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EFFECT OF CHEMICAL MODIFICATION ON THE FLOW AND ELECTRICAL PROPERTIES OF NEEM OIL DERIVATIVES

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ABSTRACT
Seed oil has been proposed as alternative base fluid for drilling mud but its stability to oxidation is still an issue to deal with. This paper reports the influence of stabilizing neem oil through chemical modification on its flow and electric conduction properties. Ester derivatives were produced from laboratory purified neem oil. The purified neem oil (PNO) was epoxidized to convert the double bonds to epoxy rings in order to eliminate the weak pi-bonds that are susceptible to oxidation. The derived products from these controlled reactions were verified using Fourier Transform Infrared (FTIR) spectroscopy. The viscosity of the epoxy neem was found to have increased. Glycerol was then removed from the epoxy oil to produce alkyl ester of epoxy neem oil. This reduced the viscosity of the epoxy oil. The mid frequency dielectric response of the PNO and its derivatives are related to ionic conduction. PNO has an electrical conductivity of 1.26 × 10^{-9} S·m^{-1} at 20ºC; this is about 100 times higher than the conductivity of diesel oil used as base oil for drilling fluid. The bulk conductivity is thermally activated (activation energy = 0.31 eV) and influenced by the liquid viscosity. A higher conductivity was observed for the ester derivatives compared to that for PNO and this may be attributed to polarization of suspended nano-impurities. The obtained results support the proposition that suspension of conducting nano-particles in the neem oil derivatives will pave way for the production of a suitable base liquid that will provide lubrication and electric current path for a sustainable oil-based drilling fluid for offshore drilling.

Keywords: *Drilling fluid, Neem oil, Epoxidation, Sustainable materials, electrical conductivity*

1. Introduction

Drilling fluids, often refer to as drilling muds, are part of the system for drilling oil and gas wells. Drilling fluids processed from different chemical formulation have been used over the history of onshore and offshore development of oil and gas resources. While drilling the well, the drilling fluid cools and lubricates the drill bit and strings, helps to convey rock debris and drill cuttings from the drilling area to the surface, and brings information back to the surface. Drilling fluids are expected to have the correct heat transfer and fluid-flow characteristics to effectively perform their function. It is required to be environmentally friendly. It also provides electric current path for resistivity measurement to study the complexity of oil and gas reservoirs. Water based Mud (WBM) is the most common drilling mud because of its ability to maintain borehole stability. Fresh water is the solvent for water base fluid (WBF) and has conductivity ranging from 1 × 10^{-2} S/m to 2 × 10^{-1} S/m [Kestin *et al.*, 1978] and viscosity of 1.002 × 10^{-3} Pa·s at ambient temperature [CTW, 2004]. Ultra-pure water is a bad conductor but dissolved ionic species in the water increased its conductivity. These ions transport electric current in WBF. Salt water has higher electrical conductivity. But the adverse reaction of WBF to shales usually triggers hole-caving and this prompted the need of Oil Based Muds (OBMs) [Cheung *et al.*, 2001]. WBM was also reported to be unstable in the drilling of deep-hole and not suitable in drilling of salt, anhydrite, gypsum, and mixed salt zones and drilling through hydrogen sulfide (H_2S) and carbon dioxide (CO_2) containing formations, etc [Neff *et al.*, 2000]. Oil-based Muds are preferred for drilling in locations with such geological configuration. OBMs have a number of advantages over WBMs: they increase lubricity and decrease torque and drag when drilling a directional well, they minimize the likelihood of differential sticking of the drill pipe in the hole, serve as a packer fluid for corrosion control, and as a work-over fluid in a place where water might damage the formation [Neff *et al.*, 2000]. The fluid frequently used in conventional OBMs is diesel oil. But unlike water, diesel oil has electrical conductivity of 1.07 × 10^{-10} S/m [Gardener *et al.*, 1983] and viscosity of 2.05 × 10^{-3} Pa·s [Esteban *et al.*, 2012] at ambient temperature. The poorly conducting diesel oil serves as solvent for OBM and the mud flakes form a resistive barrier between the electrode and the wall. This resist charge transport and renders the use of conventional resistivity-imaging technique ineffective for geologists to gain insight in the complexity of oil and gas reservoirs and limiting the options to ultrasonic devices and dip-meter tools which could increase the cost and may not give detailed geological information [Ceung *et al.*, 2001]. In recent time, environmental consideration and cost of disposal of conventional oil base muds created a renewed interest in the search for environmentally superior alternative oil based muds for use as drilling fluid for offshore drilling. But most of the sustainable synthetic drilling fluids have low conductivity [Cheung *et al.*, 2001]. There is therefore the quest for developing a chemically stable and electrically conducting liquid as base liquid for drilling mud.

Synthetic base mud (SBM) was developed to replace the mineral base oil but it is expensive [Caenn *et al.*, 2011]. Oil from plant seeds was considered as a possible alternative base liquid for drilling mud. Its high flash point made it very attractive but low pour point, poor thermo-oxidative stability and poor conducting property are still a challenge [McShane *et al.*, 1999]. But the

earlier use of vegetable oil failed but esters prepared from natural fatty acid and alcohol showed promising results and appeared to be the first commercially available synthetic fluid. Synthetic fluid is associated with specific drawbacks namely high cost, degradation at elevated temperature and expensive management of drilling waste with adverse effects. The depth of borehole also affects the properties of synthetic fluid since it is more compressible than aqueous liquid. Pressure increases with depth and dynamic viscosity increases with increase in pressure. Decrease in temperature can also significantly increase the dynamic viscosity [Growcock *et al.*, 2011]. Suspension of carbon nanotubes in a vegetable oil was reported to produce a base fluid of 1 S/m but the patent was silent on the stability of the nano-fluid from vegetable oil [Zanten, 2014].

The introduction of Oil-Based Drilling Mud was based on its high stability. But high temperature degrades the carbon-to-carbon double bonds of drilling muds with time and the rate of degradation is a function of temperature [Caenn *et al.*, 2011]. One of the basic challenges in producing natural ester-based industrial fluid is synthesizing a fluid with low pour point and high thermo-oxidative stability at high temperature. The thermo-oxidative stability of the oil is a function of the degree of saturation. Vegetable oil becomes more susceptible to oxidation as the degree of unsaturation progresses from mono-unsaturation to poly-unsaturation. The relative instability of fatty acids to oxidation is estimated to be 1:10:100 for saturated, mono-, and poly-unsaturated C-18 triglycerides respectively [Oommen, 2002]. The commonly used method for thermo-oxidative stability of drilling fluid formulated from vegetable oil is the addition of antioxidant to the natural ester based fluid [Caenn *et al.*, 2011].

Neem trees are scattered around Nigeria and neem seeds which contained oil sometime constitute environmental nuisance. Development of Industrial fluids from these seeds will add to its economic importance. Neem oil extracted from the seed is triglyceride with 13.8% palmitic fatty acid, 18.2% stearic fatty acid, 1.8% Archidic fatty acid, 52.6% oleic fatty acid and 13.6% linoleic acid [Dasa Rao *et al.*, 1942]. 66.2% of fatty acids in neem oil are unsaturated and that makes it susceptible to thermo-oxidative degradation at elevated temperature. Thermo-oxidative stability of natural ester has been found to have improved significantly with modification of oil structure through the C=C bond [Hwang *et al.*, 2001]. The chemical modification can also have effect on the viscosity and electrical properties of the fluid [Abdelmalik *et al.*, 2011]. The chemical processing can influence the electrical properties of the fluid as mobile charges may be higher in such fluids [Growcock *et al.*, 2011]. Modification of the chemical structure of vegetable oil is becoming more popular as the modified oil may be used as raw material for environmentally friendly products.

Development of suitable drilling mud is in three stages. The first stage involves identifying a suitable base liquid that can serve as lubricant and heat transfer liquid. This is then followed by improving the undesirable properties of the liquid. The last stage involves the suspension of solid additives in the liquid to produce the complete drilling mud. In this work, an attempt has been made to study the feasibility of using chemically modified neem oil as lubricant and a possible based liquid for oil-base drilling mud. It also evaluates the potentials of the liquid with a view to providing a current path through the drilling mud for logging with conventional resistivity imaging devices.

2. Experimental

2.1. Materials

Crude neem oil sample was extracted from need seed obtained from Zaria at the National Institute of Chemical Research, Zaria, Nigeria, citric acid, sodium hydroxide, silica gel, fuller's earth, methanol, sodium sulphate, acetic acid, toluene, sulphuric acid (H_2SO_4), hydrogen peroxide, phosphoric acid are the chemicals used for this work. CNO is the crude neem oil, PNO is the purified neem oil, ENO is the epoxidized neem oil and ENOE is the methyl ester of the epoxidized neem oil.

2.2. Purification

A modified Dijkstra and Opstal purification method [Abdelmalik *et al.*, 2011] was adopted for the purification of the sample to obtain the Purified Neem Oil (PNO) sample. 200 ml of crude neem oil sample was heated in a 500 mL conical flask to 70˚C and 8 vol.% of 64% aqueous citric acid solution was added gently and mixed thoroughly with a magnetic stirrer for 15min. 4 ml of 8% NaOH solution was gently added and the mixture stirred at 400 rpm for 15min. The mixture was then dried in vacuum oven at 85°C for 30 min to reduce the water content. 2 g of silica gel was added to the mixture at 70°C and agitated for 30 min at 300 rpm to prevent it from settling out. Fuller's earth was then added and continuously stirred for 30 min at 85%. The sample was then filtered with filter paper in vacuum oven at 85°C.

2.3. Epoxidized Neem Oil

PNO was passed through epoxidation reaction to eliminate double bond and create epoxy ring in its place as shown in Figure 1. This was prepared by placing 50 g of Purified Neem Oil (PNO) in a three-necked flask. 12.5 g of glacial acetic acid was poured into a round bottom flask and this was followed by the addition of 12.8 g of toluene. 12.5 g of H_2SO_4 was added to the mixture as a catalyst. This was followed by dropwise addition of 35 g of H_2O_2 from 250 ml quick fit separatory funnel fitted to one of the necks of the reactor flask. The mixture in the separatory funnel was then added drop wise to the mixture in the reaction flask. The reaction was allowed to continue for 4 hours at moderate stirring speed at 55°C. On completion, the reaction mixture was poured into a separatory funnel and the bottom layer was discarded and the upper layer was collected and washed with cold distilled water 3 times and then with slightly hot distilled water. The sample was then dried over sodium sulfate and impurities were removed from the prepared Epoxidized Neem Oil (ENO) and the toluene in the oil was removed.

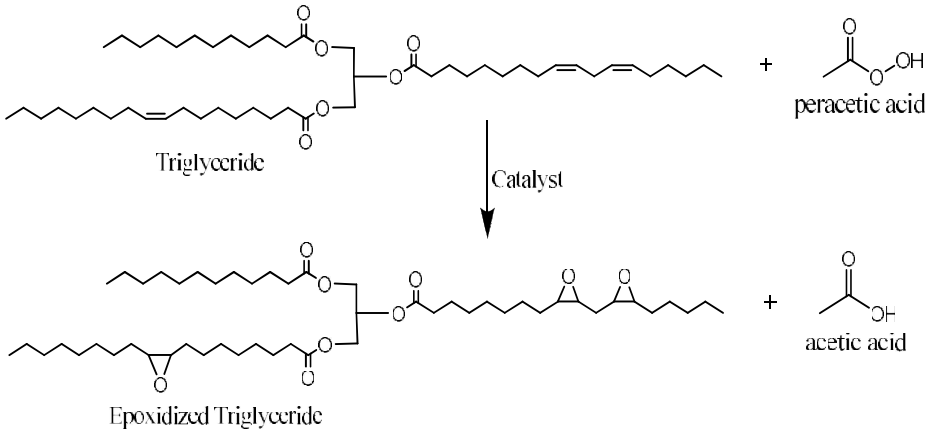

Figure 1: Reaction scheme for epoxidation reaction

2.4. Epoxidized Neem Oil Ester

ENO was then transesterified to separate the glycerol from the neem fatty acids to produce epoxidized ester. 50 g of purified sample of ENO was introduced into 250 mL flat bottom glass vessel along with a magnetic stir bar. 15 mL methanol was then added to the oil sample in the flask. This was the followed by the addition of 0.5 g (1 wt %) NaOH into the mixture. The mixture was then heated to 50°C on a hotplate and continuously stirred for 2 hours. The mixture was transferred into a 500 ml separatory funnel and left for about 4 hours to separate. The bottom layer which contained glycerol was discarded and the top layer was collected. The collected mixture was washed with 0.015 M phosphoric acid and the emulsion was kept in oven under 60ºC for an hour to separate. The bottom layer which contains remnant impurities and NaOH was discarded and the top layer containing alkyl ester of the oil and methanol was collected and dried over anhydrous sodium sulfate. Finally, the collected sample was then heated in vacuum oven at temperature above boiling point of methanol to discard the remnant.

2.5. Characterization

The yield of the neem oil samples was monitored by taking aliquot of the sample for FTIR analysis. The FTIR analysis was performed using Shimadzu-8400s Fourier transform infra-red spectrophotometer. The functional groups present in the three (3) neem oil samples (PNO, ENO and ENOE) were analyzed using the FTIR spectroscopy. The viscosity of the liquid samples was measured using Brookfield Viscometer with the appropriate spindle. The sample container was placed in water bath heated on a thermostatically controlled hot plate to vary the temperature of the sample within accuracy of ±0.1ºC. Temperature sensor was immersed in the water bath to monitor the temperature. Each measurement was taken three times and the average calculated for accuracy. The conduction property of the liquid samples was studied using HM8118 LRC Bridge within frequency of 20 to 200 kHz. The liquid was placed in a cylindrical test cell and placed in a temperature controlled heat chamber. The dissipation factor was measured from the bridge within temperature range from 20°C to 70°C. The study under frequency spectrum is to understand the loss mechanism that result in conduction in the liquid. The dissipation factor which is also referred to as loss tangent or tan δ is obtained from the ratio of the leakage current density to the capacitive current density.

3. Results and Discussion

The samples are shown in Figure 2.

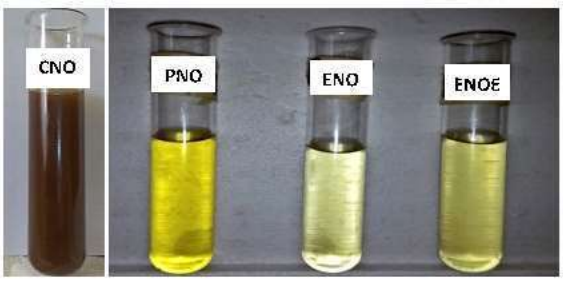

Figure 2: Oil Samples

Earlier experiment shows that unsaturated fatty acids in vegetable oil can break down at elevated temperature over a period of time and results in polymerization as shown in figure 3 [Abdelmalik, 2012]; this can affect fluid flow in an application where vegetable oil is expected to flow freely. Epoxidation removed the unstable carbon-to-carbon double bond in the oil to create a stable liquid that is less susceptible to thermo-oxidative degradation.

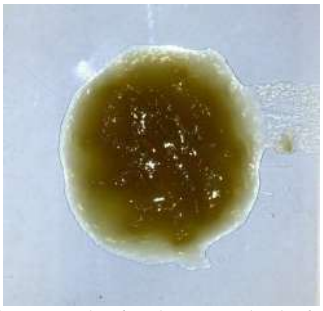

Figure 3: Photograph of Polymerized oil after 84 Days at 150˚C in Ageing Vessel

3.1. FTIR Analysis

The FTIR spectra (Figure 4) showed characteristic peaks of the functional groups contained in the samples. The spectra for PNO sample possesses peak at about 1660 cm^{-1} which is typical of C=C stretching vibration usually seen as moderate to weak absorption in the range of 1667 to 1640 cm^{-1}. The absence of the peak in ENO and ENOE

samples is an indication that there is a complete conversion of the double bonds in the PNO during the epoxidation reaction. Characteristic band due to =C–H stretching vibration of the carbon-carbon double bond was observed around 3003 cm^{-1} in PNO spectra. This band was observed to be absent in the spectra for ENO and ENOE indicating the absence of carbon-carbon double bonds in the epoxidized samples. The peak at 1024 cm^{-1} which was observed on the spectra for ENOE is a fingerprint of the alkyl ester of long-chain fatty acid [Silverstein *et al*., 2005]. The fingerprint for epoxy ring was reported to appear around 844 and 829 cm^{-1} [Abdelmalik *et al.*, 2011]. The observed broad peak in ENO and ENOE sample in figure 4 within the lower region of the spectrum (about 850 cm^{-1}) is due to the presence of epoxy functional group. ENO and ENOE exhibit a broad peak around 853 cm^{-1}. This is an indication that epoxy ring produced is not destroyed during transesterification. Peaks at 719, 730 and 728 cm^{-1} in the three samples respectively, are associated with methylene rocking (bending) vibration of CH_2 in which all methylene groups of straight chain alkanes in the samples vibrate in phase.

3.2. *Viscosity*

The base oil for drilling mud is expected to be able to flow at extreme low temperature and stable during oxidation at elevated temperature to enable it perform the cooling and lubricating function effectively. The flow property of drilling mud plays a major role in the removal of rock chips from the cutting face of the bit to the surface and the velocity of the drilling mud. Low viscous liquid is often preferred for effective cleaning at the bit face and rapid settling of cuttings at the surface. There are situations where high viscous liquid may be necessary. This includes the removal of coarse sand from the hole or to stabilize gravel. But the high viscosity has a negative influence on retarding settling of the cuttings at the surface. Viscosity of the base fluid therefore plays a vital role in the flow of drilling mud. The viscosity of PNO was measure to be 0.065 Pa·s. The conversion of the double bond to epoxy ring produced epoxidized oil (ENO), a liquid more viscous than purified neem oil. The dynamic viscosity of ENO was determined to be around 0.091 Pa·s at 30˚C. When the glycerol is separated from the fatty acids, change in the physical properties occurred. The measured dynamic viscosity of ENOE was observed to reduce compared with the viscosity of both PNO and ENO. The dynamic viscosity was measure to be 0.025 Pa·s at 30ºC.

The dynamic viscosity of a liquid is directly related to its heat transfer coefficient and dependent on temperature change. In view of the above, the viscosity of the produced oil samples with changing temperature is given graphically in figure 5. The systematic decrease in the viscosity of the oil samples is as a result of increase in temperature of the oil samples brought about by the increase in the thermal or kinetic energy of the molecules in the oil samples and consequently reduces the cohesive energy of the molecules and the resistive frictional force acting between layers in the oil samples travelling at different speeds. This increases the mobility of the molecules in the oil samples and subsequent reduction in the viscosity values was observed as shown in figure 5 [Massey *et al.*, 2006].

The influence of temperature on fluid viscosity varies for different fluids and this is seen in their use for different technological purposes. As such, this varying influence on the viscosity of the oil samples are revealed with discernable activation energies obtained from the Arrhenius plot in figure 6 for the samples. The obtained activation energy for PNO is 0.12 eV while that for ENO is 0.042 eV. This implies that the introduction of epoxy ring on the long chain may have possibly slow down the speed of the molecules thereby leading to a decrease in the response of the kinetic energy of the molecules to temperature change. From the activation energy for ENOE which is 0.190 eV, the removal of glycerol from epoxy neem reduced the viscosity but did not seem to have much influence on the response of kinetic energy of the molecules to temperature change. The removal of the glycerol from ENO to produce ENOE lead to reduction in the concentration of the molecules in this oil sample. This may have resulted in increase in the speed of the molecules with increase in temperature.

3.3. *Electrical response*

The loss tangent is inversely proportional to frequency with a slope of about -1 within the frequency range of 20 Hz to 2×10^4 Hz as shown in Figure 7. This is an indication of a conduction mechanism dominating loss in the liquid.

AC conductivity is sometimes considered as the effective electrical conductivity of a dielectric material since it includes all the loss factors in the dielectric [Growcock *et al.*, 2011]. An effective AC conductivity may be defined as the ratio of the leakage current density to the magnitude of the field at a given frequency arising from conduction and dipole orientation losses; this may be expressed as [Bartinikas, 1987]:

$$\sigma = \frac{\bar{J}_l}{\bar{E}} = \omega\varepsilon' \tan\delta \qquad (1)$$

where σ is the electrical conductivity, ε'(ω) is the real component of the relative permittivity of the liquid, tan δ (ω) is the loss tangent which measures the energy dissipated by the dielectric liquid, ε'(ω) and tan δ (ω) are function of angular frequency, ω, $\bar{J}_l$ is the loss current density, and $\bar{E}$ is the electric field. The conductivity of the samples was evaluated using equation 1. The evaluation of the conductivity of the samples from the loss tangent data shows that the conductivity of the liquid is relatively constant between 20 Hz to 2×10^4 Hz. PNO has electrical conductivity of 1.26×10^{-9} S/m at 20ºC. After epoxidation reaction, the effective conductivity of the epoxy oil (ENO) increased to 7.30×10^{-9} S/m. Transesterification further increased the conductivity of the epoxy alkyl ester (ENOE) to 1.38×10^{-8} S/m. The conductivity of ENOE is a factor of 1 higher than the purified neem oil sample (PNO). The conductivity of the samples increased with increase in temperature as shown in Figure 8. The charged particles in the liquid are expected to be more mobile as the viscosity of the liquid decrease.

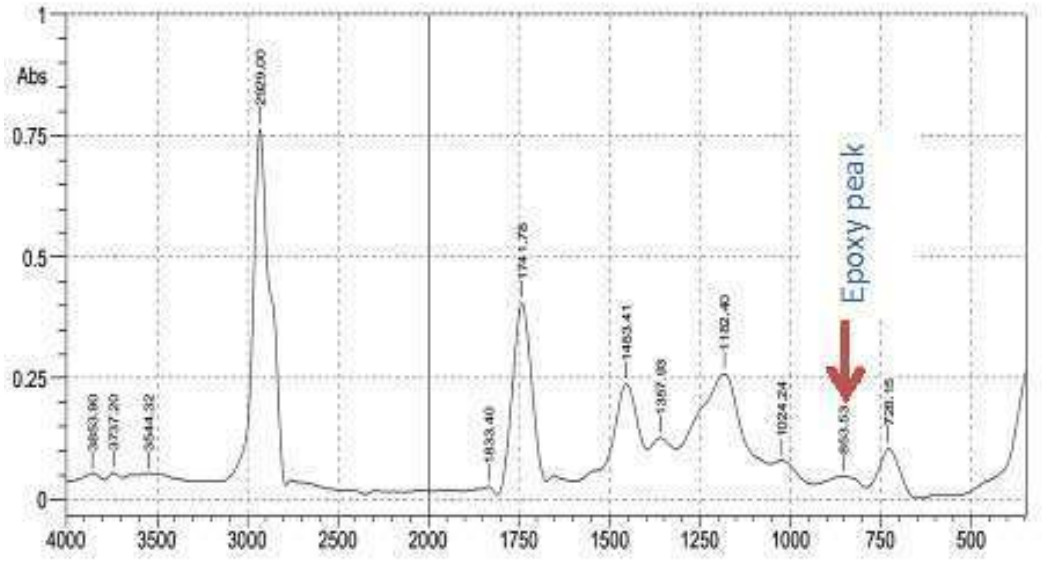

Figure 4: FTIR Spectra of Epoxidized Neem Oil Ester (ENOE)

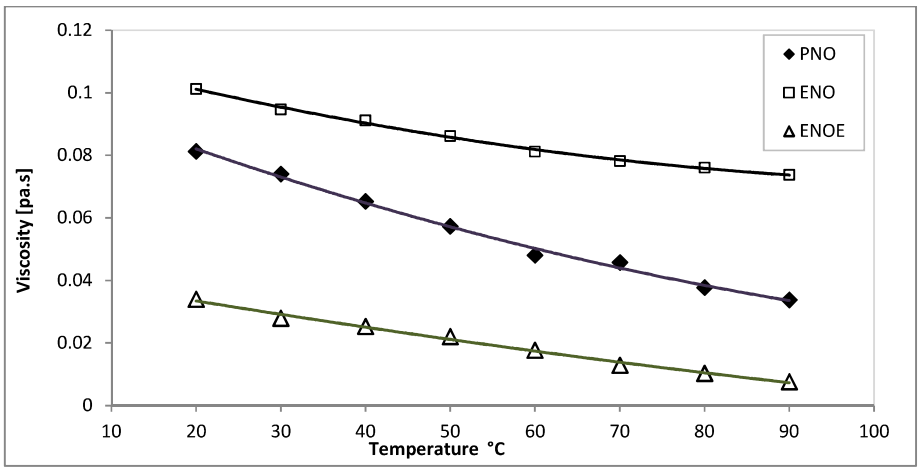

Figure 5: Viscosity versus Temperature for PNO, ENO, ENOE samples

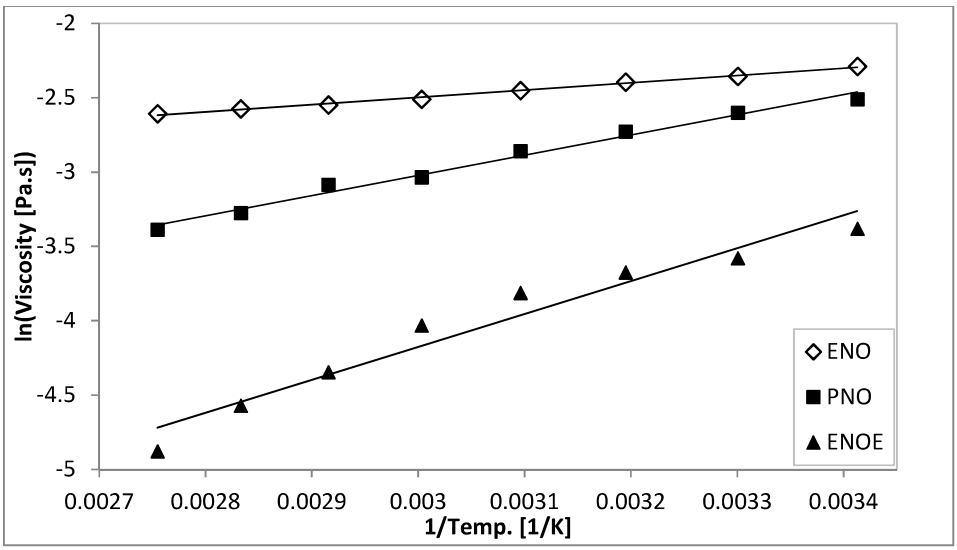

Figure 6: Viscosity-Temperature Plotted for ENO, PNO, ENOE on Arrhenius axes

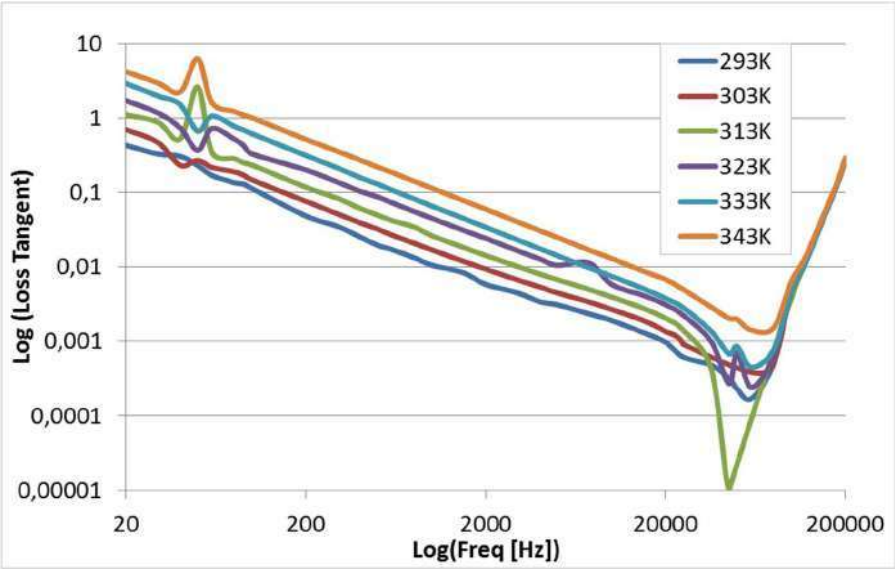

Figure 7: Loss Tangent versus Frequency for PNO

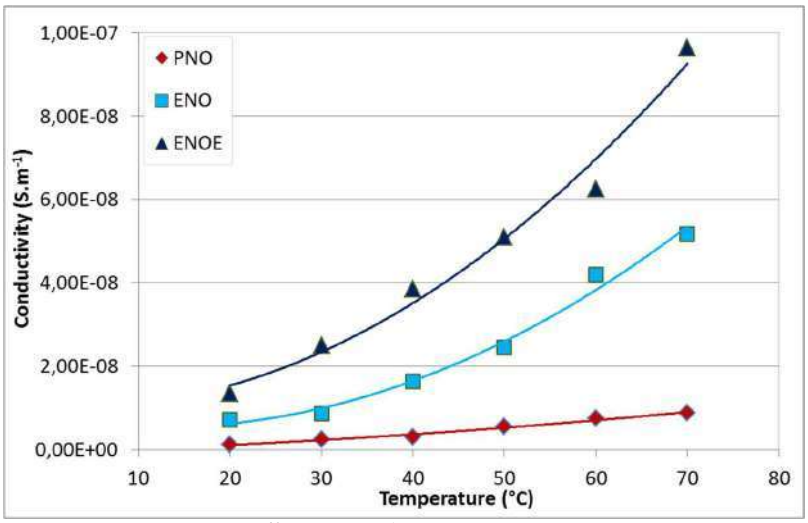

Figure 8: Effective Conductivity versus temperature

The motion of the charged particles responsible for conduction in the liquid may have resulted from the dissociation of solid impurities in the liquid. Besides causing conduction current, they give rise to polarization of the dielectric material. If the total loss in the liquid is considered as a combination of DC conductivity and loss due to polarization, the measured dielectric loss can be express as:

$$\sigma(\omega) = \sigma_0 + \omega\varepsilon_0\varepsilon'' \qquad 2$$

where σ_0 is the DC conductivity and $\omega\varepsilon_0\varepsilon''$ is loss due to polarization, ε" is the imaginary relative permittivity and ε_0. Polarization of charged particles in ENO and ENOE may be responsible for the increase in the effective conductivity of the oil samples.

The plot of change in effective ac conductivity of the samples on Arrhenius axes follows simple Arrhenius relation (figure 11). This is an indication of thermally activated transport mechanisms. The three (3) samples have close activation energies which fall within 31 kJ/mol and 35 kJ/mol. While the electrical conductivity of the samples increased from PNO through ENOE, PNO has higher activation energy and ENOE has least activation energy. This may be an indication that conductivity activation energy is a function of viscosity.

The measured electrical conductivity and viscosity of the synthesized ester fluid is compared in Table 1 with the values obtained from literature for fresh water and diesel oil [Kestin *et al*., 1978], [CWT, 2004], [Gardener *et al*., 1983], [Esteban *et al*., 2012]. The ester of epoxidized neem oil (ENOE) has viscosity higher than the viscosity of fresh water and diesel. The high viscosity may have the capability of removing coarse sand from the hole or to stabilize gravel. The electrical conductivity of ENOE is a factor of 2 higher than the conductivity of diesel oil and a factor of 6 less than the viscosity of fresh water. The presence of conducting particles may enhance the conductivity be several order of magnitude.

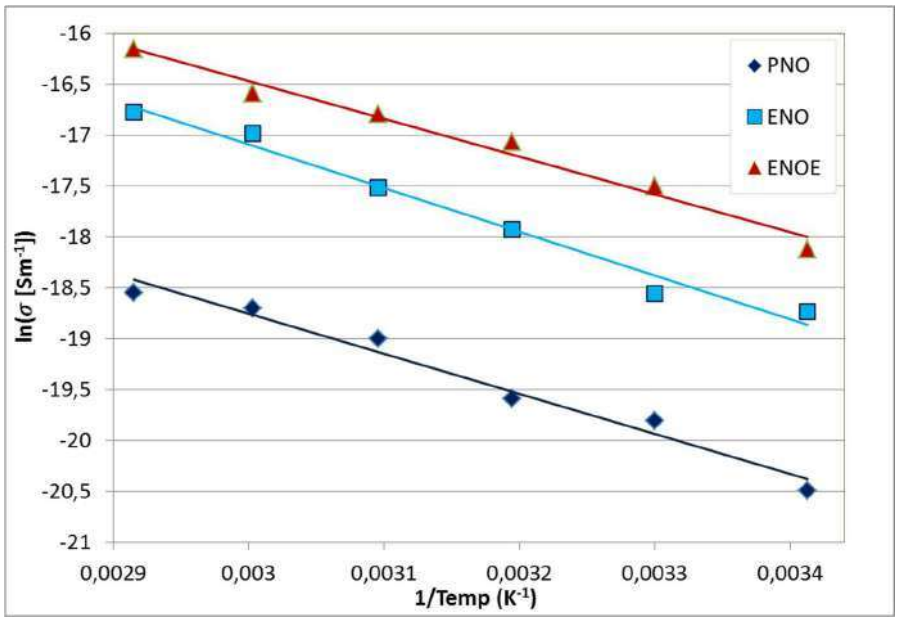

Figure 9: Arrhenius Plot for Electrical Conductivity of the Ester samples

Table1: Conductivity and Viscosity of the produced oil samples compared with selected liquid

Properties	Fresh Water	Diesel oil	PNO	ENO	ENOE
Viscosity (Pa.s)	1.002×10^{-3}	2.05×10^{-3}	0.065	0.091	0.025
Electrical conductivity (S/m)	0.01 – 0.2	1.07×10^{-10}	1.26×10^{-9}	7.3×10^{-9}	1.35×10^{-8}

4. Conclusion

Laboratory purified neem oil has been processed to produce ester of epoxidized neem oil. Replacement of the carbon-to-carbon double bond in the neem fatty acid structure with epoxy ring slightly increased the viscosity. Transesterification lead to reduction in the viscosity of the epoxy oil. The response of viscosity of the epoxy neem alkyl ester with change in temperature is slower than that of the neem oil. The dynamic viscosity of the epoxy derivatives is more stable to temperature change within the studied temperature range. The purified neem oil has conductivity of 1.26×10^{-9} S/m which is a factor of 1 higher than diesel oil (1.07×10^{-10} S/m) that is often used as base fluid for oil-based drilling fluid. After chemical modification, an increase of AC conductivity (1.35×10^{-8} S/m) by a factor of 2 was observed. This increase may not necessary be a product of the modification but that of the impurities introduced during modification. Temperature increase leads to an increase in conductivity 9.65×10^{-8} S/m. This work has produced a thermo-oxidative stable ester liquid with higher dynamic viscosity, more stable dynamic viscosity with temperature change and electrical conductivity that is a factor of 2 higher than the conductivity of mineral oil used for oil based drilling fluid. A chemical modification approach through elimination of the reactive double bond sites and replacing it with epoxy ring may be a good technique to develop a stable and sustainable lubricant and base fluid for electrical conducting oil-based drilling fluids. The epoxy ring also offers further chemical modification where side chains can be attached to the long ester chain for improved pour point. There is also an indication that polarization of the charged particles in the processed oil samples may be responsible for the increased conductivity. As a result, it is anticipated that further processing with the suspension of conducting nano-particles in the synthesized ENOE sample may increase the conductivity by several orders of magnitude and can produce liquid with suitable electrical conductivity. Oil base drilling mud with conductivity of up to 1 S/m may be produced from such synthesized ester fluid.

There is an ongoing work to produce conducting nano-particles from some agricultural waste products. The presence of conducting particles may enhance the conductivity by several orders of magnitude. Suspension of the nano-particles in the ENOE is anticipated to produce a thermally stable base fluid with desired electrical conduction property for drilling mud.

5. Acknowledgment

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