

Investigating the Effectiveness of Tiger-Nut Oil (*Cyperus esculentus* L.) as Base Fluid in Synthetic-Based Mud

Favour Ehwarieme
University of Ibadan

Sarah A. Akintola
University of Ibadan

ABSTRACT

Due to the environmental effects and following the stringent regulations on drilling operations and the associated drilled cutting disposal, drilling fluid with less environmental impact are highly desirable over the conventional diesel-based mud. This study seeks to determine the effect of ester derived from the oil of tiger nut using alkali catalyzed transesterification as base fluid for the formulation of a synthetic base mud. A total of four mud samples were formulated, three samples of 10ppg synthetic based muds were formulated using the ester at 70:30, 75:25, and 80:20 Oil-water ratio. The last mud sample was formulated using diesel as base fluid but at 75:25 Oil-water ratio. The fresh oil and its ester were characterized. The tiger nut ester was purified and the compositional analysis of the ester was carried out using the Fourier transform infrared spectrometry. The API recommended procedure was used to determine the mud samples density, rheological, filtration and emulsion properties. The result of the FTIR shows an absorption band at 1743.56 cm^{-1} indicates the presence of C=O of an ester. The result of the density of the fresh Tiger Nut Oil (TNO) and the ester are 0.98 and 0.87 respectively, the viscosities are 25.7cp and 3.25cp respectively, the flash point and pour point are 173°C and -25°C, and 160°C and -15°C respectively. The plastic viscosity and yield point of the synthetic based mud ranges between 7.0cp to 16.0cp and 9.0lb/100sqft to 46.0lb/100sqft, while 9.0cp to 14.0cp and 20lb/100sqft to 27lb/100sqft was measured for diesel oil-based mud. The filtrate volume and mud cake thickness for the synthetic based mud at 70:30, 75:25, and 80:20 oil-water ratio are 2.6ml and 1.2mm, 3.8ml and 1.1mm, 4.1ml and 1.0mm respectively, while 15.6 and 5mm were recorded for diesel oil based mud. The gel strength at 10sec and 10min of the synthetic based mud ranges between 6.0 and 11.0, while 7.0 and 8.0 were recorded for the oil-based mud. These result shows that the ester based synthetic oil compared favorably with diesel in the formulation of drilling mud. From the result, it is observed that esterified TNO can as a base fluid for a synthetic base mud, but further research on the most cost-effective way of producing the oil from tiger nut.

Keyword: Tiger nut, Trans Fourier transform infrared spectrometry, esterification, Synthetic Based Mud

INTRODUCTION

The success of any oil exploration is dependent on drilling a well to access the sub-surface resource. Drilling of hydrocarbon reserves is usually the most essential aspect of the oil and gas

industry primarily because of its consequences on the economic feasibility of the entire project. The necessity to drill a functional hole with low cost and reduced environmental impact has been a dream long held by drillers. The drilling mud generally referred to as “the blood of the drilling process” determines whether the drilling operation is either materialized or not. This is owing to the fact that the fluid plays a number of invaluable roles. Such as: cooling the drill bit, cuttings removal, controlling pressure in the penetrated formation, deposition of thin, soft and slippery filter cake on wall of the borehole, e.t.c.

The choice of the right drilling fluid and maintenance of the right properties greatly influence the total well cost as rate of penetration, avoidance of delays resulting from stuck pipes, loss of circulation, caving shales, etc., greatly affect the number rig days require to drill to total depth. Since the 1990's there has been a ban on the site discharge of drilled cuttings of Oil based muds. This has led to the development of synthetic based drilling muds. These drilling muds provides the same or even improved properties of oil-based muds coupled with the low toxicity of water-based muds, thereby allowing for discharge of drilled cuttings especially deep-water exploration thereby reducing pollution hazard caused by diesel oil-based muds.

Petroleum-based oils used for drilling mud contains large amounts of aromatics and at least a substantial concentration of n-olefins both of which may be harmful to plant and animal life, as such, the petroleum drilling industry has overtime developed various oil-based muds which reconcile performance and economic objectives with unique environmental and technical obstacles or constraints. The harmful impact of diesel OBMs on the environment and most importantly that of humans has led to several research for the past decades. This has to the use of vegetable oils as substitutes for diesel in the formulation of OBMs.

These vegetable oils are extracted from edible and non-edible sources. The most common are; Jatropha Oil, Sesame Oil, Palm Oil, Coconut Oil, Palm Kernel Oil, Rapeseed Oil, etc. In recent times, synthetic oils are now considered to be more environmentally friendly than mineral oils and diesel oil. Esters are formed by the reaction of a carboxylic acid with an alcohol under acidic conditions (Norman 1997). The ingredients of esters used in SBFs include fatty acids (carboxylic acids) with 8 to 24 carbons and alcohols with different chain lengths. 2-Ethylhexanol ($C_8H_{18}O$, molecular weight 130.2) is the alcohol used most frequently; however, mono- and poly-hydric alcohols (glycerols) may also be used. The fatty acids usually are derived from natural vegetable or fish oils. They also can be made by oxidation of the terminal double bond of LAOs Friedheim and Pantermuehl 1993).

SBF are more biodegradable and less toxic than most OBF, while retaining many of the technical advantages of OBF (Neff et al., 2000). Because of their high cost, SBF usually are recycled rather than disposed of in the environment or re-injected. However, some SBF reaches the ocean in drill cuttings discharges, where these are permitted. Cleaned SBF cuttings usually contain about 10 percent synthetic chemical (Annis, 1997; Neff et al., 2000). Cuttings cleaning technology is being developed that can reduce the concentration of synthetic on cuttings to below 5 percent. Cuttings containing less than about 5 percent synthetic do not clump when discharged to the ocean; they disperse and settle over a wide area, preventing development of a cuttings pile and speeding biodegradation (Getliff et al., 1997). A permit requires that

retention of synthetic base chemical on cuttings not exceed 6.9% for internal olefins or 9.4% for esters (Rabke et al., 2003).

A good number of deep offshore exploration has taken place in Nigeria in which Synthetic Based Muds (SBM) are been used. A good number of deep offshore exploration has taken place in Nigeria in which Synthetic Based Fluids (SBF) are been used.

Okorie, et al. (2015), among other researchers carried out tests on Soya bean Oil as the external phase of OBMs and the toxicity effect on beans plant. The formulated soybean mud properties were compared with diesel oil mud properties. The compared properties were rheological properties, yield point and gel strength, and mud density and filtration loss properties, fluid loss and filter cake. The results obtained showed that soybean oil mud exhibited Bingham plastic rheological model with low yield point and gel strength when compared with the diesel oil OBM. Mueller et.al (1993), formulated and tested SBFs which were prepared from iso-butyl oleic ester and iso-butyl rapeseed oil ester. Both esters showed a low kinematic viscosity but their poor thermal stability resulted in undesirable high plastic viscosity after aging for 16h at 125°C. Canola oil also has been used in the formulation of SBMs. Ismail et al (2001), showed that Palm oil-based esters have shown a potential of been used as drilling fluid. Studies have showed that branched ester such as isopropyl laureate shows better fluid loss fluid loss compared to linear esters such as methyl laureate. Dina kania et al (2018), evaluated the performance of polyol esters as thinners and lubricity enhancers in invert emulsion SBMs, the study showed that polyol esters enhanced the filtration properties of drilling mud. Dina kania et al (2021), investigated the rheology of synthetic base mud containing non-ionic surfactant pentaerythritol ester using full factorial design, the study showed that the plastic viscosity of SBM increases with increasing non-ionic surfactant primary emulsifier ester concentration as water-in-oil emulsion improves. The study also showed for that a better drilled cutting transport, a high concentration of secondary emulsifier and a low concentration of primary emulsifier should be used Findings have suggested that less brine in the formulation gave a better performance of canola oil drilling fluid

From the work done so far, the contribution of vegetable oils has been significant plant oil source as a substitute for diesel.

MATERIALS AND METHOD

Dry tiger nut seeds, grinding machine sodium hydroxide pellet beaker containing, Methanol. Magnetic stirrer 5000ml graduated beaker, thermometer, clamp. Heating mantle, mixing bowl. Mud Samples A, B, C represent SBMs oil-water ratio of 70:30, 75:25, 80:20 respectively, while sample D represent diesel OBM with oil-water ratio of 75:25.

Extraction of Tiger Nut Oil

Dry tiger nut seeds were bought at Otovwodo market Ughelli, Delta State, Nigeria. The nuts were sorted to remove all the dirt and bad nut. The sorted dried tiger nut was grounded using a grinding machine, kept inside a sack before being placed under a mechanically pressurized platform to remove the liquid. The extracted fluid was kept inside a bowl heated in a water bath before been heated in a pot using firewood as the source of heat. The heating process was to drive off water. Finally, it was filtered to remove dirt.

Transesterification of Tiger Nut Oil

The alkali trans-esterification of vegetable oil is the method employed in the production of the ester used in this study. A solution of sodium methoxide (CH_3ONa) was prepared by adding 6.2g of sodium hydroxide pellet into a beaker containing 104ml of Methanol. The resulting mixture was stirred with a magnetic stirrer until the sodium hydroxide pellet completely dissolves to form sodium methoxide. The transesterification reaction was carried out in a 5000ml graduated beaker placed on top of a magnetic stirrer with a thermometer held in place with a clamp. 3400ml of Tiger Nut Oil was put in the 5000ml beaker and heated to 60°C. Sodium methoxide was gradually added to the Tiger Nut Oil in the beaker. The mixture in the reaction beaker was stirred with the help of the magnetic stirrer at 200rpm and the temperature maintained at 60°C for 30 minutes.

The reaction reached completion at 30 minutes. The phase separation was achieved by allowing the mixture to settle for 1 hour. The mixture separated into two layers with the ester on top and glycerol at the bottom. The ester on top was decanted.

The decanted ester was washed with water to remove the excess catalyst and other impurities present. The washing was done in a separating funnel. The ester was poured into the separating funnel followed by water, the mixture was allowed to settle and decanted. The process was repeated until a clear water was seen at the bottom of the separating funnel, indicating that the ester is purified. The entire chemicals used were of analytical grade and the equipment used were from the department of petroleum Engineering, University of Ibadan.

Compositional Analysis of the Ester

The detection and characterization of extracted oil ester was performed directly using spectroscopic methods of analysis such as FTIR,

Characterization of the Ester

The methyl ester of Tiger Nut Oil (TNO) was characterized for its Pour point, density, kinematic viscosity, cloud point, fire point, flash point,

Formulation of Muds

The Mud samples was formulated mud as shown in the Table 1.0

Table 1.0: Composition and procedure for the mud formulation at 10ppg.

Additives	Unit	OWR			Mixing Time (min)
		Sample A 70:30	Sample B 70:25	Sample C 80:20	
Base Oil	ml	207	222	238	-
Organo philic Clay	g	1.0	1.0	1.0	10
Lime	g	0.5	0.5	0.5	10
Primary Emulsifier	g	10	10	10	15
Secondary Emulsifier	g	6	6	6	15
Brine		110.3ml H ₂ O + 48.7g CaCl ₂	94.5ml H ₂ O +48.7g CaCl ₂	78.8ml H ₂ O + 48.7g CaCl ₂	30
Fluid loss	g	5	5	5	10
Barite	g	138	142	143	20

Measurement of Mud Weight

A mud balance was used to determine the mud density. The mud balance was calibrated with water before been. To obtain the required mud weight of 10.0 ppg, the appropriate barite was added to the different mud,

Measurement of Mud Properties

The properties of the formulated muds were evaluated to ascertain the suitability of the mud

Measurement of Mud Viscosity:

A Fann V-G Viscometer was used to determine the rheological properties of the formulated SBM samples and the mud Plastic Viscosity (PV), Yield Point (YP) and Apparent Viscosity (AV), were calculated based on two point data approach from the viscometer dial reading as presented in the equation 1.0, 2.0 and 3.0. The 10 seconds and 10 minutes gel strength was also determined

$$PV = \theta_{600} - \theta_{300} \quad 1.0$$

$$YP = \theta_{300} - PV \quad 2.0$$

$$AV = \frac{1}{2} \theta_{600} \quad 3.0$$

Measurement of Filtration Properties:

Filtration test was performed using a High Pressure High Temperature (HPHT) cell at conditions of 500psi differential pressure and temperature of 250°C.

Electrical Stability Measurement

The stability of the emulsion is one of the most crucial properties of the oil-based drilling mud; hence it becomes of utmost importance to investigate the stability of the oil-based mud formulated.

Retort Test for Oil, Water and Solid Concentration

A mud retort kit heating equipment is used to determine the quantity of liquids and solids in a drilling mud.

RESULTS AND DISCUSSION

The results for the characterization and mud test for the SBF and the SBM are as presented

Characterization of the Base Oil and Ester

FTIR:

The spectra for the ester were is shown in the Appendix A display the characteristics of some functional group common to the alkyl ester samples. The absorption band at 1743.56 cm^{-1} indicates the presence of C=O of an ester.

Compositional Analysis of TNO and Its Ester:

The base fluid was characterized and the viscosity, specific gravity, pour point, cloud point, flash point and fire point were determined. The physical properties of the raw tiger nut oil and its ester is presented in the Table 2.0. it was observed that the kinematic viscosity of the TNO was high (25.7cst) which isn't for use. Also the result of the cloud point shows the oil stability at low temperature of above -6°C . while that of the ester had a the kinematic viscosity, cloud point and flash point of 3.25cst, -5°C and 160°C , respectively, This is within the API standard of thus it suitable to be used as a base fluid for the formulation of a synthetic-based mud.

It is observed that the specific gravity of the ester at 30°C is 0.867 while that of the raw oil is 0.97. The value of the ester compared favorably to the API 0.76 to 0.88 specification for diesel. The cloud and pour point of both the raw and ester compared favorably with API standard of ambient temperature. The flash point and fire point of the raw oil and the synthetic oil both compare favorably with API standard of flash point and fire point of $>66^{\circ}\text{C}$ and $>80^{\circ}\text{C}$.

Table 2.0: Physical properties of Tiger Nut Oil (TNO) and TNO Ester

SAMPLE	TNO	TNO ESTER
Physical form	Liquid	Liquid
Solubility in water	Insoluble	Insoluble
Cloud point	-6°C	-5°C
Pour point	-25°C	-15°C
Flash point	173.5°C	160°C
Fire point	175°C	162°C
Density	0.98g/ml	0.98g/MI
Kinematic viscosity	25.7cst (40°C)	3.25cst (40°C)
Specific gravity	0.967 (25°C)	0.867 (25°C)

Mud Sample Properties Results

Density:

The density of the mud samples are presented in the Table 3.0

Table 3.0: Physical properties of Tiger Nut Oil (TNO) and TNO Ester

OWR	Samples							
	70:30 (SBM)		75:25 (SBM)		80:20 (SBM)		75:25 (Diesel OBM)	
	Premix	Weighted Mud	Premix	Weighted Mud	Premix	Weighted Mud	Premix	Weighted Mud
Mud weight(ppg)	7.1	10	7.2	10	7.1	10	7.2	10

Viscosity and Gel Strength:

The viscosity reading at different dial readings of the viscometer at varying temperatures and time were recorded. The plot of the dial reading against viscometer speed are shows a trend of the (Figures Bingham plastic model. The figure 4.1 shows that the mud samples had low plastic viscosity, this indicates its suitability for greater energy at the drill bit, increased rate of penetration and low fuel consumption in the circulating systems. The Figures 4.1 to 4.4 indicate a high yield point at all temperatures, this implies that the fluid exhibit a good carrying capacity in the annulus.

The high gel strength indicates that the ability of the mud to suspend cutting is good as it compared favorably with that of diesel oil OBM.

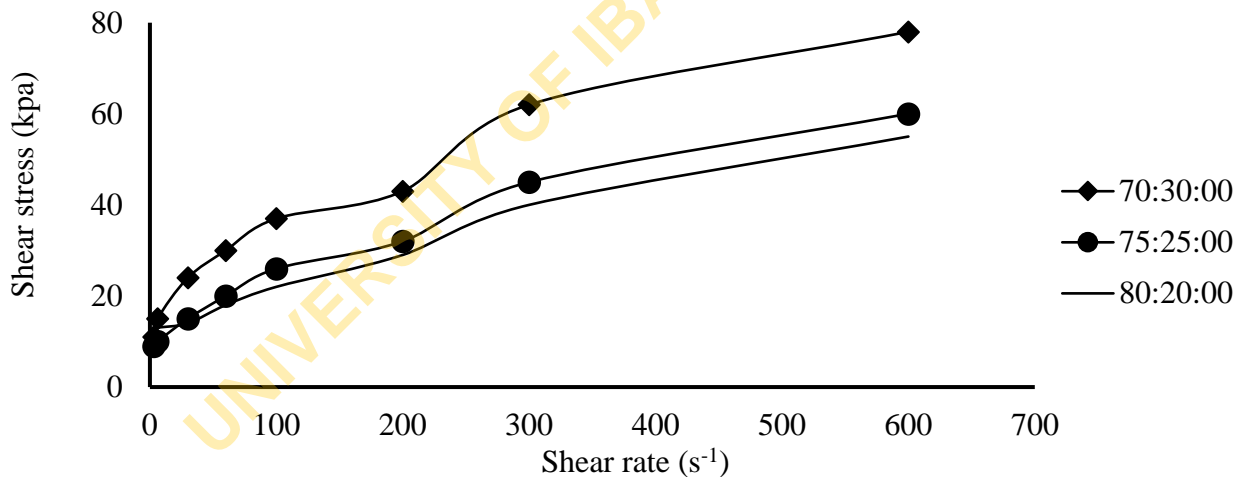


Figure 1.0: Shear stress against Shear rate at 86°F

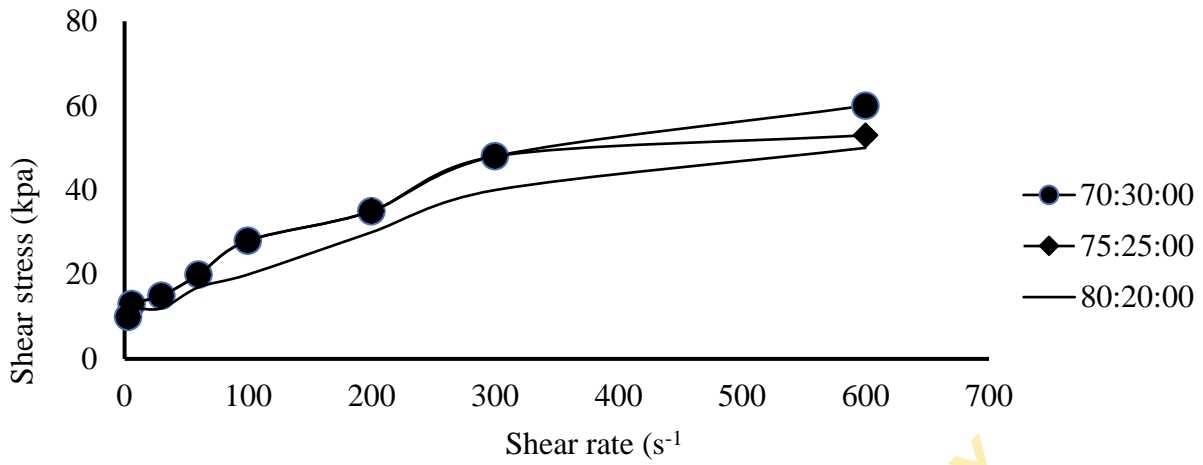


Figure2.0: Shear stress against Shear rate at 86°F and 24 hour

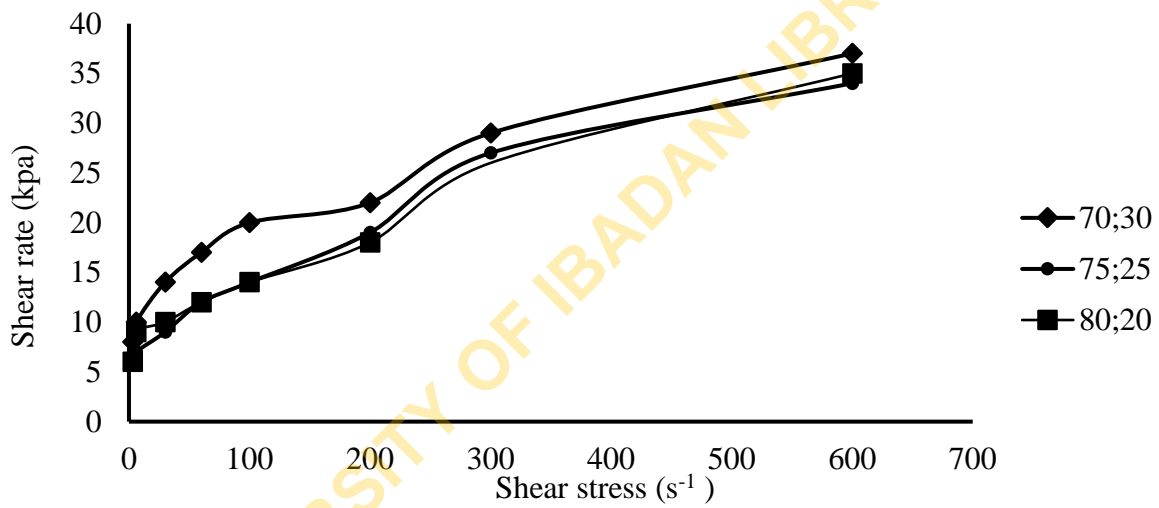


Figure 3.0: Shear stress against Shear rate at 150°F and 24 hours

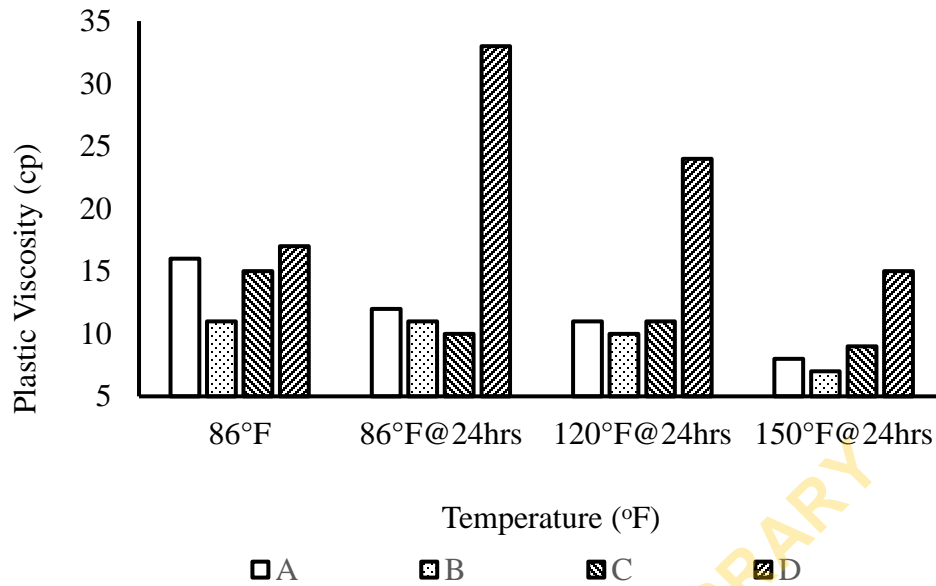


Figure 4.0: Plastic viscosity at varying temperatures

High Temperature High Pressure Filtration Results:

The filtration results of the mud samples are presented in Table 5.0 and 6.0. The result shows that the diesel mud has the highest filtration loss and filter cake thickness, indicating that its mud it would cause more formation damage. The filtrate loss and filter cake thickness of the synthetic based mud compared favorably with API standard of low filtrate loss and thin, slippery filter cake.

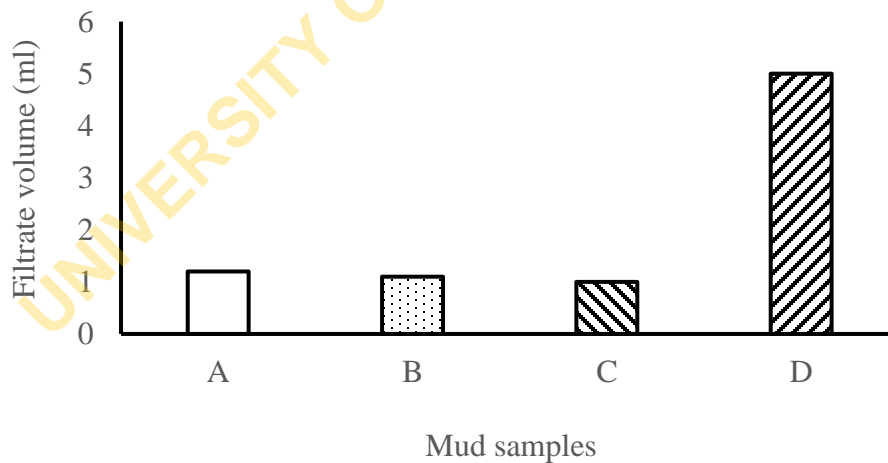


Figure 5.0: Fluid loss volume of the different mud samples

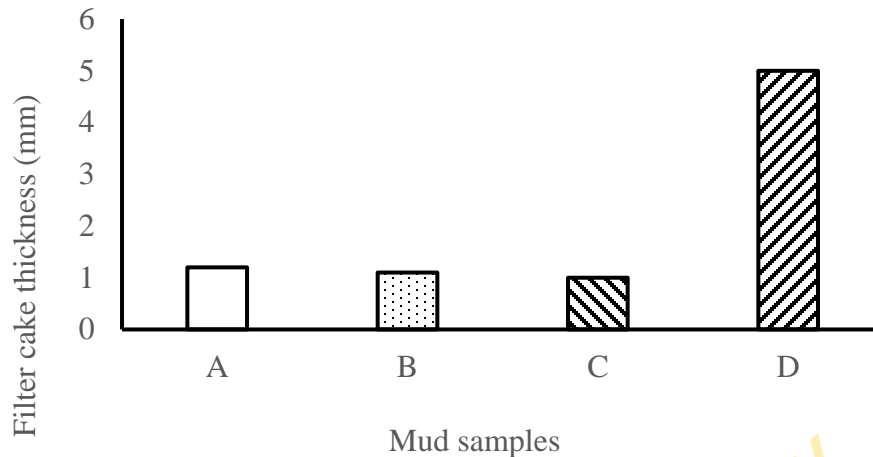


Figure 6.0: Mud Cake thickness of the different mud samples

Electrical Stability:

The electrical stability values represent the mud water-in-oil stability. The results of the electrical stability of the mud samples as shown in Table 4.0 indicate that the mud samples are stable as they all had electrical stability greater than 400 volts

Table 4.0: The Electrical stability of the Various Mud Samples

OWR	Samples							
	70:30 (SBM)		75:25 (SBM)		80:20 (SBM)		75:25 (Diesel OBM)	
	Premix	Weighted Mud	Premix	Weighted Mud	Premix	Weighted Mud	Premix	Weighted Mud
ES (volt)	400	561	418	463.67				

Retort:

The value of the retort analysis is presented in the Table 5.0 from the result, it is observed that

Table 5.0: The Retort Values of the Various Mud Samples

OWR	Samples			
	70:30 (SBM)	75:25 (SBM)	80:20 (SBM)	75:25 (Diesel OBM)
Filtrate Nature	Oil	Oil		
Retort (wt.%)				
Oil	4.6	5.3	4.2	5.3
Water	1.8	1.5	1.2	1.5
Solid	3.6	3.2	4.6	3.2

CONCLUSION

Transesterification reduced the viscosity of the Tiger Nut Oil. The methyl ester product of the transesterification of Tiger Nut Oil confirmed by the FTIR test reveals that the flow properties compare favorably with diesel. However, some of the compounds identified by the test are used in making hair cream and body lotion. Therefore, the ester is less toxic to plants and most importantly humans. The FTIR spectra also reveals that the ester is free from aromatic compounds. The high flash point of the ester equally compared favorably with diesel oil, hence, no regulatory issues pertaining to its transportation and storage.

The laboratory scale mud test reveals the ester formulated SBMs have greater chances of being a technically viable replacement of diesel oil OBMs. The HTHP filtration test shows that using the mud will not lead to hydrous disintegration of shale as the filtrate is small and it is only oil. The thin and slippery filter cake formed indicates less hole problems, stuck pipe problems etc. The result of the density of the premix: 7.1, 7.3 and 7.2 were lower than that of diesel OBM, this indicate that more barite is needed to achieve a set density of 10ppg. The result of the flow properties shows that the sample with 80:20 oil-water ratio has better rheology that the other sample having higher percentage of water.

RECOMMENDATION

There is a future in the use of this ester as a substitute for imported base oils as it would improve local content development, however more research has to be done on the most cost effective way of producing the oil and the effect of various additives on the rheology and filtration properties of the mud,

References

- Alkabbashi A. N, Mohamed E. S (2009). Biodiesel Production from Crude Palm Oil by trans-esterification process. *Journal of Applied Science*.
- Alan R., Dutton E. (2003). History, Regulation and Closure of Abandoned Centralized and Commercial Drilling Fluid Disposal Sites in Louisiana, New Mexico, Oklahoma and Texas.
- Aluyor E. O, Obahiagbon K. O, Ori-Jesu M. (2009) Biodegradation of vegetable oils: A review, *Scientific Research and Essays*.
- Airende O. E (2011) Predicting the downhole behaviour of environmentally friendly oilbased drilling fluid at varying temperatures using artificial neural network. B.Sc. Project.
- Bourgoyne A. T, Chenevert M. E, Millhien K. K, Young F. S. (2003) *Applied Drilling Engineering*, vol. 2 of SPE Textbook Series, Society of Petroleum Engineers (SPE), Richardson, Tex, USA.
- Adesina F., Adeyemi A., Ameloko A., Falode O. (2011) Modelling the effect of temperature on environmentally safe oil based drilling mud using artificial neural network algorithm. *Petroleum and Coal*.
- Akintola S., Orij O., Momodu M. I
- Dina k., Robiah Y., Rozita O., Suraya A.R., Badrul M.J., (2021) Rheological Investigation of Synthetic-based drilling fluid containing non-ionic surfactant pentaerythritol ester using full factorial design, Elsevier.
- Destiny A. J., Amanze F. C. (2021) Performance of rubber seed oil as an alternative to diesel in oil-based drilling mud formulation, *International Journal Applied Science*.

Dosunmu A., Ogunrinde J. (2010) Development of environmentally friendly oil based mud using palm oil and groundnut oil. Society of Petroleum Engineers (SPE 140720).

Erhan S. Z, Perez J. M. (2002) Bio-based Industrial Fluids and Lubricants, The American Oil Chemists Society.

Fadairo A., Ameloko A., Adeyemi G., Ogidigbo E., Airende O. (2012) Environmental impact evaluation of a safe drilling Mud. Society of Petroleum Engineers (SPE).

Fadairo A., Falode O., Ako C., Adeyemi A., Ameloko A. (2012) Novel formulation of environmentally friendly oil” New Technologies in Oil and Gas Industry (Chapter 3). InTech Publisher.

Fadairo A., Orodu O. D, Falode O. (2013) Investigating the carrying capacity and the effect of drilling cutting on rheological properties of jatropha oil based Mud,” Refereed Proceeding, Society of Petroleum Engineers, (SPE Paper 167551).

Combs G. D, Whitemire L. D. (1968). Capillary Viscometer simulates bottom-hole conditions, Oil and Gas Journal.

Himanshu k, Amit S., Shivanjali S. (2020) Novel Jatropha oil based emulsion drilling mud out performs conventional drilling mud: A comparative study, Energy Sources.

Okorie E., Anieta N., Francis D. (2015) A comparative study of diesel oil and soybean oil-based drilling mud, Journal of Petroleum Engineering.

Orisamika B. O. (2015) Development and Evaluation of Synthetic Base Drilling Fluids from Palm Kernel Oil and Coconut Oil Ester. MSc Thesis, (Unpublished).

Oseh J. A., Norddin M. N., Ismail I. (2019) Performance evaluation of a benign oil-based mud from non-edible sweet almond seed, Society of Petroleum Engineers (SPE-198717-MS)

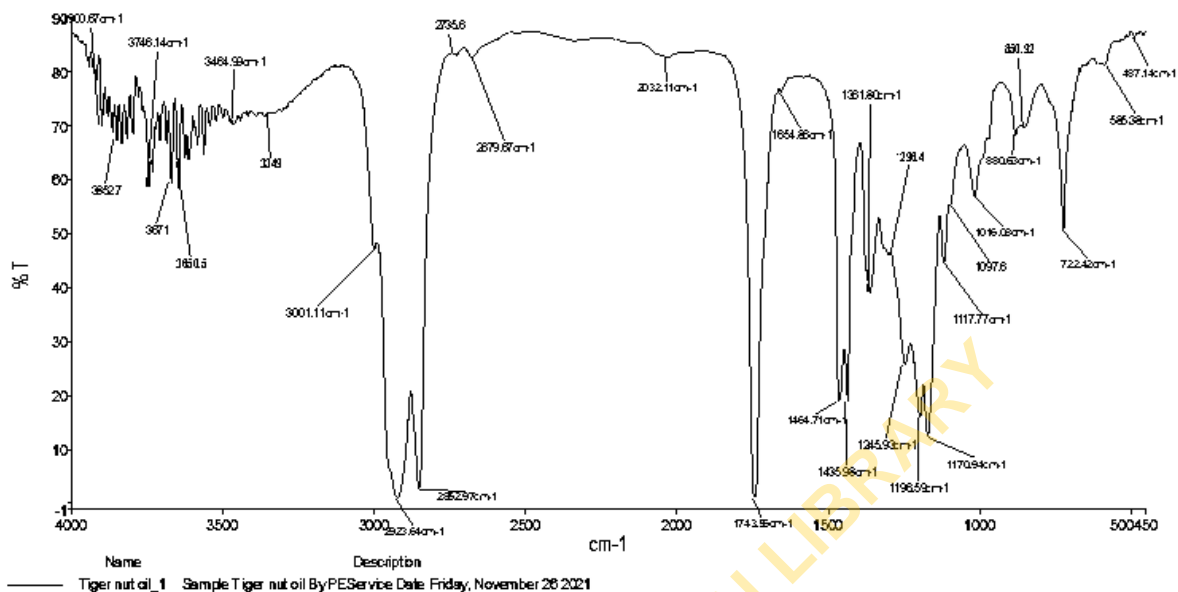
Politte M. D. (1985) Invert oil mud rheology as a function of temperature and pressure. Society of Petroleum Engineers (SPE 13458).

Potman G. J, Neder L. A, Mij B. V. (1990) Responsible Approach to the use of Oil Based Muds. Society of Petroleum Engineers (SPE).

Saket k., Aarti T., Nitesh K., Maen M. (2019) A novel oil-in-water drilling mud formulated with extracts from Indian mango seed oil, Petroleum Science.

Yassin A., Kamir A., Mohamad O. A. (1991) Formulation of an environmentally safe oil based drilling fluid, Society of Petroleum Engineers (SPE23001).

Xiaoging H., Lihui Z. (2009) Research in application of environment acceptable natural macro-molecular based drilling fluids, Society of Petroleum Engineers (SPE).



Search Score	Search Reference Spectrum Description
0.938375	METHYL LINOLEATE NATURAL
0.930639	METHYL ELAIDATE GC REFERENCE
0.873791	DIMETHYL AZELATE 90-95%
0.871694	BUTYL STEARATE
0.86792	ETHYL PALMITATE
0.854797	ETHYL MYRISTATE
0.824568	ETHYL LINOLEATE
0.797049	BIS(2-ETHYLHEXYL) SEBACATE
0.789134	METHYL N-VALERATE
0.689189	1-BROMOHEPTADECANE