



# Rheology of mineral oil-SiO<sub>2</sub> nanofluids at high pressure and high temperatures



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## ABSTRACT

Nanofluids, engineered colloidal suspensions of nano-sized particles (less than 100 nm) dispersed in a basefluid, have shown potential for use as industrial cooling fluids due to their enhanced heat transfer capabilities. Many industrial applications often involve heat transfer fluids at pressures and temperatures above average atmospheric condition. Understanding the rheological characteristics of nanofluids is necessary for implementing them in these extreme conditions. Even though the effect of temperature on the viscosity of nanofluids at atmospheric pressure has been well studied, viscosity measurements of nanofluids at elevated pressures and temperatures have not yet been investigated. This work investigates the rheological characteristics of mineral oil based nanofluids at high pressure and high temperature (HPHT). The nanofluids used in this work were prepared by mechanically dispersing commercially available SiO<sub>2</sub> nanoparticles (~20 nm) in a highly refined paraffinic mineral oil (Therm Z-32, QALCO QATAR), which has wide applications in industrial heat exchangers. Mineral oil and nanofluids, with two volume concentrations of 1% and 2%, are studied in this work. The rheological characteristics of the basefluid and nanofluids are measured using an HPHT viscometer. During experimentation, viscosity values of the nanofluids are measured at pressures of 100 kPa and 42 MPa, with temperatures ranging from 25 °C to 140 °C, and at varying shear rates. The results show that the viscosity values of both nanofluids, as well as the basefluid, increased as the pressure increased. In addition, nanofluids exhibit non-Newtonian characteristics at elevated temperatures and pressures.

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## 1. Introduction

The constant increase in demand for and price of energy has caused researchers to look for more energy-saving methods in their applications. Among the many active and passive methods undertaken, improving the heat transfer capabilities of cooling fluids by suspending solid particles seemed promising as they could enhance fluids' heat transfer performance that was inherently limited by their poor thermo-physical properties. However, suspending micron size particles with higher thermal conductivity has introduced undesirable effects such as erosion of walls, increase in pumping power, and sedimentation. Recent innovations in nanotechnology has brought out a new breed of cooling liquids known as nanofluids, which are engineered colloidal suspensions containing nano-sized particles (less than 100 nm) [1]. The smaller size

of the particles used in nanofluids potentially minimizes issues related to sedimentation and erosion.

The interest in nanofluids application mainly stems from its enhanced thermal conductivity values that were reported earlier [2–6]. However, in addition to conductivity, the primary thermo-physical property that needs to be considered while implementing nanofluids in practice are their rheological characteristics. Viscosity of the cooling fluids is a crucial parameter, as it affects pressure drop in the system which impedes with its thermal performance. Initial studies on rheological characteristics of nanofluids, show a higher increase in the viscosity values of nanofluids than the values predicted by conventional suspensions theories [2,3]. Prasher et al. [7] showed that the overall thermal performance of a nanofluid system becomes worse than that of the basefluid when its viscosity is increased by more than a factor of four relative to the increase in the thermal conductivity. In another study, Garg et al. [8] stated that nanofluids perform poorly as a coolant when compared to basefluids due to the larger increase in viscosity than that of thermal conductivity. However, they showed that the higher thermal conductivity of the nanofluids could be

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### Nomenclature

$C$	enhancement coefficient
$P$	pressure (Pa)
$T$	temperature ( $^{\circ}\text{C}$ )
$\alpha$	pressure viscosity coefficient ( $\text{M Pa}^{-1}$ )
$\phi$	volume fraction
$\gamma$	shear rate ( $\text{s}^{-1}$ )
$\eta$	viscosity (cP)

### Subscript

$a$	at atmospheric conditions
$k$	for thermal conductivity
$\eta$	for viscosity

proved beneficial if the characteristic pipe diameter in the application was increased [8].

The effects of parameters such as particle type, concentration, size, shape, working temperature, and basefluids, have been investigated on the rheological characteristics of nanofluids in the recent past. There is unanimous agreement that viscosity of nanofluids increase with an increase in particle concentration. Pak and Cho [3] reported an anomalous 300-fold increase in viscosity of the alumina–water nanofluid compared to that of the basefluid. In contrast to the above observation, some researchers [4,5] measured an increase in viscosity of order only 85% for similar concentrations and similar nanoparticle size. Furthermore, the increase in viscosity of nanofluids was reported to be inversely proportional to the mean particle diameter [9–11]. However, Nguyen et al. [12] has observed that the viscosity values of 36 nm particle suspensions were 5% lower than that of nanofluids with 47 nm particles. The shape of the suspended particles was also observed to be an influencing parameter for an increase in effective viscosity. For example, Timofeeva et al. [13] found that nanofluids with platelets (elongated particles) or cylinder shaped particles resulted in a higher viscosity values for the same volume fraction.

It is generally agreed that, similar to most fluids, the viscosity of nanofluids decreases with an increase in temperature [14,15]. Namburu et al. [11] reported an exponential decrease in viscosity with an increase in temperature for  $\text{SiO}_2$ –ethylene glycol/water mixture nanofluids in a working temperature range from  $-35^{\circ}\text{C}$  to  $50^{\circ}\text{C}$ . They observed that these nanofluids exhibit non-Newtonian behavior at lower temperatures, whereas at higher temperatures Newtonian nature is observed. In a separate investigation, Namburu et al. [16] demonstrated that  $\text{CuO}$  nanoparticles in an ethylene glycol/water mixture exhibits Newtonian behavior even at low temperatures. Contrary to the above observations, Chen et al. [17] reported a slight decrease in the viscosity of carbon nanotubes–water nanofluid with a volume concentration of less than 0.4%, following an increase viscosity after this concentration. The initial decrease in viscosity was attributed to the lubrication effects of nanoparticles.

Variations in the rheological behavior of nanofluids have also been reported to vary, depending on the type of the basefluid and how it is formulated [15]. Timofeeva et al. [18] suggested that for nanofluids' viscosity depends more on the type of basefluid than it does on the nanoparticle's characteristics. Most water-based nanofluids at lower concentrations exhibited Newtonian characteristics, whereas at higher concentrations and for non-aqueous basefluids, non-Newtonian behaviors are observed. Chen et al. [19] observed that ethylene glycol-based  $\text{TiO}_2$  nanofluids exhibited Newtonian behavior where the relative increase of the shear viscosity was independent of temperature. They claimed that aggregation of particles was the cause for the observed increase in

viscosity [19]. Contrary to the above observation, shear thinning behavior was reported by He et al. [20] for  $\text{TiO}_2$ –water nanofluid. They reported a rapid decrease in the shear viscosity with an increasing shear rate and beyond a shear rate of  $100 \text{ s}^{-1}$  viscosity was observed to be constant. Phuoc and Massoudi [21] investigated the rheological behavior of  $\text{Fe}_2\text{O}_3$ –water nanofluids in a Polyvinylpyrrolidone dispersant. Nanofluids exhibited Newtonian behavior at lower concentrations, whereas, beyond a particular loading, shear-thinning behavior was observed.

Industrial applications are often subjected to higher operating temperatures and pressures for fluids than those usually examined in nanofluids studies. A high pressure water jet, for example, is used for surface preparation, concrete hydro-demolition, oil drilling and stabilization, and manufacturing procedures [22]. In manufacturing, for example, machining with high pressure cooling (HPC) (with pressures up to 200 MPa) is shown to reduce the temperature of the cutting zone, providing higher productivity and an increase of the machining performance of the tool [22,23]. The application of nanofluids in this area is promising due to the enhanced thermal and rheological properties that it exhibits, and other possible effects that the addition of nanoparticles may have on machining performance. Drilling fluids are an integral part of several industries such as oil, gas, and geothermal industries. Drilling fluid is pumped to the bottom of the bore hole, through a drill string, to cool and lubricate the drill bit, to remove the rock debris from the drilling area, and to convey debris up to the surface. The operating pressure and temperature of the drilling fluid varies depending on the depth of the well and geothermal condition of the field, where pressure and temperature can reach up to 272 MPa and  $300^{\circ}\text{C}$ , respectively [24]. For example, in the Persian Gulf region, commonly encountered pressure and temperature rates are reported to be 48 MPa ( $\sim 7000$  psi) and  $110^{\circ}\text{C}$ , respectively [25,26]. The application of nanofluids provides stability and the potential to improve the rheological, mechanical, and thermal properties of drilling fluids [27]. Sedaghatzadeh et al. [28] experimentally investigated the impact of the addition of carbon nanotubes (CNT) on the thermal and rheological properties of a drilling fluid. Their results showed that addition of CNT enhanced the thermal conductivity of the drilling fluid up to 31%, with a fluid temperature of  $53^{\circ}\text{C}$ . They reported viscosity enhancement as a function of well depth (maximum of 12.2% at the bottom of a well of 2365 m deep), however they, failed to provide specifics of the effect of pressure on viscosity [28].

Even though the effect of temperature and particle types have been studied in some detail in the past, the effect of pressure on rheological characteristics of nanofluids have not yet been investigated. Understanding their rheological behavior under these extreme operating circumstances is the first step to facilitate application of nanofluids in HPHT industrial applications. In this work, an industrial paraffinic Mineral Oil (MO), commonly used as a coolant liquid in heat exchanger applications in the oil and gas industry, is used as the basefluid. Rheological behavior of  $\text{MO-SiO}_2$  nanofluid under elevated temperatures and pressures at different shear rates are investigated. Viscosity measurements are conducted for the basefluid and nanofluid with temperatures ranging from  $25^{\circ}\text{C}$  to  $140^{\circ}\text{C}$  at near-atmospheric conditions and an elevated pressure of 42 Mpa. To the best of the authors' knowledge, this is the first time that rheological characteristics of nanofluids have been investigated under these extreme conditions.

## 2. Experimental procedure

### 2.1. Preparation of nanofluids

A highly refined paraffinic mineral oil (Therm Z-32, QALCO QATAR) which is a widely used coolant liquid for heat exchangers

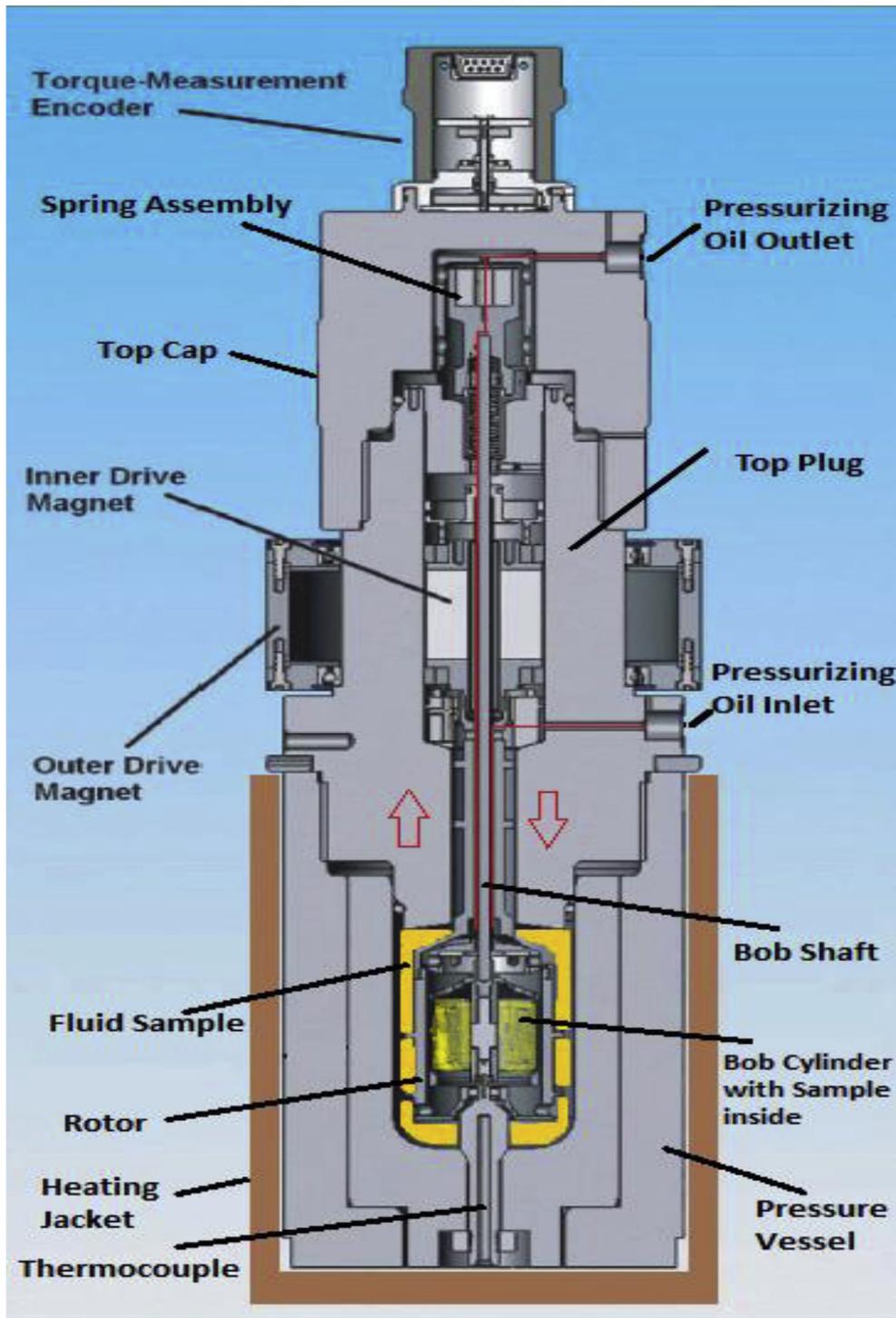


Fig. 1. Schematic of the HPHT viscometer.

in oil and gas industries in Qatar is used as the basefluid in the present study. The basefluid has a specific gravity of 0.872 and its flashpoint temperature is 215 °C. It should be noted that this heat exchanger oil has some additives (proprietary to the manufacturer) to enhance its viscosity index value to 99 and to increase its stability at higher working temperatures (~200 °C).

Nanofluids are formulated in a top down approach where commercially available SiO<sub>2</sub> nanoparticles are mechanically dispersed into a basefluid. Mechanical dispersers such as

ultrasonic baths and homogenizers have proved to be excellent tools in breaking down the agglomerates [29]. In the present case, a combination of a magnetic stirrer, a bath, and a probe-type ultrasonicator were used for dispersion of MO-SiO<sub>2</sub> nanofluids in the basefluid. Initially, appropriate amounts of SiO<sub>2</sub> nanoparticles (~20 nm average diameter, Sigma–Aldrich, #637238) were dispersed in the mineral oil using the magnetic stirrer until a homogeneous mixture was obtained. The mixture was then placed in an ultrasonic bath (VWR ultrasonic cleaner,

35 kHz) for 45 min. Furthermore, the colloidal suspension was subjected to an intensified ultrasonication by immersing a probe-type sonicator in the suspension (QSonica S-4000, 20 kHz). Cyclic ultrasonic pulses were given to the suspension for about 1 h to achieve de-agglomeration of the particles. The ultrasonication time was derived based on the suggested values for viscous oil-base fluids in the literature [29]. Experiments were conducted immediately after preparing the nanofluid to avoid chances of sedimentation. A qualitative observation of the nanofluids samples showed no sedimentation of particles in a stationary vessel, even 12 h after preparation.

## 2.2. Experimental setup

Rheological characteristics of the nanofluids were measured using an automated high pressure high temperature (HPHT) viscometer (Model 7600, AMETEK Chandler engineering) specifically designed to determine rheology of drilling fluids under extreme oil well conditions [30]. The viscometer uses a rotor and bob geometry that is widely accepted in petroleum industrial applications. This equipment meets ISO and API standards for viscosity measurement of completion fluids under HPHT conditions up to 320 °C and 275 MPa [31]. Fig. 1 shows the schematic of the entire vessel assembly of the Chandler 7600 Viscometer.

The assembly consists of three major parts: pressure vessel at the bottom, top plug in the middle, and top cap on the upper part. The bob and rotor cylinders are centered inside the pressure vessel where the fluid sample was gathered. The fluid sample (approximately 200 ml) occupies the spaces between, around, and inside the two concentric cylinders of the bob and the rotor. The rotational movement is transferred from a rotating outer drive magnet, placed around the top plug, to the rotor through the inner drive magnet, placed inside the top plug. The bob shaft extends through the whole length of the assembly and then is attached at the end to a spring assembly that is placed at the upper part of the top plug inside the top cap. The rotational movement of the rotor, at specified shear rates, causes the fluid in contact to move, which results in deflection of the bob cylinder. The angular deflection of the bob then causes a metal piece inside the spring assembly to deflect. This deflection is detected and recorded by an encoder that is placed above the top cap.

Pressurization of the system is carried out by a pump that injects hydraulic oil inside the vessel. Before pressurizing the system, the oil is circulated at low pressure in the whole system to push air outside of the pressure lines and the vessel assembly. After this stage, the system operates in a closed mode at a set pressure and temperature. The pressurizing oil enters the vessel assembly through an inlet in the top plug and pressurizes the fluid sample in a separate conduit without getting mixed in with it in the bob-rotor section. The oil pressure is adjusted through a pump and a valve and set by the user through the equipment's software program. The oil outlet in the assembly is located at the upper part of the top cap. The red arrows (in web version) in Fig. 1 show the direction of movement of the oil in the vessel assembly during oil circulation. Once the valve is closed, no oil movement occurs and the pressure in the whole system will remain the same. Heating is achieved by inserting the pressure vessel inside the heating jacket which transfers heat from an electrical heater to the fluid sample through conduction. The heat of the sample is detected by a thermocouple. This system is not equipped with a cooling system.

The equipment was calibrated before experimenting using a Newtonian calibration fluid with a known viscosity value. For a specific range of shear rate values, the equipment automatically calculates the values of shear stress using the known viscosity

values. The calculated shear stress is then compared to the angular bob deflection recorded by the Encoder.

## 2.3. Experimental schedule

The HPHT viscometer used in this work is specially designed for oil drilling exploration. The viscometer is an automated unit that requires initial programming of an operating schedule for the entire experimental cycle. This schedule includes duration and magnitude of each experimental condition for the viscometer at which viscosity data is recorded automatically in a special order. Extensive initial tests were conducted to obtain a steady state operating schedule to obtain repeatable experiments.

Fig. 2 depicts a sample of an experimentation schedule used in the present study for basefluid as well as nanofluid experiments conducted at 42 MPa. Variation of pressure, temperature, and shear rate is illustrated and normalization in the plot is done with respect to those respective maximum values. The viscometer operates in a closed controlled volume mode for measurement of heavy suspension fluids at high pressure and temperatures where the system pressure may slightly increase with an increase in temperature. Thus, it is only possible to achieve a near-constant pressure condition for viscosity measurements. This could be cited as one of the limitations of the high-pressure viscometer in estimating viscosity values at atmospheric conditions.

The sample schedule in Fig. 2 shows that in one measurement initially the system pressure is raised and is then maintained at the desired value with a variation of  $\pm 0.1$  MPa. A temperature change is then applied to the sample fluid. Once the sample temperature is almost steady, measurements are made at varying shear rates. For each temperature step, the rotor speeds are varied in increments of 100, 200, 300, 450, and 600 rpm generating shear rates of 170, 340, 510, 766, and 1021  $s^{-1}$  respectively. Measurements are taken for a time period of 1 min for each shear rates. While the fluid is heated for the next set temperature, the rotor speed is kept at a value of 150 rpm until reaching a steady state. This constant stirring is expected to prevent nanoparticles from sedimentation, as the total experimentation time is around 3 h.

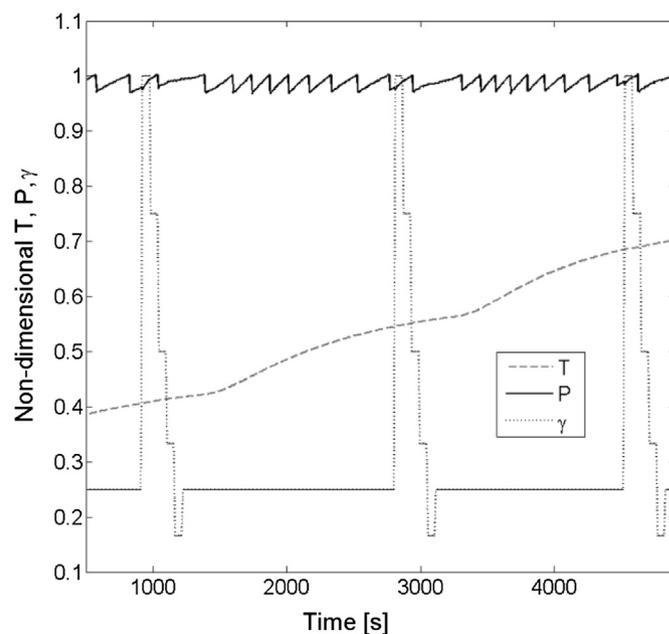


Fig. 2. Sample of experimentation schedule (at 42 MPa) that was used for rheological evaluation.

### 3. Results and discussion

After careful preparation, rheological characteristics of nanofluids were quantified. Experiments were carried out immediately after dispersion of the nanofluids to avoid any effects of particle sedimentation. Even though ultrasonication helped in degassing the nanofluids, an occasional marginal and unavoidable bubble entrapment was observed while filling the viscometer due to the high viscous nature of the basefluid. Extra care was taken to avoid the air entrapment or bubble formation while filling the pressure vessel with test samples in the viscometer.

Initially, viscosity measurements were carried out at near-atmospheric pressure conditions. Experimentation was conducted using the basefluid and nanofluids with 0.3% volume fraction to establish a working schedule for the viscometer and to validate the repeatability of the data. Initial results showed no detectable change of viscosity for nanofluids beyond the uncertainty range of the experiments. Consequently, detailed experiments were conducted only at a higher concentration of nanofluids. In this work, two particle volume concentrations of 1% and 2%, corresponding to the weight ratio of 3% and 6%, respectively, are presented for rheological evaluation.

Fig. 3 illustrates the viscosity values measured for basefluid and the two nanofluids at near-atmospheric pressure conditions. Viscosity values were evaluated as the ratio of shear stress to shear strain and are plotted against measured sample temperatures. The depicted values of viscosities are averaged values collected for 1 min at a sampling rate of 1 Hz. Viscosity values are presented for a shear rate of  $510 \text{ s}^{-1}$  for all the samples. The viscosity of the basefluid, as specified by the manufacturer, is 32 cP at  $40^\circ\text{C}$ . The measured value from the rheometer (38 cP at  $40^\circ\text{C}$ ) matches comparatively with that of the specified value. As mentioned before, since the measurement volume of the viscometer is closed, the system pressure increases slightly with the increase in temperature. Thus, it is only possible to achieve a near-atmospheric pressure condition where the system gauge pressure is observed to have increased slightly above 1 MPa when the fluid temperature goes beyond  $100^\circ\text{C}$ . The maximum uncertainty in viscosity measurements is estimated to be  $\pm 1.2$  cP.

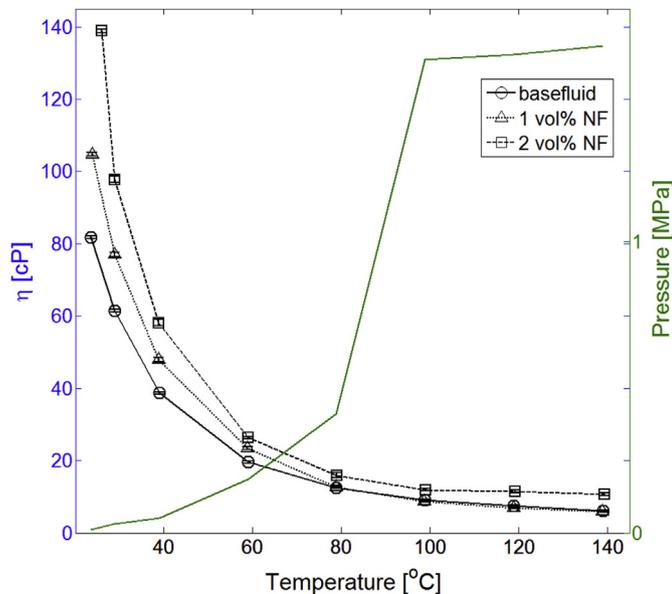


Fig. 3. Viscosity of nanofluids measured at near atmospheric pressures.

Note that the viscosity values of both nanofluids and the basefluid decrease non-linearly with an increase in temperature. This non-linear trend agrees with other earlier observations for nanofluids [11,14]. As expected, viscosity values were also observed to have increased with an increase in particle loading. However, the increase in viscosity occurred more at lower temperatures than at higher temperatures. The viscosity values are almost not changing after reaching  $100^\circ\text{C}$  for 2% nanofluids. Similar trends for an ethylene glycol–CuO mixture were reported by Namburu et al. [16] at a temperature range of less than  $50^\circ\text{C}$ .

Fig. 4 shows the consolidated results of viscosity measurements made at both near-atmospheric conditions and at elevated pressure of 42 MPa, measured at a constant shear rate of  $510 \text{ s}^{-1}$ . This figure shows that with the increase in pressure the viscosity values of both nanofluids and the basefluid increases, however, the rate of change of viscosity with temperature remains almost the same. It was also observed that viscosities of MO as well as  $\text{SiO}_2$ -MO nanofluids decreased with the increase in temperature following a power law. For the basefluid, at near-atmospheric conditions, the power law follows as:

$$\eta = 8980T^{-1.49} \quad (1)$$

A careful observation of Fig. 4 reveals that slopes of the curves in the log–log plot remain constant at lower temperatures. However, this initial trend in the variation of viscosity with temperature suddenly changes at higher temperatures. Around  $100^\circ\text{C}$ , a less temperature sensitive trend is observed for nanofluids at all pressures even at low particle loading. Probable cause for this behavior could be due to the combined effect of the non-Newtonian nature of these fluids along with a change in the chemical composition that might have occurred at high temperatures and pressures, especially in the presence of the nanoparticles. Analyses of the shear dependence and chemical composition of the samples tested here are provided in later sections.

Fig. 4 demonstrates the effect of pressure on the viscosity of nanofluids for different particle concentrations. This variation may be put in perspective of lubrication theory where a pressure–

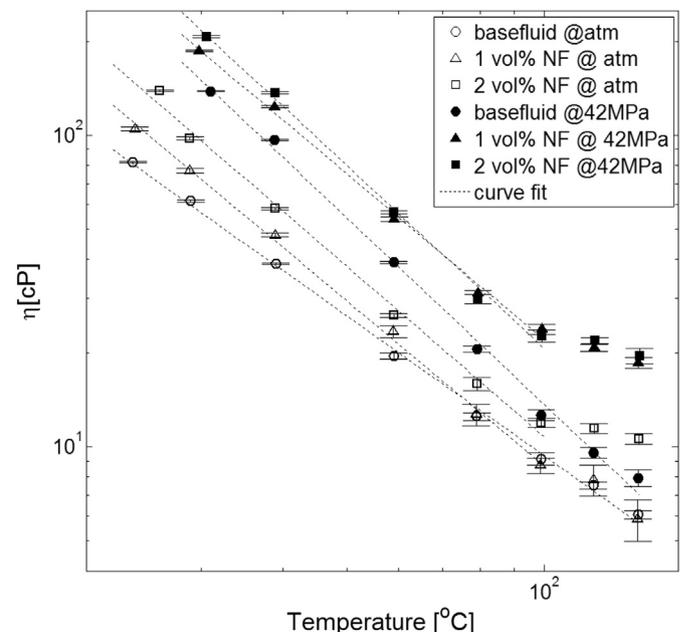


Fig. 4. Comparison of viscosity values of nanofluids at near atmospheric pressures and at 42 Mpa.

viscosity coefficient is estimated in an exponential function in accordance [32].

$$\eta = \eta_a e^{\alpha P} \quad (2)$$

Table 1 shows the pressure coefficient values evaluated for the basefluid and nanofluids at various temperatures. As observed in Fig. 4, this table shows that at atmospheric temperature (~30 °C) the effect of pressure on both the basefluid and nanofluids is of similar magnitude. It was observed that the increase in temperature reduced the pressure sensitivity of the basefluid by about three folds. The nanofluids, on the other hand, were able to maintain a similar magnitude of pressure dependence even at higher temperatures because of the additional particles suspended inside them. It is important to note that the above arguments are qualitative as the estimated pressure coefficients are based on only two pressure points. More detailed experiments are required to quantify these effects.

When applying nanofluids in heat transfer application, an important factor that needs to be considered is the increase in viscosity values with respect to the change in thermal conductivity [7]. Prasher et al. [7] demonstrated that application of nanofluids would be advantageous in a laminar flow through a tube if the ratio of  $C_\eta/C_k$  is less than a value of four (assuming similar Nusselt number for nanofluid and basefluid).  $C_\eta$  and  $C_k$  are viscosity and thermal conductivity enhancement coefficients obtained, respectively.

$$\frac{\eta_{nf}}{\eta_{bf}} = 1 + C_\eta \phi \quad (3)$$

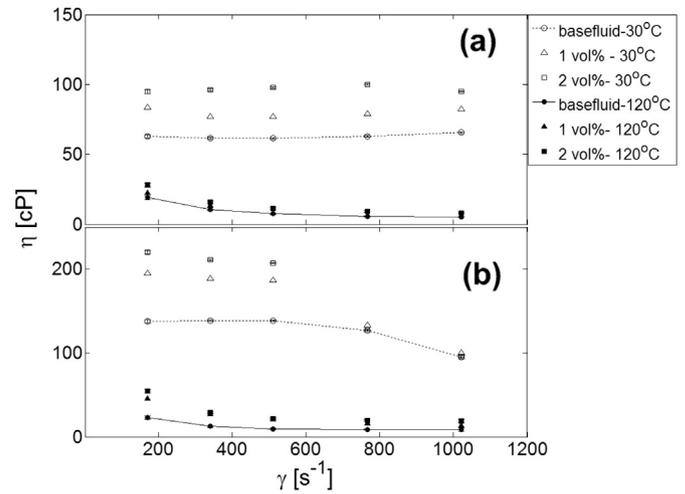
$$\frac{k_{nf}}{k_{bf}} = 1 + C_k \phi \quad (4)$$

For the nanofluids investigated here,  $C_\eta$  has an approximate value of 20. Following the effective medium theory [7],  $C_k$  has a value of 3, which makes the  $C_\eta/C_k$  ratio to be more than 4, suggesting that the application of these nanofluids for cooling would be non-beneficial.

Another interesting observation is the non-Newtonian behavior of nanofluids at elevated pressures and temperatures. Fig. 5(a) shows viscosity variation with shear rate at near-atmospheric pressure. It is observed that both the basefluid and nanofluids exhibit Newtonian characteristics at room temperatures (~30 °C), however, at elevated temperatures (~100 °C), a slight shear thinning behavior is observed. The reason behind the non-Newtonian behavior of the basefluid at elevated temperatures is unknown. It is possible that the additives in the basefluid (for increasing viscosity index values) could have caused the non-Newtonian behavior. For the nanofluid suspensions, these results are consistent with the non-Newtonian behaviors previously reported in the literature for oil and glycol-based nanofluids [14,21]. Cheng et al. [27] suggest that shear thinning in colloidal suspensions could occur when shear effects overrides the thermally induced Brownian motion, which otherwise causes the random dispersion of particles in the suspension.

**Table 1**  
Values of pressure coefficient  $\alpha$  (MPa<sup>-1</sup>) at various temperatures ( $\pm 1$  °C).

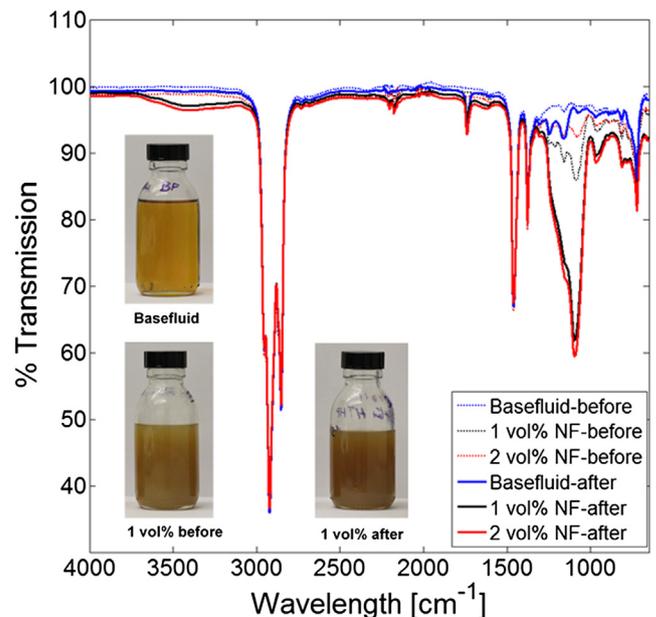
	30 °C	60 °C	100 °C	140 °C
Basefluid	0.019	0.016	0.007	0.006
1% nanofluid	0.021	0.019	0.023	–
2% nanofluid	0.018	0.018	0.015	–



**Fig. 5.** (a) Variation of viscosity with shear rate (a) at near atmospheric pressures and (b) at 42 MPa.

Fig. 5(b) shows the measured viscosity at the elevated pressure of 42 MPa. A small shear thinning behavior is observed at 30 °C for shear rates of less than 500 s<sup>-1</sup>. The magnitude of this shear thinning seems to be enhanced at higher temperatures. This figure shows that at higher shear rates the shear thinning behavior of the basefluid is slightly enhanced whereas the change in the trend for nanofluids is more. Small shear thinning behavior is observed at the higher temperature of 120 °C, similar to that at 30 °C, except that the nanofluids show stronger non-Newtonian behavior at this temperature. No effort was made to propose correlations for the nanofluids' viscosity at HPHT condition due to the observed non-Newtonian nature.

The nanofluid samples taken after the experiments showed slight discoloration (the nanofluid samples were darker) which called for chemical analysis of the samples. An infrared spectroscopy analysis (FTIR) of the fluids before and after experimentation was performed in order to investigate whether any chemical



**Fig. 6.** FTIR spectrum of basefluid as well as nanofluids taken before and after HPHT experimentation.

modification of the nanofluid composition had occurred during the HPHT tests. Infrared spectroscopy has been an important technique for materials analysis in laboratories. This technique works on the basis that different molecules of the test sample absorb specific wavelengths of light. The resulting spectrum of the transmitted light is then analyzed to provide a “fingerprint” of the sample by identifying the absorption peaks which correspond to the frequencies of vibrations between the bonds of the atoms of the sample material. Since each material is a unique combination of atoms, no two compounds produce the exact same infrared spectrum. Therefore, infrared spectroscopy can be used to identify functional groups by cross-referencing the sample spectrum with those of their standard spectrums, such as alkanes, amines, carboxylic acids, etc. [33], and to detect a change in the material structure [34,35]. Kole and Dey [36] applied the FTIR technique to verify the absence of any chemical interaction between nanoparticles and the basefluid in a CuO-gear oil nanofluid.

Fig. 6 shows the Fourier Transform Infra-red (FTIR) spectra obtained using a Perkin Elmer Spectrum-one VB spectrometer for the basefluid as well as the nanofluids before and after high pressure and temperature application. Here, the spectrum of the basefluid and nanofluids before the HPHT are represented with dotted lines, whereas their spectrum after experimentation is represented in continuous lines. A photograph of the basefluid together with nanofluid (1% concentration), before and after experimentation, is given in the inset of Fig. 6. Note that the spectrum of the basefluid, before and after experimentation, exhibited no major differences. In addition, the spectrum of both nanofluids before experimentation also showed similar absorption peaks as that of the basefluid. This indicates that the addition of nanoparticles is not making a major change in the spectrum. However, after the high pressure/high temperature experimentation, the nanofluids showed a major absorption peak occurring around a wavelength of  $1100\text{ cm}^{-1}$ . The observed change at this wavelength may be due to the poly-chain formation of Si–O–Si structures in nanofluids that could occur due to high pressure and temperature. The Si–O–Si absorption becomes broader and more complex when the chain becomes longer and branched [37]. This indicates that the high pressure and temperature condition of the fluid facilitates a chemical composition change in the nanofluid system that might be undesirable in many of the practical applications. This difference in chemical composition is believed to be the reason for the change of the measured viscosity trends at higher temperatures as shown in Fig. 4.

#### 4. Conclusions

Rheological characteristics of SiO<sub>2</sub>-mineral oil nanofluids were investigated at elevated temperatures and pressures using a high pressure high temperature viscometer. In addition to the basefluid, two volume particle concentrations of 1% and 2% of nanofluids were investigated. The following observations were derived from present investigation:

- Nanofluid viscosity values increased with an increase in particle concentration.
- With the increase in temperature, viscosity values decreased following a power law for all the cases and, at temperatures below  $100\text{ }^{\circ}\text{C}$ , there was no substantial reduction in viscosity values of nanofluids.
- The viscosity values of both the basefluid and nanofluids increased with the increase in pressure. The measured viscosity values for the basefluid and nanofluids show similar changes with pressure at room temperature. However, at higher temperatures viscosity of the basefluid is more affected by the pressure than that of the nanofluids.

- Viscosities of nanofluids are unexpectedly altered at higher temperatures and pressure. The infrared spectroscopy analysis of the tested samples indicated a chemical alteration of the nanofluids when operated at high temperatures and pressure.

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