(\text{mol})^{-1}$	60.08	101.96	79.87
Thermal conductivity, $\text{Wm}^{-1}\text{K}^{-1}$	1.4	36	11.8
Melting point, $^{\circ}\text{C}$	2230	2040	1850

compressor work reduces by 11% and consequently COP increases by 17%. The investigation of Chin \bar{a} s and Spikes (2003); Rapoport et al. (2003); Liu et al. (2004); Wu et al. (2007); Ginzburg et al. (2002); Hu et al. (2002); Xiaodong et al. (2007) and Tao et al. (1996) revealed the mechanism of action of nanoparticles in lubricating oil. Lee et al. (2009) conducted experiments on the lubrication characteristics of refrigerant oil containing fullerene nanoparticles and reported its friction reduction capability. Zhang et al. (2014) studied the tribological properties of graphene and Multi Walled Carbon Nanotubes (MWCNTs) as additives in diamond-like carbon/ionic liquids hybrid films indifferent lubricating states. The results indicated that MWCNTs and graphene show different nano-scale tribological mechanisms and lubricating effect on the hybrid films. Rasheed et al. (2016) investigated the performance of graphene basednanolubricant in a 4-stroke IC engine and the test results provided understanding about the interaction between graphene and the piston rings.

Regardless of the fact, the thermophysical, rheological and tribological properties of nanofluids are widely investigated by various researchers; studies related to PAG oil based nanolubricants are scarce. In addition to thermophysical properties, the tribological characterization of nanolubricants can provide valuable insights for its practical applications. A comparative study on the thermal conductivity and viscosity of Al_2O_3 and SiO_2 based nanolubricants were conducted by Redhwan et al. (2017) and proposed correlations to predict thermal conductivity and viscosity of nanolubricants at various concentrations and temperatures. However, detailed rheological and tribological characteristics have not been reported in their studies.

This work presents experimental investigations on the thermal, rheological and tribological characteristics of Polyalkylene glycol oil suspended with oxide (TiO_2 , SiO_2 and Al_2O_3) nanoparticles. The effects of particle concentration and temperature on the thermophysical properties are investigated. The effects of temperature, volume fraction and shear rate were studied to elucidate the rheological behaviour of the nanolubricant. Studies on the rheological and tribological characteristics of PAG oil appended with different oxide nanoparticles are scarce. The wear prevention and friction reduction properties are studied with the optimum volume fraction to evaluate the tribological characteristics.

2. Experimental method

2.1. Materials and characterization

SiO_2 , Al_2O_3 and TiO_2 nanoparticles, spherical in shape supplied by Sigma Aldrich Limited, USA were used for the preparation of nanolubricant. Table 1 shows the properties of nanoparticles used for the experimental study. The commercially available, fully synthetic, oil based on Polyalkylene Glycol (PAG) was used as the base fluid. PAG lubricants have better tribological performance than mineral oils when used together with HFCs. Table 2 shows the properties of PAG lubricant. Scanning Electron Microscopy (SEM) and Transmission electron Microscopy (TEM) were employed for

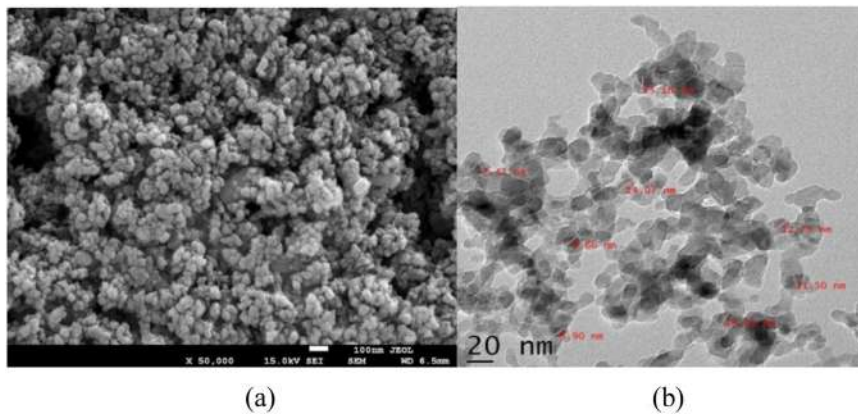


Fig. 1. (a) SEM image of Al_2O_3 nanoparticles (b) TEM image of Al_2O_3 suspension.

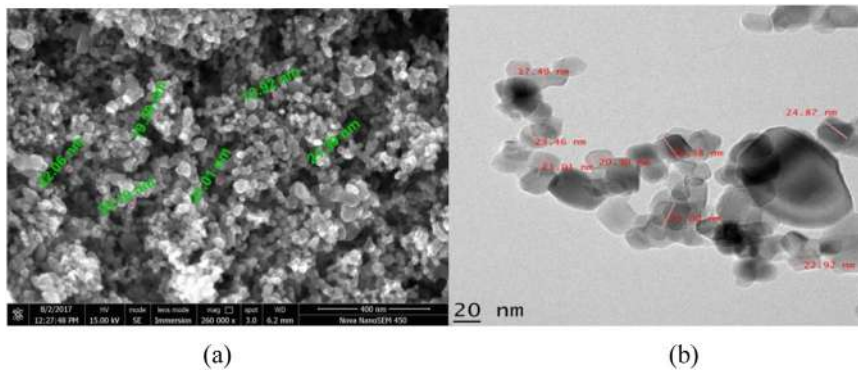


Fig. 2. (a) SEM image of TiO_2 nanoparticles (b) TEM image of TiO_2 suspension.

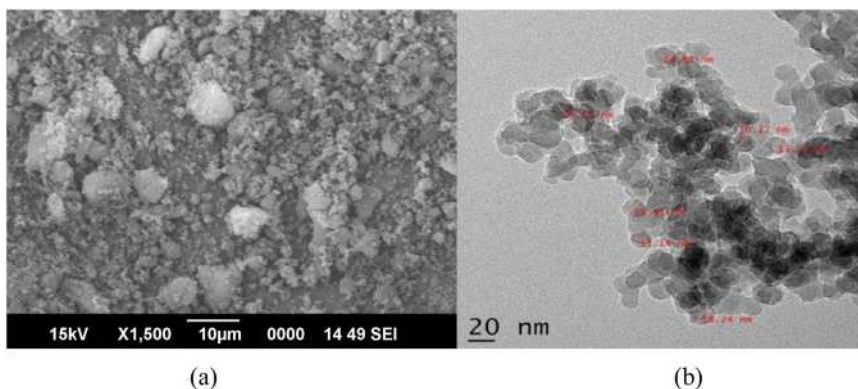


Fig. 3. (a) SEM image of SiO_2 nanoparticles (b) TEM image of SiO_2 suspension.

Table 2
Properties of PAG lubricant (Tung et al., 2006).

Density, $\text{g}\cdot\text{cm}^{-3}$ at 25 °C	0.94
Kinematic viscosity, cSt at 40 °C	32–40
Viscosity index	146
Pour point, °C	–57
Fire point, °C	242

the morphological characterization of nanoparticles and nanolubricant, respectively.

The distribution and shape of dry nanoparticles and nanoparticle suspension are shown in the SEM and TEM images (Figs. 1–3). The particles are spherical in shape. It can be seen from the SEM images that, the particles are in the form of agglomerates and these agglomerates have to be broken by magnetic and ultrasonic

agitation in order to produce stable suspension. According to TEM images the suspension is homogeneous and nanoparticles are well dispersed. The nanoparticles have almost similar characteristics such as physical appearance, shape and size in the suspension also.

2.2. Preparation of nanolubricant

Preparation of nanolubricant is the key step in the experimental studies. Nanofluids are not mere solid to fluid suspensions. In order to achieve even, stable and durable suspension with negligible aggregation of particles, special processes are necessary. Samples examined in this study were prepared by the two-step method and no surfactants were added. Nanolubricants were prepared at five different particle concentrations (0.07 to 0.6 vol %). The required mass of nanoparticles corresponding to the volume fractions were calculated (Eq. (1)) and weighed using a high precision electronic

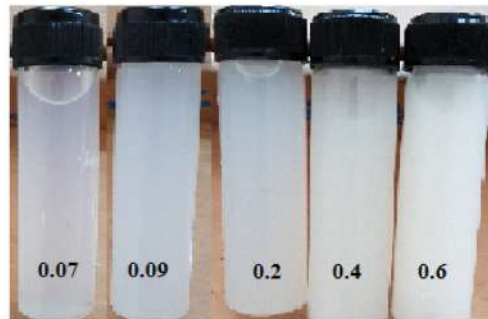
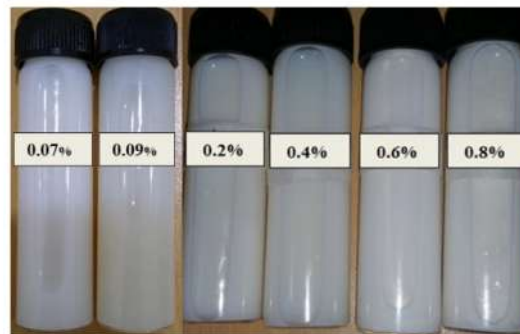
4(a) SiO₂/PAG4(b) Al₂O₃/PAG4(c) TiO₂/PAG

Fig. 4. Samples of nanolubricant at different volume fractions (a) SiO₂/PAG (b) Al₂O₃/PAG (c) TiO₂/PAG.

balance.

$$\Phi = \frac{(m/\rho)_{np}}{(m/\rho)_{np} + (m/\rho)_{PAG}} \quad (1)$$

The preliminary mixing process of samples were carried out using the magnetic stirrer for 1 h, and then agitated for 6 h using ultrasonic agitator (BRANSON-3800) at a frequency of 40 kHz to homogenize the samples. No evidence of sedimentation or coagulation was noticed after 120 h of preparation. Nanolubricant samples at different volume fractions are illustrated in Fig. 4.

2.2.1. Dispersion stability study

Spectral absorbance analysis is one of the most effective techniques to study the stability of nanofluids (Xia et al., 2016; Kumar and Sharma, 2018). The stability of nanolubricants was measured with UV-vis spectroscopy. The dispersion stability of the nanolubricants was evaluated at room temperature by measuring the absorbance. Fig. 5 shows the UV-vis spectra of the oxide nanolubricants. Fig. 5(a) shows the results after 1 day of preparation of samples. The peak absorbance obtained for all the three samples (Al₂O₃, SiO₂ and TiO₂.) was 6.0 at a wave length of 312, 313 and

311 nm, respectively. This is an indication of presence of higher population of nanoparticles in the nanolubricant to interact with the light. The absorbance tests were conducted again after 5 days and the same is shown in Fig. 5(b). The peak absorbance of Al₂O₃ and TiO₂ remained the same and that of SiO₂ nanolubricant decreased marginally to 5.13. This is an indication of the stability of nanolubricant.

2.3. Measurement of thermal conductivity

The thermal conductivity of base lubricant and nanolubricants at different volume fractions were measured with KD2 Pro thermal property analyser (Decagon devices, Inc., USA). Thermal conductivity in the range of 0.02 to 2.00 W m⁻¹ K⁻¹ with accuracy of ±0.001 could be measured by this device. The principle of operation of the device is transient hot wire and the maximum deviation is ±5.0%. The probe of this instrument consists of a needle with a heater and temperature sensor inside. Electric current is supplied through the heater and the temperature of the probe is measured over time. Since the transient hot-wire measurement lasts for only a few seconds, the problems related with convec-

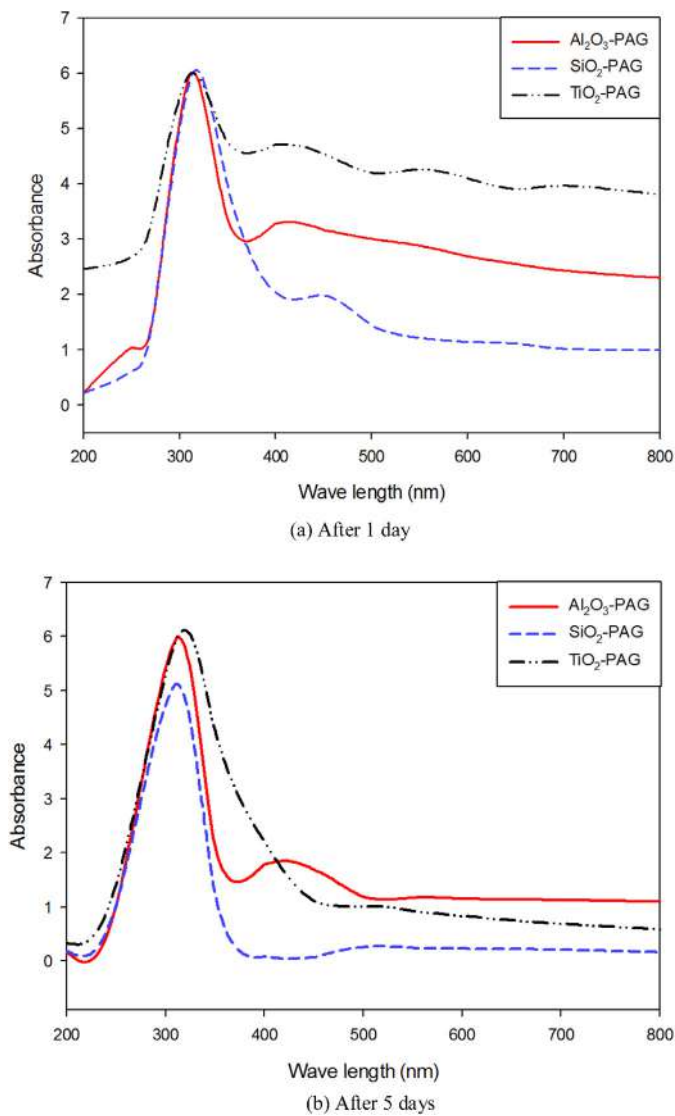


Fig. 5. Uv-vis spectrum of nanolubricants (0.4 vol %).

tion is been eliminated. The apparatus meets the standards of both ASTM D5334-14 and IEEE 442. A refrigerated and heating circulator (F-25, Julabo, Germany) was used to maintain constant temperature of the sample with an accuracy of 0.01 °C. Thermal conductivity of nanolubricants at five different particle volume concentrations (0.07 to 0.6 vol%) were measured in the temperature range of 20 °C to 50 °C. The thermal property analyser was calibrated with verification standard fluid (Glycerine, CAS 56-81-5) at a controlled temperature of 28 °C and at atmospheric pressure. The measurement of thermal conductivity was performed repeatedly. Based on deviation between thermal conductivity of standard fluid and the measured thermal conductivity during the calibration, the error in measurement is estimated and the thermal conductivity data reported in the study represents an average of ten measurements with an estimated error of $\pm 1.6\%$.

2.4. Measurement of rheological properties

The rheological behaviour of the nanolubricants has been investigated experimentally over a temperature range of 20–80 °C, shear rates range of 3.75 s⁻¹ to 225 s⁻¹ and particle concentrations range of 0.07–0.6%. A Brookfield LVDV-II +Pro, plate-and-cone rheometer having measurement range between 1.0 and 2000 cP,

accuracy $\pm 1.0\%$, and repeatability $\pm 0.2\%$ together with a constant temperature circulator (JULABO F-25, Germany) was used for the measurements. The method of measuring follows ASTM D2196-10 which is the standard test methods for rheological properties of materials by the rotational (Brookfield type) viscometer. The torque required to turn the spindle of the rheometer in a fluid is a measure viscosity of the fluid. Torque is applied through a calibrated spring to the spindle immersed in test fluid and the spring deflection measures the viscous drag of the fluid against the spindle. The amount of viscous drag is proportional to the amount of torque required to rotate the spindle. Rheocalc software was used to acquire the data. Spindle used for this study was calibrated with Brookfield viscosity standard fluid. The maximum uncertainty was found to be 1.9%. All the measurements were performed under steady state conditions within torque range of 10–100%.

2.4.1. The power law model

Ostwald De Waele model is the most generalized model for non-Newtonian fluids. Ostwald–De Waele power law model (Eq. (2)) was used to quantify behaviour of nanolubricant, i.e., whether it comes under Newtonian or non-Newtonian fluid.

$$\tau = m\gamma^n \quad (2)$$

The Power Law model is described by two parameters, consistency coefficient (m) and flow behaviour index (n). Consistency index is a product's viscosity at one reciprocal second and flow behaviour index indicates the degree with which a material exhibits non-Newtonian flow behaviour. The viscosity of the fluids which follow power law is defined by following equation:

$$\mu = m\gamma^{n-1} \quad (3)$$

Fluids which obey power law models are classified into shear-thinning and shear-thickening under increasing shear rates. If magnitude of $n < 1$, the fluid is known as shear-thinning or pseudo-plastic. This means that the apparent viscosity decreases with increase in shear rate. When $n > 1$, it is shear-thickening or dilatant, i.e. their apparent viscosity increases as shear rate increases. In order to obtain these indices, logarithmic diagram of shear stress vs shear rate has been drawn and the indices were calculated by the following equation.

$$\ln(\tau) = \ln(m) + n\ln(\gamma) \quad (4)$$

2.5. Measurement of tribological properties

The tribological properties of the base lubricant and the lubricant modified with SiO₂, TiO₂, and Al₂O₃ nanoparticles are studied. The relative wear preventive characteristics and friction coefficients of nanolubricants and base oil were determined using a four-ball tribo tester according to ASTM D 4172-94 (2014). Three 12.7 mm diameter steel balls were clamped together and filled with the lubricant to be tested. A fourth steel ball, the top ball, having 12.7 mm diameter is pressed with a force of 392 N into the cavity formed by the three clamped balls so as to achieve three point contacts. Temperature of the lubricant was set at 75 °C and the top ball is rotated at 1200 rpm for 60 min. An optical microscope with an accuracy of 0.01 μm was employed to characterize the wear scar morphology. The wear prevention characteristics of lubricants are compared by the average size of the scar diameters on the three lower balls.

3. Results and discussion

3.1. Thermal conductivity

The thermal conductivity of oxide based nanolubricants was measured covering a temperature range 20 °C–50 °C and volume

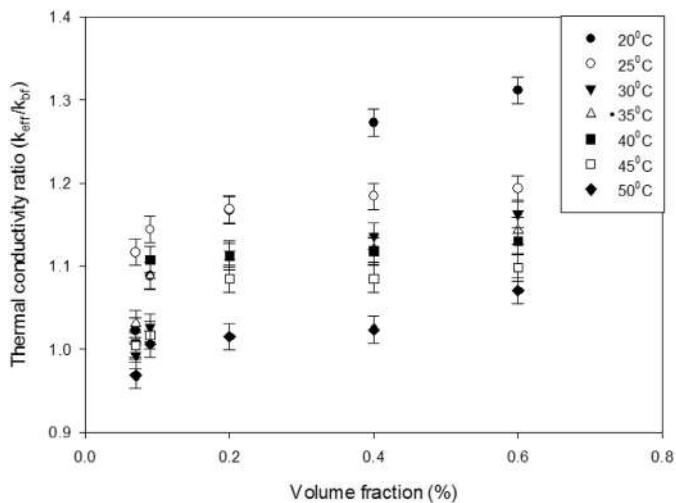


Fig. 6. Thermal conductivity ratio of SiO₂-PAG nanolubricant with volume fractions at different temperatures.

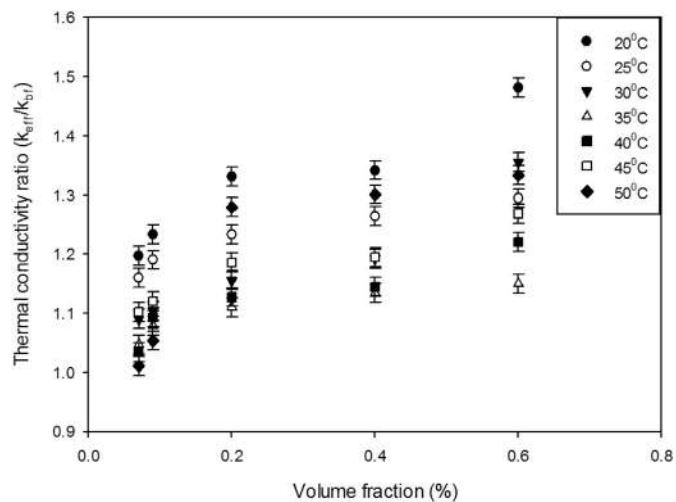


Fig. 7. Thermal conductivity ratio of Al₂O₃-PAG nanolubricant with volume fractions at different temperatures.

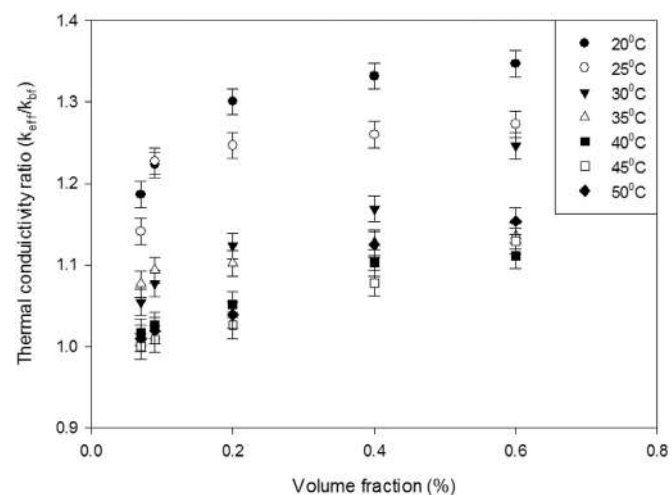


Fig. 8. Variation of thermal conductivity ratio of TiO₂-PAG nanolubricant with volume fractions at different temperatures (Sanukrishna and Jose Prakash, 2018a).

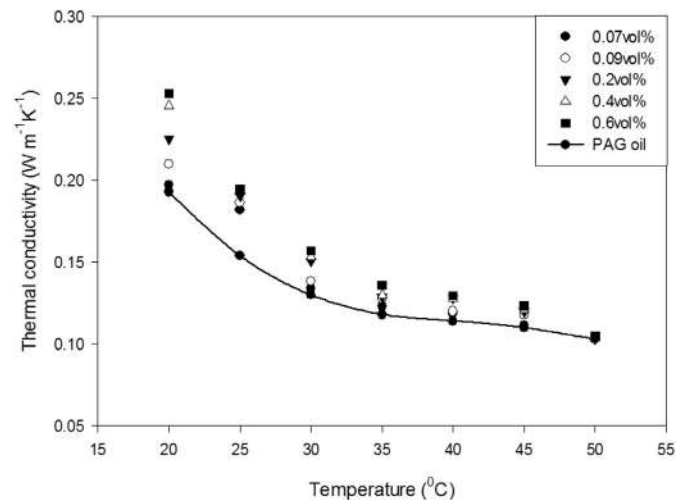


Fig. 9. Effect of temperature on thermal conductivity at different particle concentrations (SiO₂-PAG).

fraction range of 0.07–0.6 vol.%. Figs. 6–8 depict the thermal conductivity ratio (k_{eff}/k_{bf}) of SiO₂, Al₂O₃ and TiO₂ nanolubricants, respectively as a function of nanoparticle volume fraction at different temperatures.

The oxide nanolubricants exhibit higher thermal conductivity than that of pure lubricant at all particle volume fractions. Among the three oxide based nanolubricants, Al₂O₃-PAG nanolubricant has highest thermal conductivity ratio. The maximum enhancement in thermal conductivity ratio obtained was 1.48 at a volume fraction of 0.6% and temperature 20 °C. The corresponding enhancement for SiO₂ and TiO₂ nanolubricants are 1.31 and 1.35, respectively. There are many potential factors responsible for the anomalous enhancement in thermal conductivity of nanofluids: (i) The Brownian motion effect of nanoparticles in the base fluid is a vital factor for the observed enhancement. The heat is carried by phonons by propagating lattice vibrations in the crystalline solids suspended in fluids. Such phonons are propagating in random direction. Some ballistic phonon effects could possibly lead to an increase in thermal conductivity. If the ballistic phonons initiated in one particle can reach a nearby particle (Shen et al., 2009). The particles may be closer enough due to the increase of particle concentration, and thus enhance coherent phonon heat flow among particles due to Brownian motion; eventually the thermal conductivity becomes a function of nanoparticles concentration. (ii) molecular layering of the liquid: Existence of a nanolayer at the solid-liquid interface and nanoparticle aggregation may constitute major contributing mechanisms for thermal conductivity enhancement in nanofluids. The liquid molecules close to particle surfaces are known to form layered structures and behave much like a solid and which will act as a thermal bridge between fluid and solid particles. (iii) the higher thermal conductivity of suspended solid particles compared to base fluid.

Figs. 9–11 portray the temperature dependence of thermal conductivity at different particle concentrations. The thermal conductivity of pure lubricant and nanolubricants at all particle concentrations decreases with increase in temperature. The phenomenon can be attributed to the following factors: (i) at elevated temperatures, the liquid molecules move away from each other, and hence the mean free path between them increases. This reduces the probability of collision between the molecules and which eventually decreases the thermal conductivity (Sharif et al., 2017). (ii) at higher temperatures the separation distance between the particles increases and consequently the near-field radiation (i.e., Coulomb interaction) deteriorates and which in turn reduces thermal con-

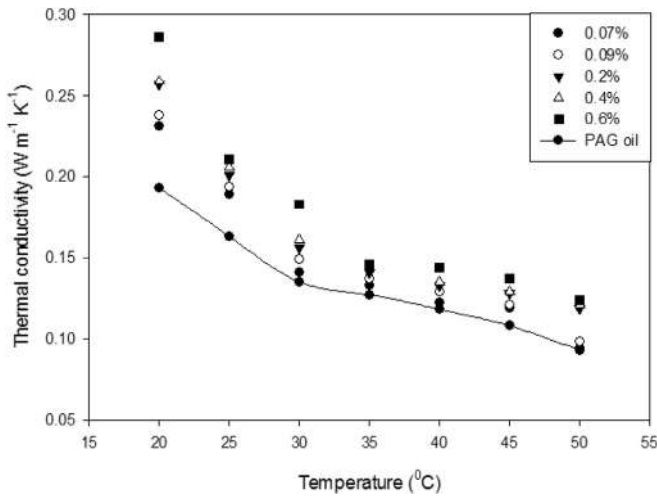


Fig. 10. Effect of temperature on thermal conductivity at different particle concentrations (Al₂O₃-PAG).

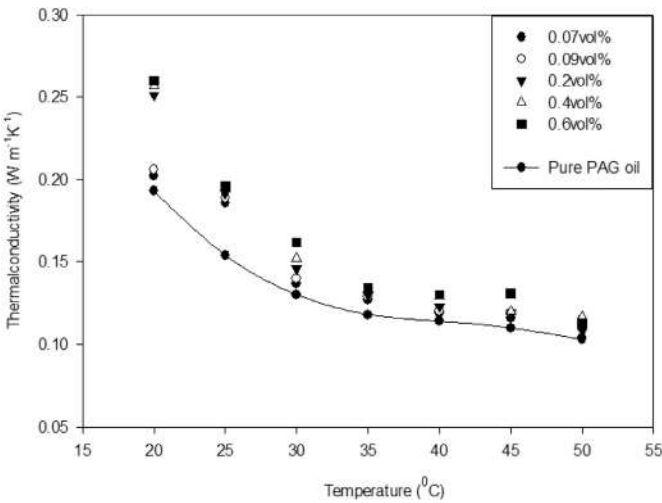


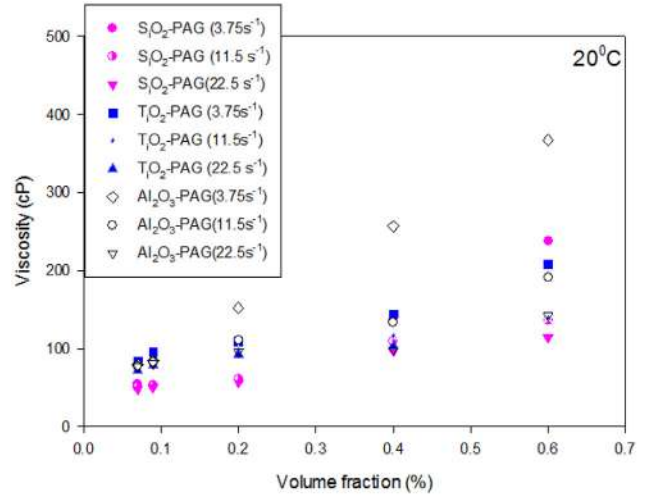
Fig. 11. Effect of temperature on thermal conductivity at different particle concentrations (TiO₂-PAG), (Sanukrishna and Jose Prakash, 2018a).

ductivity (Domingues et al., 2005). However at all volume fractions, the thermal conductivity of nanolubricants is found to be higher than that of pure lubricant. For the case of SiO₂ nanolubricant (Fig. 9), at higher temperature, the increase in thermal conductivity compared to pure lubricant was trivial. This is due to the lower thermal conductivity of SiO₂ particles compared to the other oxide nanoparticles.

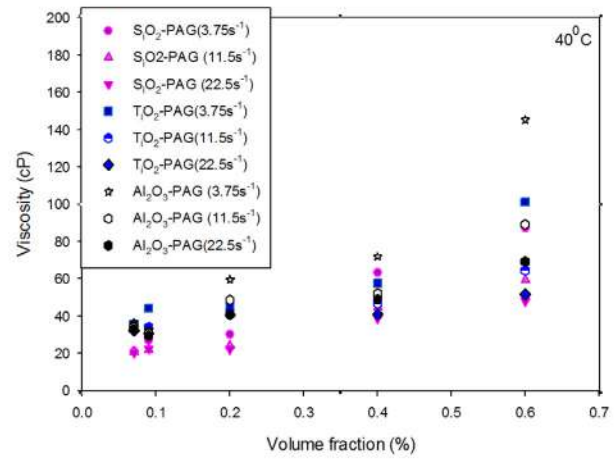
The thermal conductivities of SiO₂ and Al₂O₃ nanolubricant obtained from the present experimental study were found to be higher than that reported by Redhwan et al. (2017). This deviation is probably due to the difference in sonication period and particle size.

3.2. Rheological characteristics of nanolubricants

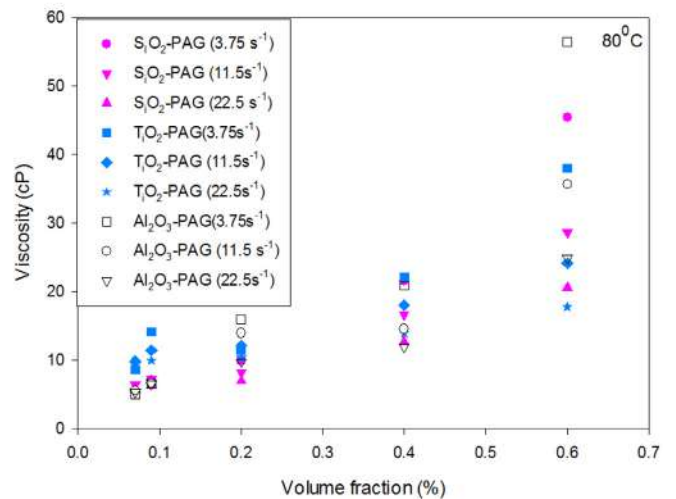
Viscosity of pure PAG oil and three oxide based nanolubricants at different volume fractions (0.07 to 0.6 vol %), shear rate (3.75 s⁻¹ to 225 s⁻¹) and temperatures (20 °C to 80 °C) were measured. Ostwald–de Waele power law model was considered to evaluate the rheological behaviour.



(a)



(b)



(c)

Fig. 12. Variation of viscosity of nanolubricants with volume fractions at different shear rates at (a) Temperature = 20 °C, (b) Temperature = 40 °C, (c) Temperature = 80 °C.

3.2.1. Effect of particle concentration on viscosity of nanolubricants

Figs. 12 and 13 show the variation of viscosity and viscosity ratio (μ_{eff}/μ_{bf}) with volume fraction at different temperatures and shear rates.

It is obvious that shear rate and volume fraction have significant role in the alteration of viscosity of nanolubricant. As the volume fraction increases, the viscosity of nanolubricants increase and there is substantial increase in viscosity at lower shear rates and temperatures. The highest viscosity obtained was 367cP for Al₂O₃-PAG nanolubricant and it happens at volume fraction of 0.6% and a shear rate of 3.75 s⁻¹. Viscosity is a property occurring due to the internal frictional force that develops between different layers of fluids as they move relative to each other. The suspended nanoparticles in the PAG oil would raise its viscosity as a result of the collisions between nanoparticles and the base fluid. By intensifying the amount of solid nanoparticles in a fixed volume of a liquid, greater nano-racemes arise due to Van der Waals forces. In other words, as the concentration of nanoparticle increases, the particles make

agglomerations within the suspension. This consequently results in the increase of internal shear stress in nanolubricant and hence an increase in viscosity.

Fig. 13 shows the variation of viscosity ratio (μ_{eff}/μ_{bf}) with volume fraction and temperature at three different shear rates. The viscosity ratio increases with increase in temperature and this is because of the fact that, the rate of decrease of viscosity of pure lubricant is higher than that of nanolubricants, in other words, the nanolubricant sustains adequate viscosity than pure lubricant at elevated temperatures. An abrupt increase in relative viscosity at higher volume fractions and at lower shear rates also been manifested. The maximum viscosity ratio is found as 10.34 for SiO₂-PAG nanolubricant, which is obtained at a lower shear rate of 3.75 s⁻¹ at volume fraction of 0.6% and at 80 °C. The viscosity ratios for TiO₂-PAG and Al₂O₃-PAG nanolubricants are 5.64 and 9.71, respectively. The reason for this anomalous increase in relative viscosity may be related to the fact that at higher volume fraction the nanoparticle clustering is more and the applied lower shear rate

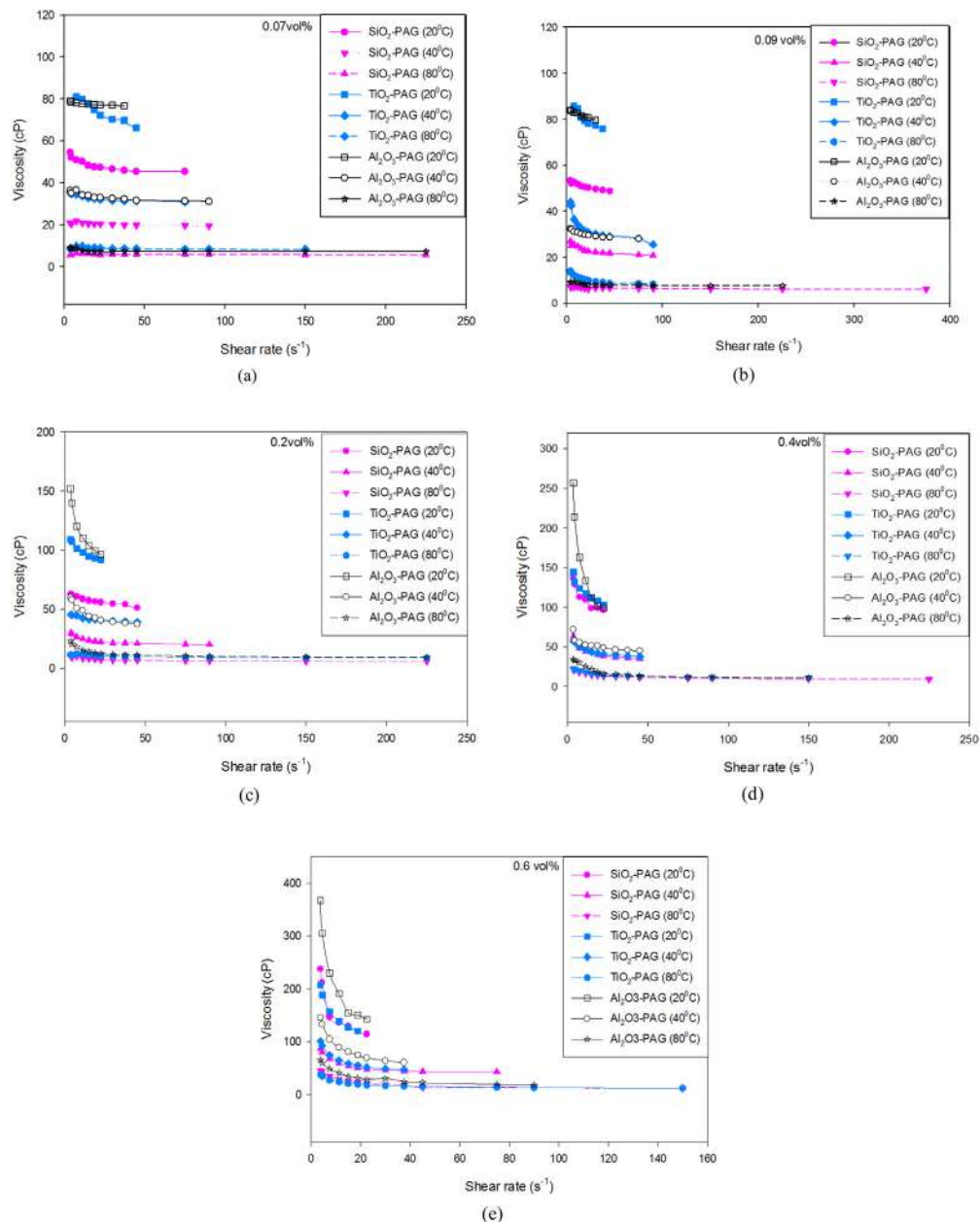


Fig. 16. Variation of viscosity with shear rate of SiO₂-PAG, TiO₂-PAG and Al₂O₃-PAG nanolubricants at (a) 0.07 vol% (b) 0.09 vol% (c) 0.2 vol% (d) 0.4 vol% (e) 0.6 vol%.

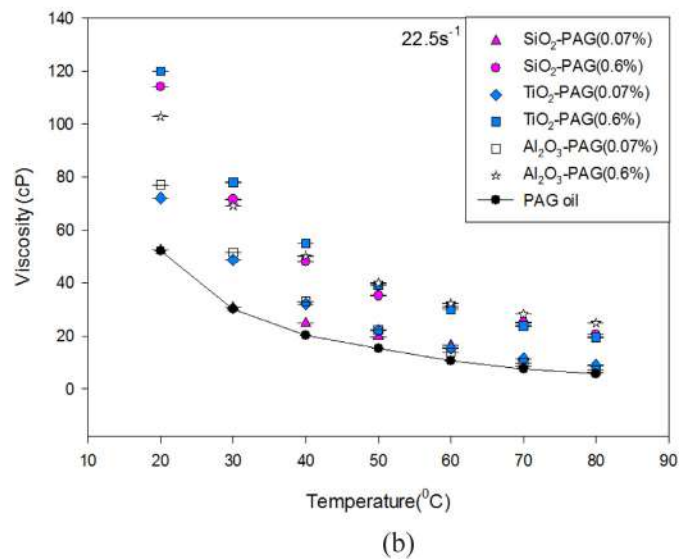
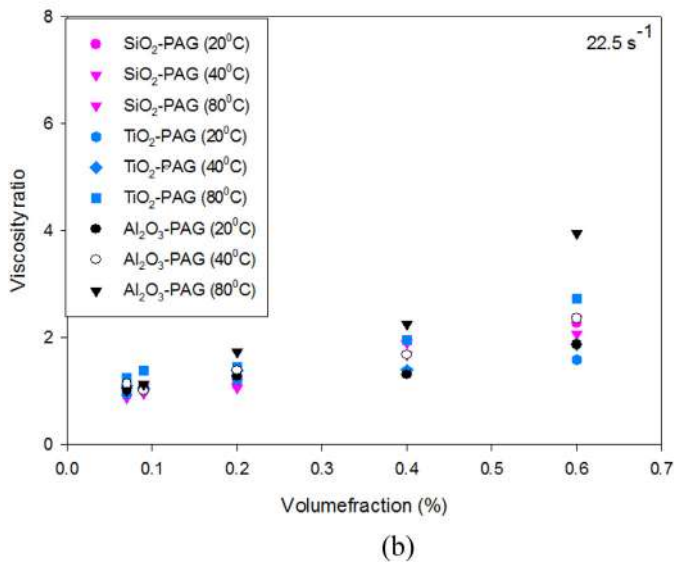
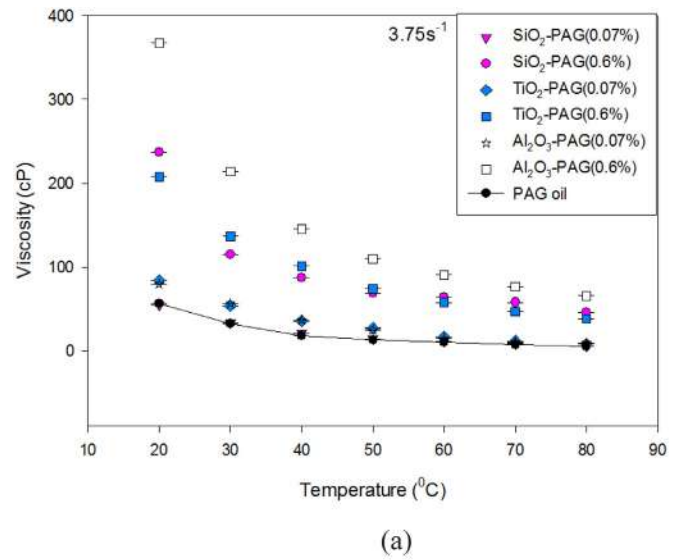
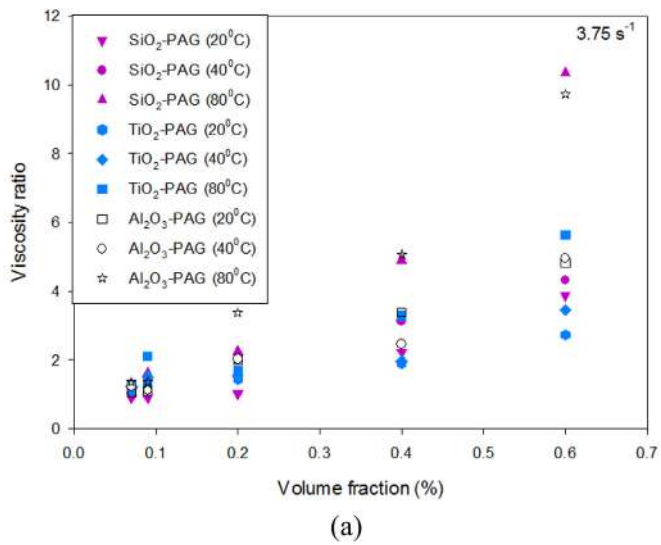


Fig. 13. variation of viscosity ratio with volume fraction at different temperatures at (a) shear rate = 3.75 s⁻¹ (b) shear rate = 22.5 s⁻¹.

Fig. 14. Variation of viscosity with temperature at (a) at shear rate 3.75 s⁻¹ (b) at shear rate 22.5 s⁻¹.

(3.75 s⁻¹) is not sufficient enough to break the nano-clusters. As the shear rate increases, the relative viscosity is found to be decreasing. The maximum viscosity ratio observed at a higher shear rate of 22.5 s⁻¹ is 3.9 for Al₂O₃-PAG a particle concentration 0.6%.

Furthermore, up to 0.4% volume fraction, all the three oxide based nanolubricants sustains adequate relative viscosity at elevated temperatures and moderate shear rates. When the particle dosing level increased to 0.6% the viscosity increase more than twice. That is, the nanolubricants are more suitable to use at a volume fraction up to 0.4% and at moderate shear rates. These conditions are more suited with refrigerant compressors. These finding are in line with the studies reported by Redhwan et al. (2017), Sanukrishna and Prakash, (2018) and Sharif et al., (2016).

3.2.2. Effect of temperature on viscosity

Fig. 14 provides better understanding of viscosity variation with temperature. The dynamic viscosity versus temperature at lower and higher volume fractions and shear rates is plotted for different types of nanolubricants. The results reveal that irrespective of the shear rate and volume fraction, the viscosities nanolubricants diminish with increase in temperature. The similar trend

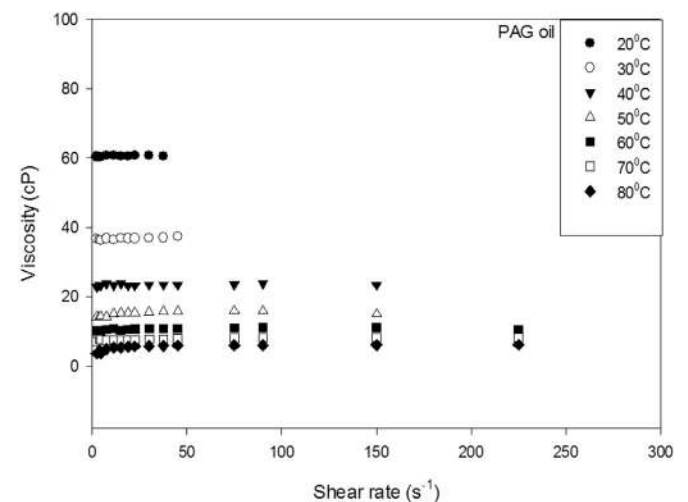


Fig. 15. Viscosity variations of pure-PAG oil with shear rates at different temperatures.

was displayed by the pure lubricant as well. This possible reason is, at lower temperatures, the nano racemes hinder the motion of lubricating oil layers on each other. Increasing the temperature helps the particles to overcome Van der Waals attractive forces and which may disintegrate the clusters of nanoparticles suspended in base fluid and hence the intermolecular interactions between the molecules become weak and this phenomenon leads to decrease in viscosity.

3.2.3. Rheological behaviour of base lubricant and nanolubricant

The rheological behaviour of pure lubricant and nanolubricants can be unveiled by exploring the relation between viscosity and shear rate at different temperatures. The rheological studies were performed over the range of shear rate from 3.75 to 225 s⁻¹ covering wide range of temperature. Fig. 15 shows the variation of viscosity with shear rate for pure lubricant. As expected, pure lubricant exhibits Newtonian behaviour within the shear rate range considered.

The viscosity of the nanolubricant samples were measured over the same range of shear rate. Fig. 16 shows the effects of shear rate and temperature on apparent viscosity of nanolubricants. The apparent viscosity decreases with shear rate increment regardless of nanoparticle type, temperature and solid volume fraction. The results illustrate that, for oxide nanolubricants at low concentrations (0.07% and 0.09%) change in viscosity with increase in shear rate is marginal. However, at higher concentrations, a considerable decrease in the viscosity of the samples with shear rate is noticed. That is viscosity of high concentration samples is more reliant on shear rate, consequently displays the non-Newtonian behaviour. The alterations in the structure and arrangement of intermingling particles can be connected to shear thinning behaviour of well-dispersed suspensions. Shearing may cause the particles to orient in the direction of flow and its gradient. This can disrupt the agglomerates in the suspension and hence reduce the amount of fluid immobilized by the nanoparticles. The interaction forces may then decrease and which in turn lowers the flow resistance and the apparent viscosity.

The viscosity decreases exponentially at lower shear rates. i.e. the nanolubricant exhibit non-Newtonian behaviour with significant shear thinning characteristics. The shear viscosity of the nanolubricant decreases considerably up to a shear rate of about 50 s⁻¹ and over this shear rate, the apparent shear viscosity of the nanolubricants tends to a Newtonian plateau.

Figs. 17 and 18 show the logarithmic diagram of shear stress vs shear rate of pure lubricant and nanolubricant, respectively. The volume fraction considered for the power law study of nanolubricants is 0.4%. The curves are fitted with R-squared value higher than 0.99. Here, *n* and *m* are the power law and consistency indices, respectively. The pure PAG oil behaves as Newtonian fluid with power law index *n* ≈ 1 at all temperatures whereas oxide nanolubricants behave as non-Newtonian fluid. All the three oxide based nanolubricants follow the power law model expressed in Eq. (2) with a power law index less than unity (*n* < 1).

Fig. 19 depicts the variation in power law index with particle dosing level and temperature. According to power law index, shear thinning is confirmed for nanolubricant at all temperatures and particle concentrations. However, significant shear thinning behaviour is manifested at volume fractions beyond 0.4%. Addition of oxide nanoparticles to the host lubricant alters the structure of the base oil by initiating a disturbance of molecular links. The nanoparticles act as interfaces to connect base oil layers to each other. Conversely, the difference between the Brownian nature of nanoparticles and the base fluid leads to breaking the molecular links and creating new links; eventually the nanolubricant exhibit shear thinning behaviour.

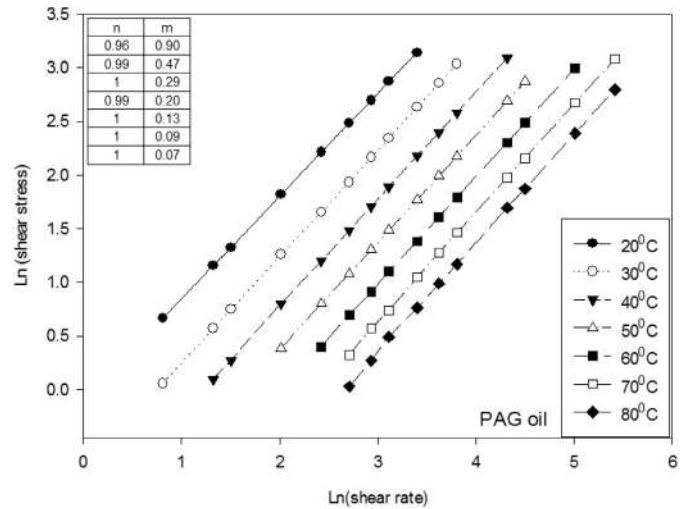


Fig. 17. Logarithmic diagram of shear stress-shear rate of pure PAG oil at different temperatures.

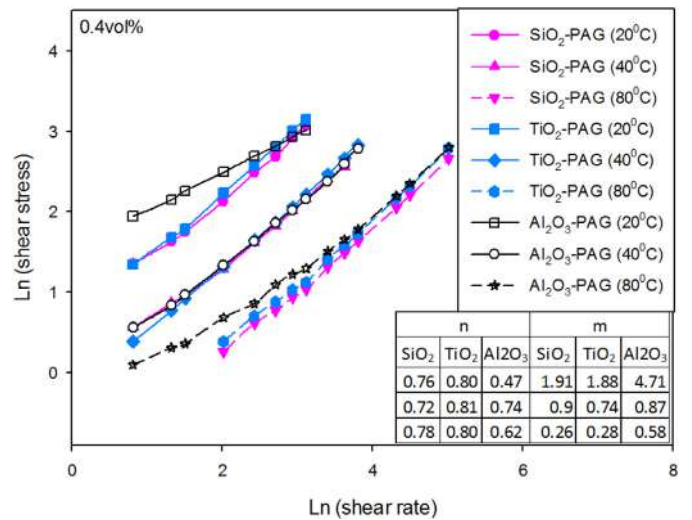


Fig. 18. Logarithmic diagram of shear stress vs shear rate of SiO₂-PAG, TiO₂-PAG and Al₂O₃-PAG nanolubricants at different temperatures.

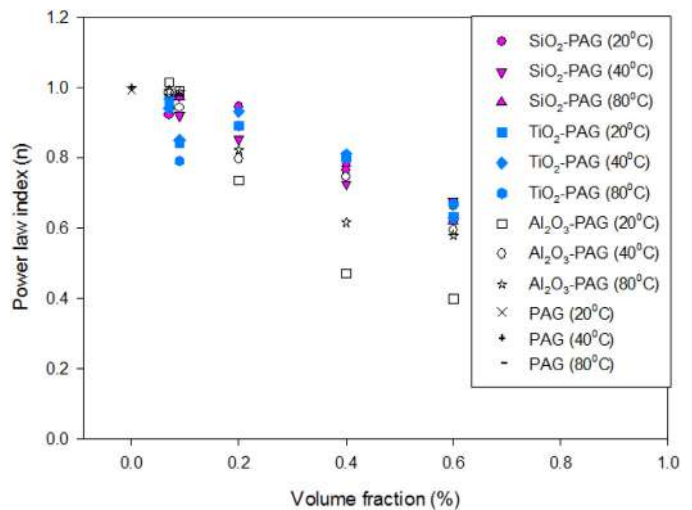


Fig. 19. Power law indexes of pure PAG and SiO₂-PAG, TiO₂-PAG and Al₂O₃-PAG nanolubricants with volume fractions at different temperatures.

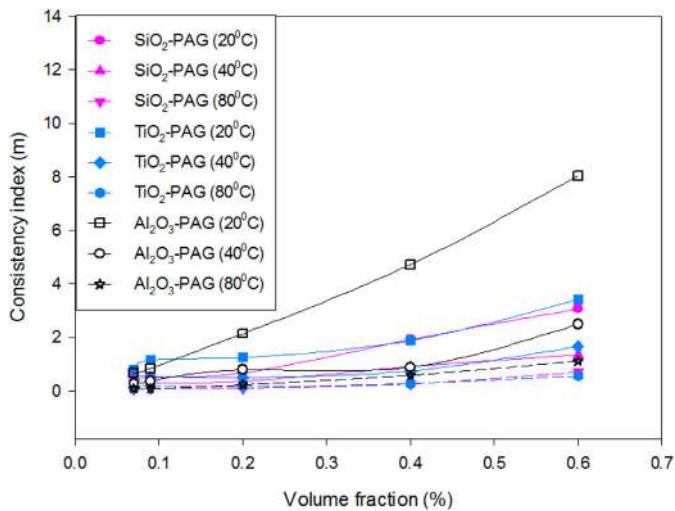


Fig. 20. Consistency index of SiO₂-PAG, TiO₂-PAG and Al₂O₃-PAG nanolubricants with volume fractions at different temperatures.

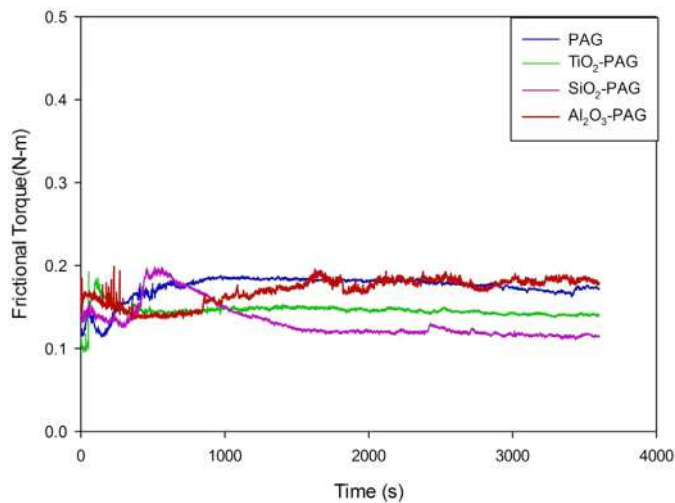


Fig. 21. Frictional torque vs time at a volume fraction of 0.4%.

The consistency index of nanolubricants is influenced by both the temperature and the particle concentration. With respect to Fig. 20, at low temperatures, intensification of particle concentration results in increase in consistency index, which means substantial variation in viscosity. But at elevated temperatures, significant variation in consistency index has not been exhibited. Moreover marginal variation in consistency index was observed at low volume fractions, except at 20 °C, which is an indication of consistency of apparent viscosity.

3.3. Tribological characterization

3.3.1. Frictional torque and friction coefficient

The time dependence of friction torque and friction coefficient for pure lubricant and nanolubricants are shown in Figs. 21 and 22. The power consumption by a compressor is influenced by the coefficient of friction. The friction coefficient was evaluated according to the friction torque measurements during the tribological tests. From the figures it is clear that after an initial increase, both frictional torque and friction coefficient, reaches almost steady state condition. The nanolubricants especially SiO₂ and TiO₂ nanolubricants exhibit reduced frictional torque than that of pure PAG oil.

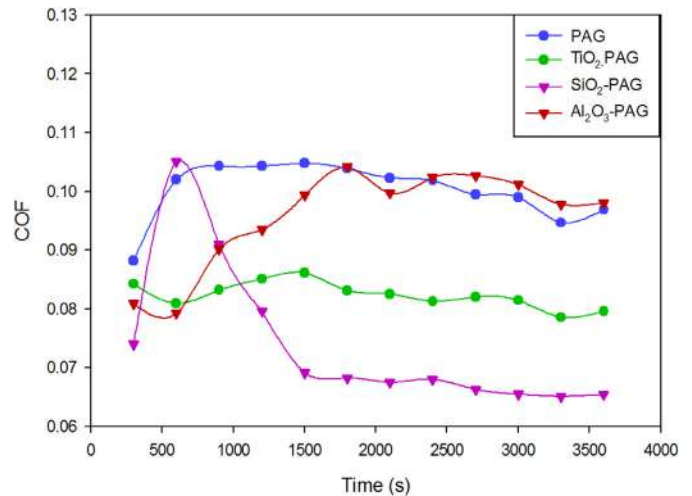


Fig. 22. Friction coefficient vs time at a volume fraction of 0.4%.

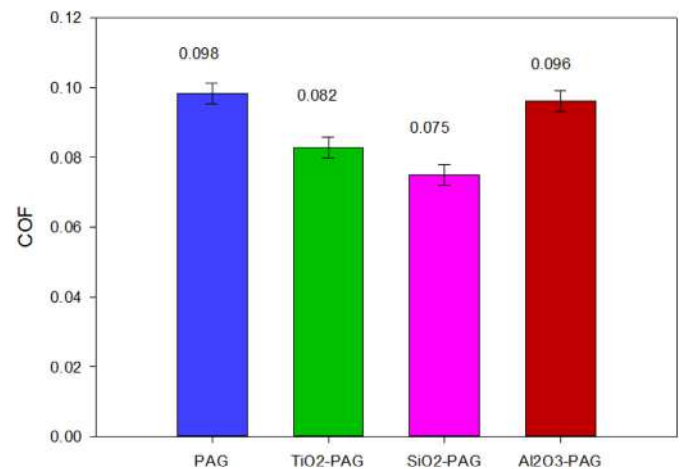


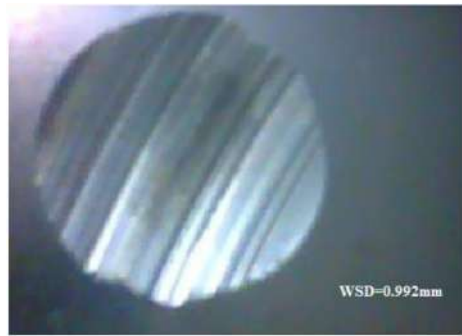
Fig. 23. Comparison of average friction coefficient (at 0.4 vol %).

The lubricant forms a thin film between the contact surfaces which reduces the frictional torque as well as the frictional coefficient. It can be seen from Fig. 22 that, for the case of SiO₂-PAG nanolubricant, the friction coefficient slightly increases initially (up to 600 s) and as the time elapses, it stabilises. Conversely, for TiO₂ and Al₂O₃ nanolubricants, marginal drop in frictional coefficient has been observed initially. This is due to the polishing effect of nanoparticulates which comes in interaction between the surface asperities (Lee et al., 2009). Among the samples tested, SiO₂-PAG nano lubricant yields better friction reduction characteristics.

Fig. 23 shows the comparison of the average friction coefficient of pure lubricant and nano lubricant at 0.4vol%. The average friction coefficient of the base oil is higher than that of oxide nanolubricants. The percentage reduction in coefficient of friction for SiO₂, TiO₂, and Al₂O₃ nano lubricants compared to pure PAG oil was 23.8%, 15.8%, and 2.3%, respectively.

3.3.2. Wear scar diameter

The wear scar examination reveals the anti-wear characteristics of lubricant samples. Larger wear scar diameter is an indication of higher wear rate. Fig. 24 shows the wear scar images of ball surface for the case of pure lubricant and three oxide nanolubricants. A comparison between average wear scar diameters of three lower balls in each run using pure lubricant and nano lubricants is shown in Fig. 25. The average pure wear scar diameters with



(a) Pure PAG

(b) TiO₂-PAG(c) SiO₂-PAG(d) Al₂O₃-PAG

Fig. 24. Wear scar images (a) Pure PAG (b) TiO₂-PAG (c) SiO₂-PAG (d) Al₂O₃-PAG.

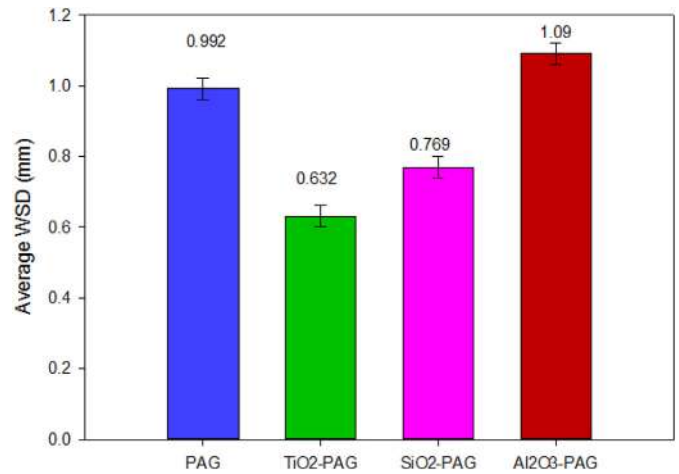


Fig. 25. Comparison of average wear scar diameter (at 0.4 vol %).

TiO₂-PAG and SiO₂-PAG nanolubricants are lower than that with base lubricant. The reduction in wear scar diameter with TiO₂-PAG and SiO₂-PAG nanolubricants are by 36% and 22.5%, respectively. It is evident from the rheological studies that, the nanolubricants show higher viscosity that of pure lubricant at elevated temperatures and shear rates. Al₂O₃ nanolubricant displays a detrimental effect on anti-wear property compared to others at a volume fraction of 0.4%. This is probably due the inherent hardness of Al₂O₃ particles than the metal substrate and abrasive nature of it, especially in agglomerated form.

The reduction in wear scar diameter can be due to (i) the direct effect of the nanoparticles that includes ball bearing effect and protective/tribo-film formation (ii) the secondary effects which contribute to lubrication enhancement are surface enhancement by repairing effect and polishing/smoothing effect (Gulzar et al., 2016). Since the grain size of the nanoparticles is smaller than the surface irregularities, the valleys between the frictional surface asperities will be stuffed with the nano-additives. This film formation is triggered by the reaction between the material and the additives under the provided environment and which in turn augments the tribological behaviour of nano lubricants compared to base oil. Gulzar et al. (2016), Rajendhran et al.(2018) and Verma et al. (2008) reported the formation a durable tribo-film with Mo, S, and P elements, reduces the chances of severe wear, friction, and seizure for nanoMoS₂-based lubricant. The spherical and quasi-spherical nanoparticles believe to function like tiny ball bearings which roll into the contact area. Nanoparticles with such shapes are believed to change the sliding friction to a mix of sliding and rolling friction. It may be assumed that the presence of nanoparticles decreases the fraction of straight asperity contact and hence improves the tribological properties of contact pairs. The mending effect or self-repairing effect is characterized by nanoparticles deposition on the interacting surfaces and compensation for the loss of mass. The polishing effect, also termed as smoothing effect, is believed to be reported when the roughness of the lubricating surface is reduced by nanoparticle-assisted abrasion. Reduced surface roughness is the responsible factor for the smoothing mechanism which leads to improved friction reduction performance.

The present experimental study indicated that addition of oxide nano particles to the lubricating oil is an effective way to improve the friction reduction and wear preventive characteristics of lubricants. However, the role of particle loading is an important parameter on the tribological properties, which need to be investigated extensively.

4. Conclusions

The thermophysical, rheological and tribological properties of PAG nanolubricant appended with TiO₂, SiO₂ and Al₂O₃ nanoparticles were experimentally investigated. The effect of particle concentration and temperature on thermal conductivity and viscosity of nanolubricant were elucidated. The rheological properties are investigated at various shear rates, particle concentrations, temperatures. Tribological characterisation is conducted at an optimum concentration. The following key conclusions have been drawn from the present experimental investigation.

- (i) The thermal conductivity of the nanolubricants increase with increase in volume concentration and decreases with the intensification of temperature. The maximum thermal conductivity ratio was obtained for Al₂O₃-PAG nanolubricant.
- (ii) The viscosity of oxide based nanolubricants increases with increase in volume fraction and decreases with increase in temperature. Moreover, the viscosity augmentation was found to be higher compared to that of thermal conductivity.
- (iii) The maximum enhancement in viscosity ratio is observed for SiO₂-PAG nanolubricant.
- (iv) Unlike pure lubricant, shear rate plays a vital role on the behaviour of the oxide based nanolubricants.
- (v) Non-Newtonian shear thinning of nanolubricants was evidenced. However at higher shear rates, shear thinning is insignificant and nanolubricants behaves almost like a Newtonian fluid.
- (vi) At elevated temperatures and shear rates, SiO₂, TiO₂ and Al₂O₃ nanolubricants sustain adequate viscosity than pure lubricant which is a desirable operating feature for refrigerant compressors.
- (vii) Oxide based nanolubricants exhibits better friction reduction capability than pure lubricant.
- (viii) Lubricants with SiO₂ and TiO₂ nanoparticles show excellent anti-wear capabilities. Wear scar diameter reduces by 36% and 22.5%, respectively as compared to PAG oil.
- (ix) The addition of SiO₂, TiO₂ and Al₂O₃ nanoparticles decreases the friction coefficient by 23.8%, 15.8% and 2.3%, respectively.
- (x) Al₂O₃ nanolubricant shows better friction reduction capability than that of pure lubricant; However, it displays a detrimental effect on anti-wear property.
- (xi) The anti-wear and friction reduction capabilities of SiO₂ and TiO₂ nanoparticles as an additive to PAG lubricant is promising and will reflect the longevity of friction pairs in refrigerant compressors.
- (xii) Thermal, rheological and tribological study of oxide based nanolubricants revealed that, the potential of SiO₂ nanolubricant as an alternative lubricant for refrigerant compressors is excellent.
- (xiii) The application of the novel type of lubricant may lead to the development of energy efficient of HVAC systems.

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