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Experimental Evaluation of Locally Synthesized Biodiesel Drilling Fluid

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Abstract

Diesel oil has been the preferred base fluid for the formulation of oil-based drilling mud. Diesel oil has negative effects on the environment and there is a growing need for more environmentally sustainable alternatives that can be technically compared to diesel base oil. In recent times, the use of vegetable oils as drilling fluid base oil has been of interest. In this study, 1378 kg/m^3 of palm kernel oil-based mud (PKOBM) and palm kernel oil biodiesel based mud (BDBM) were experimentally formulated. BDBM was synthesized by the transesterification of vegetable (palm kernel) oil. The performance of PKOBM and BDBM was then evaluated against conventional diesel oil-based mud (DOBM). The evaluation performed was based on the rheological, filtration and wall building properties, emulsion and thermal stabilities, and acute toxicity of the formulated drilling fluid systems. The results obtained from the study reveal that the mud systems (PKOBM and BDBM) show a typical Herschel-Bulkley (modified power-law) drilling mud rheological pattern at temperatures of 49°C, 66°C and 80°C. BDBM showed comparable rheological properties with better hole cleaning capacity as indicated by a lower flow index. For the filtrate loss test, BDBM exhibited a slightly lower filtrate loss compared to DOBM, whereas PKOBM had a higher fluid loss of 4.4 ml. For the filter cake and thermal stability test, there were no significant changes between DOBM and BDBM, whereas PKOBM had the least desirable performance. BDBM exhibited the most stable emulsion of 1274 volts breaking voltage and PKOBM, the least with 739 volts compared to 1169 volts breaking voltage of DOBM. This study concludes that BDBM could be used as an environmentally sustainable substitute for diesel oil-based mud (DOBM).

Keywords:
biodiesel, rheological properties, filtrate loss, emulsion stability, thermal stability, acute toxicity

1. Introduction

The dwindling price of crude oil and its consequential effect on the economics of oil well drilling has necessitated the quest for more economical but technical and feasible local sources of material inputs. Oil well drilling operations involve the use of a complex mixture called drilling fluid or mud (**Jha, et al., 2014**). The success of oil well drilling, completion, as well as productivity is a function of the selection and performance of the drilling fluid. Drilling fluids are classified by the base fluid that forms the continuous phase to which all other components of the drilling fluid are added; Water Based Mud (WBM), Oil Based Mud (OBM) and Pneumatic fluids (gas or gas-liquid mixture) (**Khodja et al., 2017**). The base fluid used in formulating the drilling fluid contributes greatly to its technical, environmental, and economic performance. WBM can further be classified according to the type of viscosifying material and other performance-enhancing agents used in its formulation, while OBM can also be subdivided based on whether the base fluid is crude, a refined petroleum product, or other sources.

A lot of environmental concerns in recent time have attracted stringent sanctions imposed by various regulatory bodies in regards to drilling fluids, mostly OBM. Some benefits are associated with OBM (better and desirable rheological properties, little or no formation damage and better well productivity), **Bol et al.** (**1992**) observed that among these, it has greater shale stability than WBM, but **Dardir et al.** (**2014**) considered aromatic compounds contained in OBM as toxic and harmful to the environment. **Growcock and Petal** (**2011**) reported that the initial cost of synthetic base oil mud (SBM) can be double that of OBM, which confirmed the earlier work of **Tapavicza and Hall** (**2002**) that the choice of OBM was based on lower cost and availability. This environmental concern has poised the industry to search for more environmentally friendly alternatives to conventional OBM. Many researchers in response to environmental challenges posed by OBM, developed mud systems with better performance with respect to envi-

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ronmental constraints (**Simpson et al., 1994; Friedheim et al., 1999; Young and Maas, 2001; Patel et al., 2001; Schlemmer et al., 2002**). Synthetic-based muds (SBMs) were developed by the oil and gas industry. SBM combines desirable operating qualities, lowers the toxicity and negative environmental impact qualities of OBM. SBM also has high biodegradability making it a better choice than OBM. SBM can be classified into synthetic hydrocarbon, ethers, esters and acetals, but synthetic ester-based mud is most frequently selected based on its rheological properties, thermal stability and filtration loss (**EPA, 1999 and Hamed and Belhadri, 2009**). **Amorin et al.** (**2015a**) observed the average cost saving in percentage for plant oils as base fluids over commercial synthetic-based fluids of above 45%. Though OBM was claimed to be cheaper and more readily available (**Growcock and Patel, 2011**), but more expensive than SBM if the cost of treatment, transportation and disposal are taken into consideration (**Onuh et al., 2019**). More savings were attributed to the use of SBM as drilling time was reduced by 35-55% according to the work of **Amorin et al.** (**2015b**). With high-quality palm kernel oil and its availability in vegetable oil production, the need for biodiesel has been confirmed (**Faessler, 2004**).

This limitation necessitated the search for petroleum-based mineral oils and low toxicity mineral oils (LTMO) with 3 – 5% aromatic content, though there are restrictions on the discharge of LTMO. This makes it imperative to formulate OBMs using base oils that are environmentally friendly, biodegradable, and also economical. At the same time, such fluids must perform the basic technical functions of a drilling fluid (**Md.Yassin, et al., 1991**), which can be broadly classified as (**IADC Drilling Manual, 2014**):

- Control of subsurface pressures: Pressure control is achieved by balancing the formation pressure with the hydrostatic pressure of the drilling fluid, plus any surface-imposed pressure on the annulus. Additional pressure losses are usually imposed on the wellbore while circulating mud during the drilling operation.
- Removal of drill cuttings from the well: The capacity of the drilling fluid to perform this function depends on the rotary speed of the drilling string and the velocity, viscosity, density of the fluid.
- Uphold wellbore stability: Factors affecting the capability of drilling fluid to perform this function include rock strength, local subsurface stresses, wellbore pressure differential and drilling fluid chemistry.

Previous studies on vegetable oils as the base fluid for the formulation of invert oil drilling mud focused on the rheological and environmental toxicity properties (**Fadairo et al., 2012**). Other technical issues like thermal stability, emulsion stability and filtration at elevated temperatures are also of great concern. The most considered vegetable oils are rapeseed oil, jatropha oil, mahua oil, cottonseed oil, sesame oil, soybean oil and palm oil (**Okullo et al., 2012; Fadairo et al., 2012**). The performance of these oils considered for drilling fluid systems was favourable in terms of their lower environmental impact. However, their technical performances were not as good as that of diesel oil, hence further beneficiation to bring their physicochemical properties close to the America Petroleum Institute (API) recommended standard for oil-based drilling fluid (**Okullo et al., 2012**). Studies on the physicochemical properties of some of these vegetable oil-based fluids used in formulating drilling fluids have been investigated by researchers. In a study conducted by **Bello et al.** (**2015**), the chromatographic analysis on palm kernel oil fatty acid showed 46% lauric acid and 23% palmitic acids, and the oil has 97.13% biodiesel with a total saturation of 78%. Palmitic and stearic acids are two of the most found saturated fatty acids in vegetable oils while oleic and linoleic are the most found unsaturated fatty acids. Many of the oils also contained some linolenic acid (**Demirbas, 2010**). Physicochemical properties of palm kernel, coconut and jatropha oils were extracted and presented in **Table 1** (**Amira et al., 2014; Musa et al., 2016**). This study attempts to examine the technical properties of vegetable oil-based drilling mud (PKOBM and BDBM) and compare their performance to that of Diesel oil-based mud (DOBM).

Table 1: Physicochemical Properties of Palm Kernel Oil, Coconut Oil and Jatropha Oil

Physicochemical properties	Palm Kernel Oil	Coconut oil	Jatropha oil
Saponification value (mgKOH/g)	280.5 ± 56.1	191.89	188 – 198
Acid value (mgKOH/g)	2.7 ± 0.3	14.025	14.025
Free fatty acid (mgKOH/g)	1.35 ± 0.15	28.025	28.025
Peroxide value (mgEq/kg)	14.3 ± 0.8	8	-
Iodine value (mgKOH/g)	15.86 ± 4.02	121.1	90.8 – 112.5
Specific gravity (S.G)	0.904	0.912	0.9186

2. Methodology

To attain the objectives of this study, three mud types; PKOBM, BDBM and DOBM were synthesized under laboratory conditions. A summary of the processes and materials implemented in this study is presented in a pictorial flowchart shown in **Figure 1**. After each test, the results from the three mud types were presented and compared.

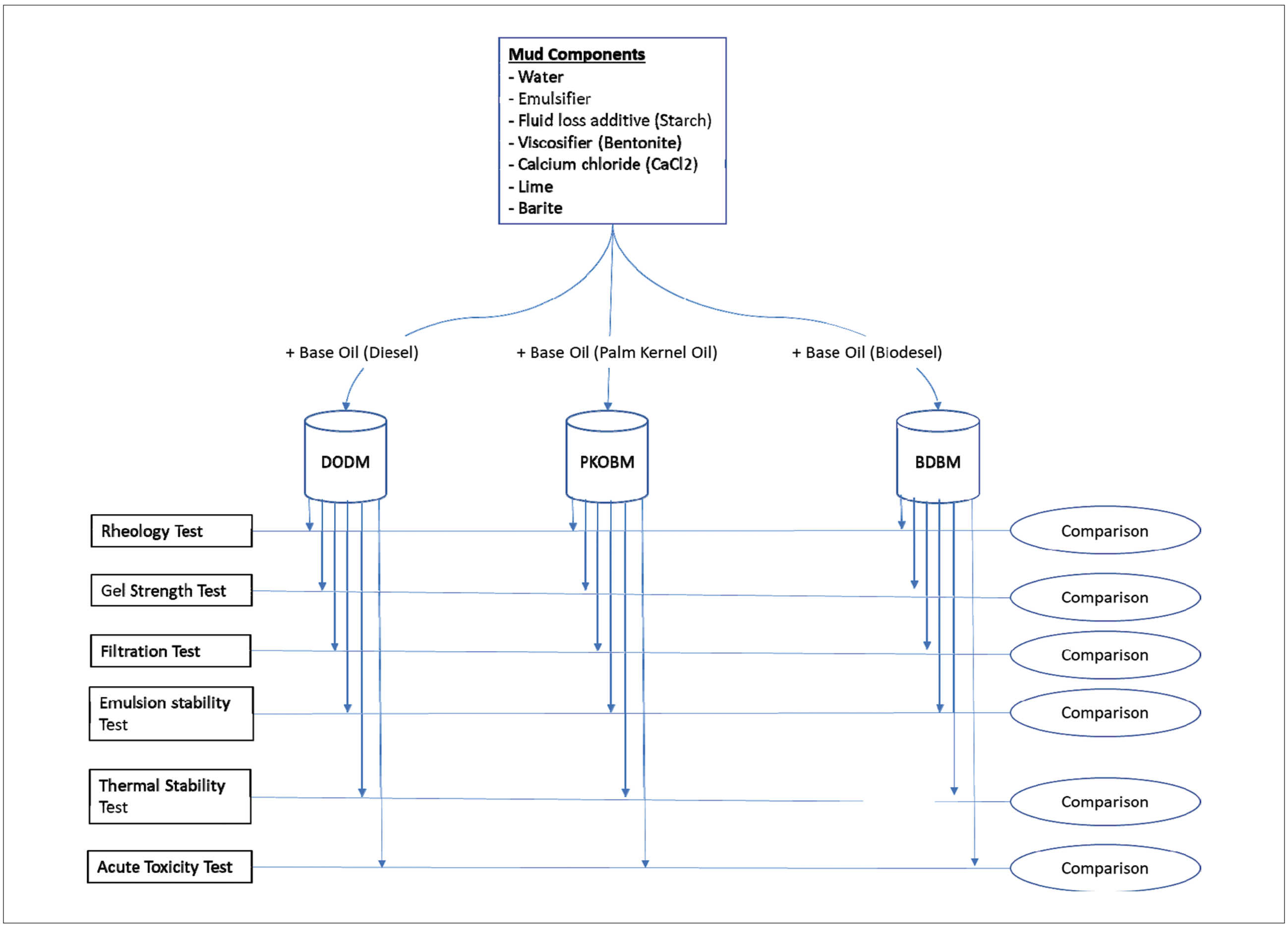


Figure 1: Flowchart of Methodology

2.1. *Equipment and Materials*

The materials and equipment used to prepare the mud samples are described in this section.

2.1.1 Materials

The materials include: water, emulsifier, fluid loss additive (starch), viscosifier (bentonite), calcium chloride ($CaCl_2$), lime, barite, catfish (D. Magna) Fingerlings, phosphoric acid, sodium hydroxide (NaOH), methanol (CH_3OH) and the base oil (diesel, palm oil, biodiesel).

2.1.2 Equipment

The choice of equipment depended on the types of experiments, accuracy (and/or precision) required and availability. The following equipment was used for this study; pH meter, mechanical grinding machine, sieve (80 – 200μm), mixing cup, beakers, test tubes, electric weighing balance, OFITE viscometer, Bariod Mud Balance and Eutech pH Tester-10, measuring cylinder, spatulas and magnetic stirrers, crucible, desiccator, electric mixer, OFITE Electrical Stability (ES) Meter, OFITE HTHP filter press with single-end test cell and OFITE Shearometer.

2.2 *Experiment Procedure*

2.2.1 Synthesis of Biodiesel from Palm Kernel Oil

The transesterification process of **Musa et al.** (**2016**) was adapted for use in the production of biodiesel from palm kernel. The palm kernel oil was first mixed with 3% of hot water and 0.05% of 75% phosphoric acid, and then stirred for 30 minutes at 70°C using a magnetic stirrer. The mixture was allowed to rest for 45 minutes for the gum (phosphatide) and other impurities to settle and be drained off. The degummed oil was dried and used for biodiesel production. A mixture of 0.5 g of sodium hydroxide (NaOH) and 30 ml of methanol (CH_3OH) was stirred in a beaker until all the NaOH dissolved to form a sodium methoxide ($NaOCH_3$) solution. 100 ml of degummed palm kernel oil was poured into a beaker and heated gently at 65°C. Methoxide was added and the resulting mixture was stirred vigorously for 1 hour to obtain a homogeneous mixture and this mixture was poured into a separating funnel to settle for 24 hours. The mixture separated into two layers with the biodiesel in the upper portion and glycerine at the bottom was drained off. The raw biodiesel was washed by adding water and stirred to remove any traces of soap, glycerine and other contaminants. The biodiesel–water mixture was allowed to settle, and the water was drained off. The washed biodiesel was collected in a beaker and dried in an oven at 105°C to evaporate the excess water and methoxide in the biodiesel. The produced biodiesel was stored in a plastic container for drilling mud formulation.

2.2.2 Formulation of the Oil Based Drilling Muds

One-lab barrel (350 ml) of diesel oil, palm kernel oil and palm kernel oil biodiesel-based drilling fluid samples were formulated using formulation specifications as shown in **Table 2**.

Table 2: Formulation Specifications for Oil Based Drilling Fluid Mud Specifications: 1378 kg/m³; 70/30

Sample Composition		Diesel	Palm Kernel Oil	PKO Biodiesel
1	Water (ml)	87.00	93.10	87.60
2	Base oil (Diesel) (ml)	208.80	210.20	210.20
3	Emulsifier (ml)	8.70	8.70	8.70
4	Fluid loss additive (Starch) (g)	4.00	4.00	4.00
5	Viscosifier (Bentonite) (g)	6.00	6.00	6.00
6	Calcium chloride ($CaCl_2$) 97% (g)	24.40	25.10	25.0
7	Lime (g)	4.00	4.00	4.00
8	Barite (g)	194.4.	178.60	186.30

The amount of oil, water, weighting material and $CaCl_2$ needed to formulate an invert emulsion fluid was calculated using the equation specified in the ***Bariod Manual of Drilling Fluids Technology*** on material requirement determination for a given volume. The mixing order used during the formulation of the three drilling mud systems is presented in **Table 3**.

Table 3: Mixing Specification for Drilling Mud Formulation

S/N	Mud Constituent	Mixing Duration (min)	Mixing Order
1	Oil	-	1
2	Primary Emulsifier	10	2
3	Lime	5	3
4	Fluid loss additive	5	4
5	Water	15	5
6	Bentonite	5	6
7	Barite	10	7
8	Powdered $CaCl_2$	5	8

Calcium chloride ($CaCl_2$) flake or pellet was dissolved in water before adding to the mud. The lowest recommended oil/water ratio for each mud density is given in **Table 4** below.

Table 4: Lowest Recommended Oil/Water Ratio for Mud Density (Baroid Manual of Drilling Fluids Technology, 1990)

Mud Density, kg/m³	Lowest Recommended Oil/Water Ratio
840 – 1080	60/40
1080 – 1320	65/35
1320 – 1680	70/30
1680 – 1920	75/25
1920 – 2280	80/20
2280+	85/15 – 90/10

The prepared mud was kept for 24 hours to age and its properties such as density, pH, rheological properties, filtration, emulsion and thermal stability, lubricity and toxicity were determined.

2.2.3 Measurements of Mud Rheological Properties

The American Petroleum Institute (API) Test standards for oil-based drilling fluid properties determination was used. A Bariod Mud Balance and Eutech pH Tester-10 were used to determine the mud weight and mud hydrogen ion potential (pH) respectively. The pH meter electrode was dipped 2 – 3 cm into the mud samples after being calibrated with pH standard buffer solutions of pH 4.01, pH 7.00 and pH 10.01. An OFITE eight-speed viscometer (model 800) was used to determine the rheological properties according to API recommended practice for field testing oil-based drilling fluids. The mud sample was homogenized at 18,000 rpm with a mixer and poured into a pre-heated Thermo cup (sample holder). The rotor sleeve was immersed into the mud up to the scribed line and the mud stirred to reach a steady test temperature of 49°C with the speed selector knob in the stir position. The speed selector knob was then rotated to 600, 300, 200, 100, 60, 30, 6 and 3 rpm settings. The speed selector was moved in the reversed direction, 3 through 600 rpm dial readings of the respective speed recorded when the dial became steady. The procedure was repeated at 66°C and 80°C test temperatures respectively. The average of the two readings was calculated. The apparent viscosity (AV), plastic viscosity (PV) and yield point (YP) were determined using **Equations 1, 2** and **3.**

$$\text{Apparent viscosity } (AV) = \frac{\theta_{600}}{2}, \text{ cP} \tag{1}$$

$$\text{Plastic viscosity } (PV) = \theta_{600} - \theta_{300}, \text{ cP} \tag{2}$$

$$\text{Yield point } YP = (\theta_{300} - PV), \text{ kg/m}^2 \tag{3}$$

Where:

θ_{600} = Dial reading at 600 rpm;

θ_{300} = Dial reading at 300 rpm.

2.2.4 Gel Strength of the Formulated Drilling Fluids

The OFITE 8-speed viscometer was used to determine the 10-second and 10-minute gel strengths of the mud systems. The mud sample was poured into a preheated Thermo cup and mounted to position. The rotor sleeve was immersed into the mud sample to the prescribed line and stirred for 15 seconds with the speed selector knob at the stir setting. The knob was then rotated to gel (3 rpm) setting and the viscometer was immediately switched off. The mud was allowed to stand for 10 seconds after the sleeve stopped rotating. The viscometer was switched on at the elapse of 10 seconds and the maximum dial deflection before the gel break was recorded in kg/m^2 as 10-second gel strength. The same procedure was repeated for 10-minute gel strength by stirring and allowing the mud to stand for 10 minutes.

10-second gel strength, kg/m^2 =
= maximum dial deflection after 10 sec.

10-minute gel strength, kg/m^2 =
= maximum dial deflection after 10 min.

2.2.5 Mud Filtration and Wall Building Properties of the Formulated Drilling Fluids

Complete OFITE HTHP filter press with single-end test cell and CO_2 pressuring assemblies was used in determining the filtrate loss and mud cake thickness of mud samples. The thermostat control knob was set at 126°C, which is 6°C above the test temperature of 120°C. The heating jacket was preheated until the pilot light came on. The mud samples were stirred for 10 minutes with a high-speed mixer and carefully poured into the cell, 12.7 mm from the O-ring groove. With both valve stems closed, the top and the bottom pressure regulators were adjusted to 690 kPa (recommended back-pressure for the test). The top valve stem was opened by ½ turn to pressurize the mud sample and the pressure was maintained on the mud sample until the test temperature was stabilized as indicated by the thermometer. The pressure on the top pressure unit was increased to 4137 kPa to give a differential pressure of 3450 kPa. The bottom valve stem was opened to initiate filtration and the filtrate was collected for 30 minutes. To correct the total filtrate volume collected to the standard filtration test area of 7.1 in^2 (45.8 cm^2), the filtrate volume collected in 30 minutes was doubled.

2.2.6 Emulsion Stability of the Formulated Drilling Fluid Systems

The OFITE Electrical Stability (ES) meter was used to measure the emulsion breaking voltage. Emulsion Stability indicates the emulsion and oil wetting qualities of oil-based and synthetic-based mud samples. The oil mud sample was preheated to 49°C and poured through a Marsh Funnel screen into a glass beaker. The mud sample was hand-stirred with the electrode probe for 10 seconds to help create a uniform composition and temperature. The ES meter test button was then pressed down automatically applying an increasing voltage (from 0 to 2000 Volts) across an electrode gap in the probe. The maximum voltage, and the mud temperature were recorded.

2.2.7 Thermal Stability of the Formulated Drilling Fluid Systems

An OFITE Shearometer was used to measure the shear strength before and after the mud samples were subjected to a higher temperature. The mud samples were poured into pressurized ageing cells and aged at 120°C for 6 hours in a Labnet HT-Rolling Oven (Model RC 100). The dynamic-ageing process was used to simulate the stress the fluid might be subjected to under drilling conditions. The mud samples were poured into the Shearometer and the aluminium cylinder was gently dropped on the mud surface. The shear strength corresponding to the depth the cylinder sank to in 1 minute was recorded as the 1-minute shear strength in kg/m^2.

2.2.8 Acute Toxicity of the Formulated Drilling Fluid Systems

The 96-hours median lethal concentration (96-h LC_{50}) mortality on catfish (*D. Magna*) fingerling as a test organism was estimated using the Microsoft Excel Forecast Function. The 96-hours median lethal concentration is the concentration that is lethal to 50% of the test organisms after 96 hours of exposure. According to **Baroid's Manual of Drilling Fluid Technology** (**1990**), a substance with an LC_{50} greater than 10000 ppm (part per million) is considered non-toxic. The test organism, catfish (*D. Magna*) are fingerling, 2 – 3 inches long. A concentration of 2000, 5000 and 10000 ppm of both the water-soluble fraction (WSF) of the base oil and the suspended particulate phase of the formulated drilling fluid systems were prepared by mixing respectively 2, 5, and 10 grams each of the base oils and the drilling mud to 1000 milliliter (ml) of the water sample. The water-oil and water-mud mixture were stirred for 5 minutes with a magnetic stirrer and allowed to stand for one hour. At the end of the settling period, the middle portion (i.e. WSF and SPP) was siphoned and stored in a plastic container.

For the toxicity test, each concentration of the WSFs and SPPs was poured into a 100ml plastic test container in duplicate. The test solutions were aerated and maintained at room temperature. Two controls using only the water sample were set up. Ten (10) active and healthy fingerling were introduced into each of the test containers. The test solutions (WSF and SPP) were decanted and a fresh solution of the various concentrations was added to the test containers every 24 hours. The number

Table 5: The physiochemical properties of diesel, palm kernel oil and biodiesel

	Diesel	Crude palm kernel oil	Crude PKO biodiesel	*Standard base oil requirement
Specific gravity → 30°C	0.846	0.904	0.876	Not available
Kinematic viscosity (mm²/s) → 40°C	3.42	5.41	3.39	2.3 – 3.5
Flash point (°C)	76	247	126	> 80
Pour point (°C)	-9	6	13	< ambient temperature
Aniline point (°C)	76	Miscible at ambient temperature of 30°C	Miscible at ambient temperature of 30°C	>= 65
pH	6.96	6.42	7.71	Not available

*Standard base oil requirement

of mortalities was recorded in every 24 hour interval for 96 hours and the dead organisms were removed on discovery. An organism is assumed dead when there is total lack of movement or response after repeated prodding with a probe (**Ogeleka and Tudararo-Aherobo, 2013**).

2.9 Statistical Analysis

The effect of the concentration of the WSF and SPP on the mortality rate of the test organism was statistically demonstrated using Chi-square test (X^2-test) of independence. The null hypothesis that the mortality is independent of the concentration was tested.

H_0: mortality rate is independent of the concentration;

H_A: mortality rate is dependent of the concentration.

$$E_{ij} = \frac{R_i C_i}{N} \qquad (4)$$

Where:

E_{ij} = Expected frequencies;

R_i = Total observed frequency in the i^th^ row;

C_i = Total observed frequency in the j^th^ row;

N = Grand total, $\left(\sum C_i = \sum R_i\right)$.

$$X^2\text{-test} = \sum_{i=1}^{r}\sum_{j=1}^{r} \frac{\left(O_{ij} - E_{ij}\right)^2}{E_{ij}} \qquad (5)$$

Where:

O_{ij} = Observed frequencies.

Decision Rule: Reject H_o if calculated chi-square is greater than tabulated chi-square, that is if $X^2_{cal} > X^2_{tab}$; Accept if otherwise. The hypothesis was tested at the 0.05 level of significance.

3. Results and Discussion

Palm kernel oil and palm kernel biodiesel were tested for suitability in formulating drilling fluid with temperature and pressure range for drilling in the Niger Delta and the results are presented below. The result of physiochemical properties of base oils, properties of mud sample formulated with the three different base oil and its rheological properties were reported. The toxicity of the mud samples with respect to the environment and aquatic life were also reported accordingly.

3.1 Physiochemical Properties of Base Oils

The results of physiochemical characterization, palm kernel oil and palm kernel (PKO) biodiesel used in the formulation of the drilling fluid systems shown in **Table 5** are promising and acceptable base oil properties compared with the standard requirement as reported by **Agwu et al.** (**2015**). The flashpoints of palm kernel oil and PKO biodiesel, 247°C and 126°C respectively are higher than that of regular diesel oil which is 76°C. These high flash point values reduce the possibility of fire hazards due to oil vapour on the rig, most especially in high temperature high pressure (HTHP) environments where a flashpoint of 76°C would be considered detrimental.

3.2 Mud Density and pH of Base Oils

The results of measured and calculated mud densities and pH of all the base oils are shown in **Table 6**. Both the Palm Kernel Oil based mud (PKOBM) and PKO biodiesel based mud (PBDBM) gave the same measured mud weight of 1378 kg/m³ as diesel oil based mud (DOBM). However, the vegetable oil and biodiesel based mud required lesser amounts of barite to maintain a relatively similar density value. This will add to the reduction in the cost of drilling mud.

Table 6: Density of mud samples at 49°C

Mud sample	Diesel	Palm kernel oil	PKO Biodiesel
Measured density, kg/m³	1378	1378	1378
Calculated density, kg/m³	1374	1376	1372
Barite added, g	194.4	178.6	186.3
pH	9.23	8.70	10.02

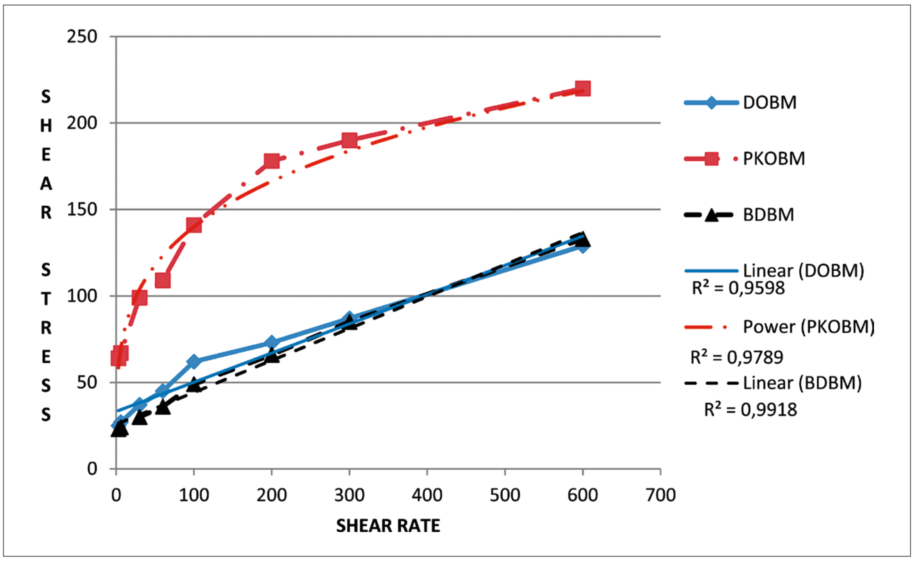
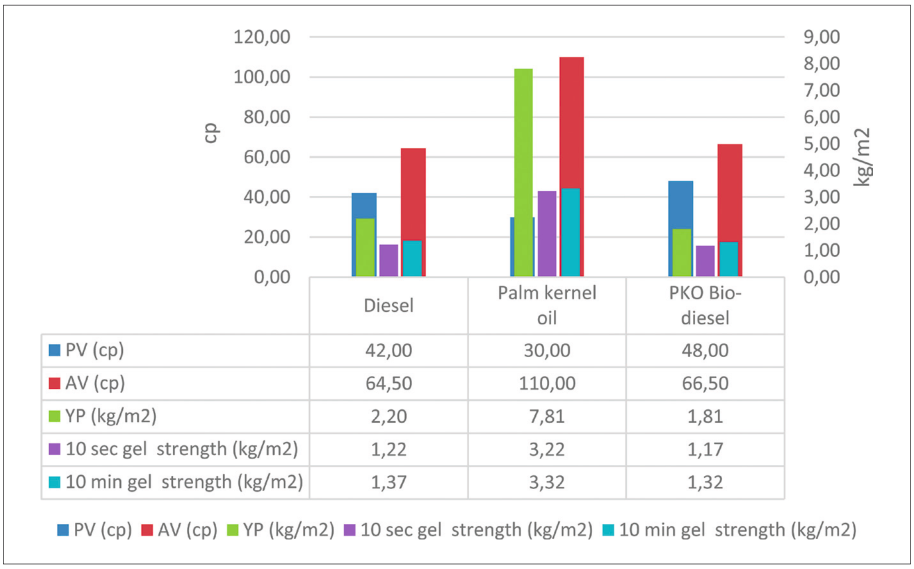


Figure 2: Plot of Shear Stress-Shear Rate of the Mud Systems at 49°C



	Diesel	Palm kernel oil	PKO Bio-diesel
PV (cp)	42,00	30,00	48,00
AV (cp)	64,50	110,00	66,50
YP (kg/m2)	2,20	7,81	1,81
10 sec gel strength (kg/m2)	1,22	3,22	1,17
10 min gel strength (kg/m2)	1,37	3,32	1,32

Figure 3: Plastic Viscosity, Apparent Viscosity and Yield Point of the Mud Systems at 49°C

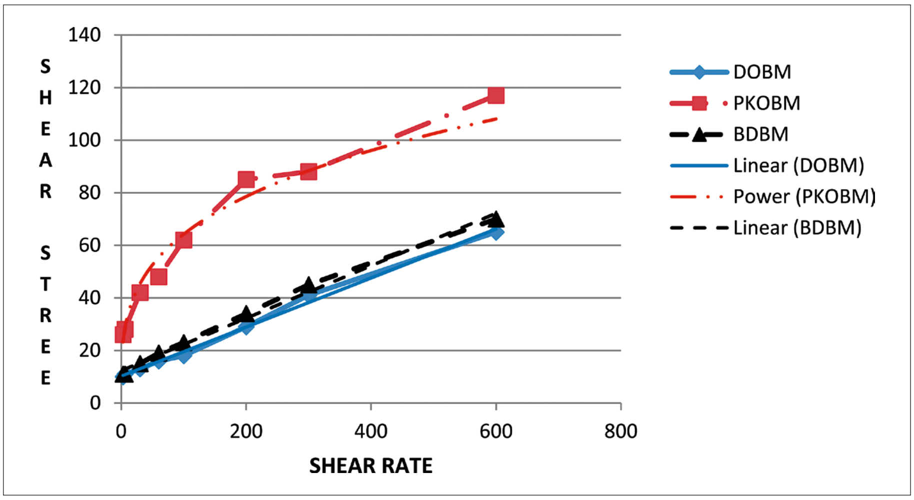


Figure 4: Plot of Shear Stress-Shear Rate of the Mud Systems at 66°C

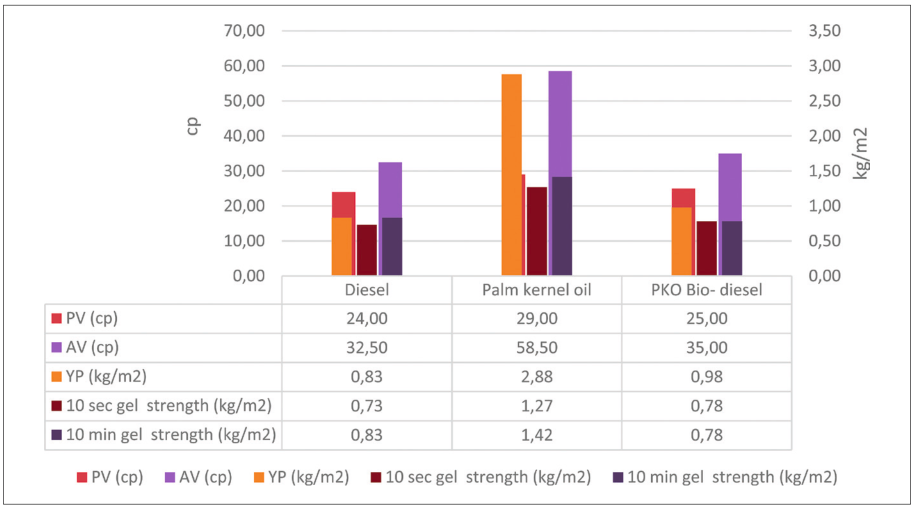


Figure 5: Plastic Viscosity, Apparent Viscosity and Yield Point of the Mud Systems at 66°C

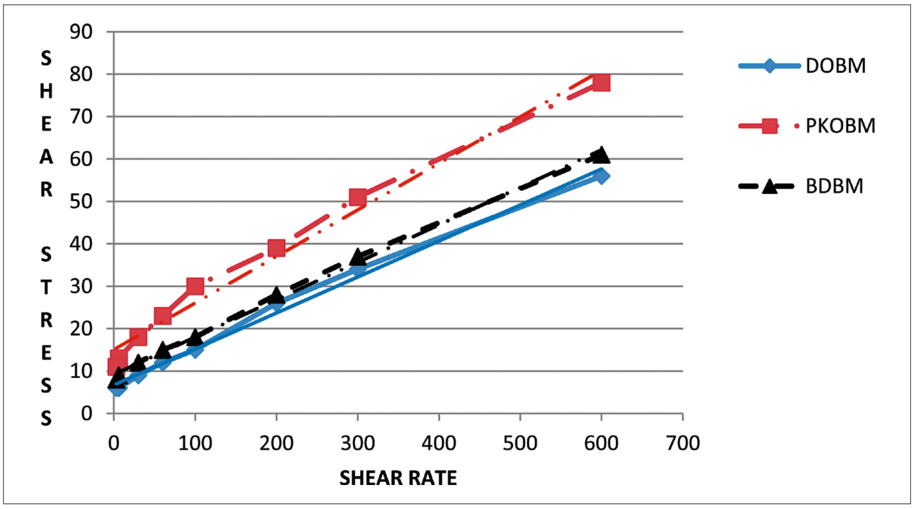


Figure 6: Plot of Shear Stress-Shear Rate of the Mud Systems at 80°C

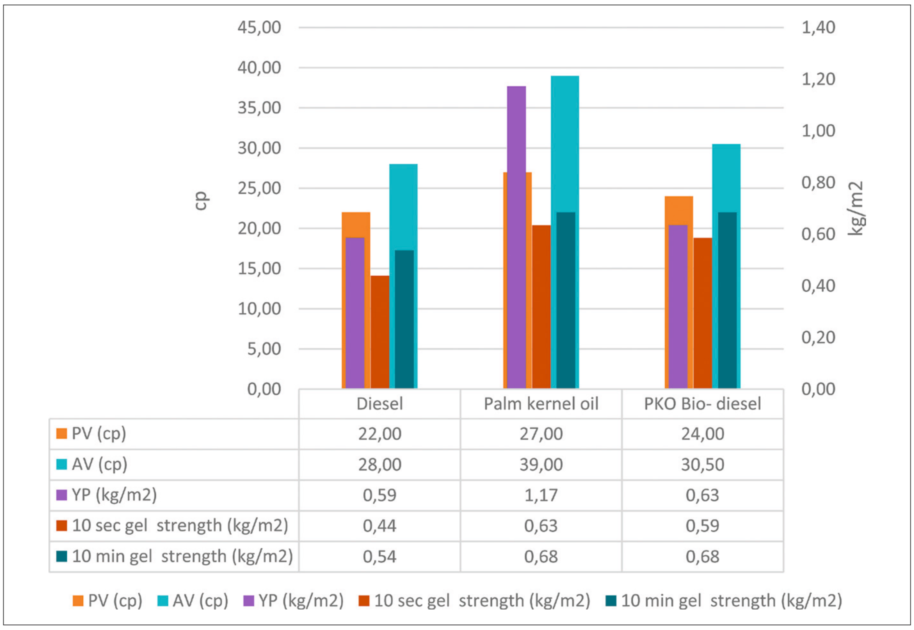


Figure 7: Plastic Viscosity, Apparent Viscosity and Yield Point of the Mud Systems at 80°C

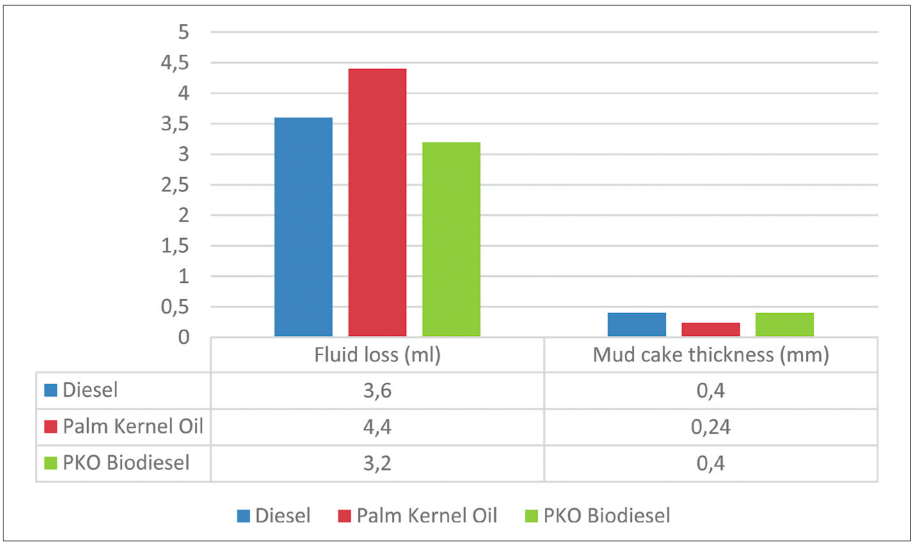


Figure 8: Filtrate Loss and Mud Cake Thickness of the Mud Systems

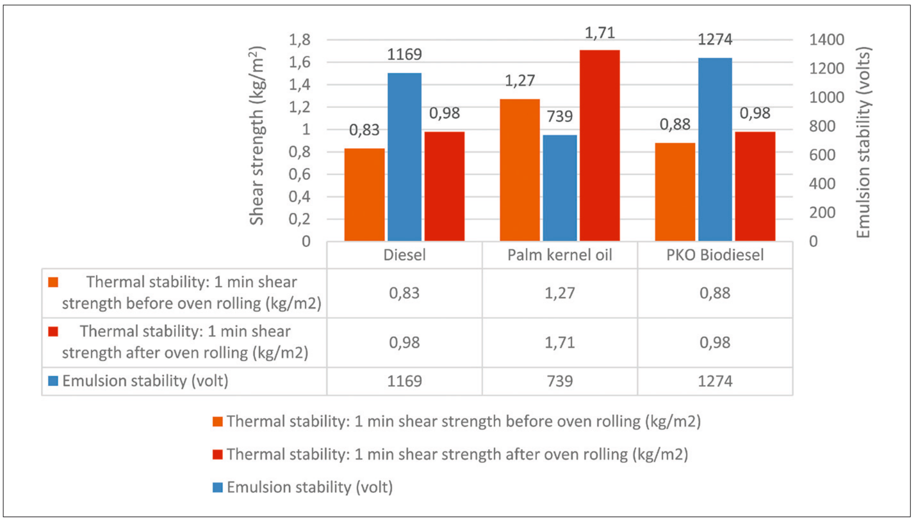


Figure 9: Thermal Stability of the Mud Systems

3.3 Rheological properties

All the mud systems conformed to Herschel-Bulkley flow pattern. At 49°C and 66°C, palm kernel oil-based mud (PKOBM) has a typical Herschel-Bulkley model (see **Figures 2** and **4**) while diesel oil based mud (DOBM) and biodiesel based mud (BDBM) behaviour move towards Bingham Plastic fluids. However, as the temperature increases, at 80°C as shown in **Figure 6**, all the mud systems flow behaviour tends towards Bingham plastic fluid. The values of the yield point of the mud samples were quite high. From the two viscometer readings at 600 rpm and 300 rpm, the fluid rheological parameters of "n" (flow index) and "K" (consistency factor) were obtained. High yield point (YP) implies a better cutting carrying capacity than the lower yield point of mud of the same mud weight. The higher YP implied that the kernel oil based mud has a better cutting carrying capacity than diesel and biodiesel based mud as shown in **Figures 3, 5** and **7**. However, this may be misleading as the mud will require excessive stress to initiate flow which may damage the formation. At higher temperatures of 66°C and 80°C as shown in **Figures 5** and **7**, palm kernel biodiesel based mud showed better performance than diesel oil based mud.

3.4 Mud Filtration and Mud Cake Results

The filtration test results obtained at 3450 kPa and 120°C using an OFITE High Pressure High Temperature (HPHT) Filter Press are presented. The mud cake thickness was measured as a fraction of a millimeter. KPOBM showed a comparatively higher HTHP volume of 4.4ml to 3.6ml of DOBM and PKBDOM of 3.2ml as shown in **Figure 8**.

Table 7: Summary of Rheological Properties

Temperature	Rheological Properties	DOBM	PKOBM	BDBM
49°C	Plastic viscosity, cp	42.0000	30.0000	48.0000
	Apparent viscosity, cp	64.5000	110.0000	66.5000
	Yield point, kg/m^2	2.2000	7.8000	1.8000
	Consistency index	0.0063	0.1968	0.0038
	Flow index	0.7458	0.3079	0.8267
66°C	Plastic viscosity, cp	24.0000	29.0000	25.0000
	Apparent viscosity, cp	32.5000	58.5000	35.0000
	Yield point, kg/m^2	0.8300	2.8800	0.9800
	Consistency index	0.0019	0.0210	0.0026
	Flow index	0.8267	0.5533	0.7947
80°C	Plastic viscosity, cp	22.0000	27.0000	24.0000
	Apparent viscosity, cp	28.0000	39.0000	30.5000
	Yield point, kg/m^2	0.5859	1.1718	0.6347
	Consistency index	0.0006	0.0041	0.0014
	Flow index	0.9994	0.7437	0.8694

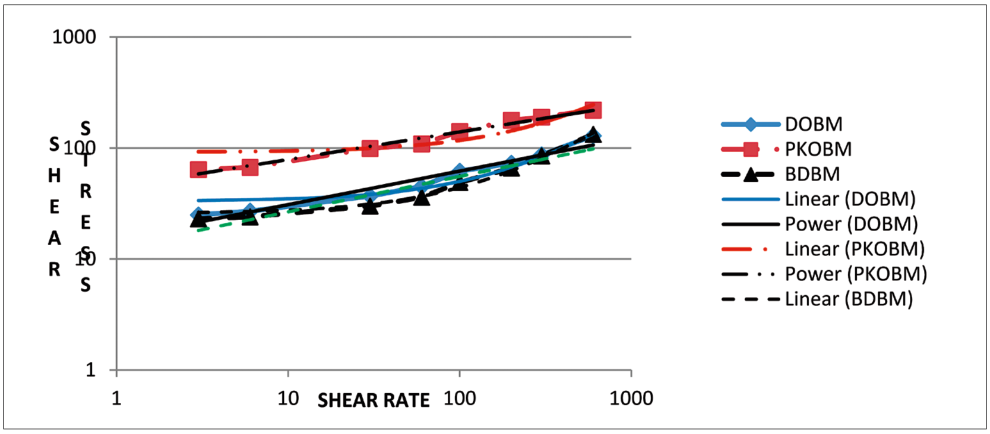


Figure 10: comparison of the flow profile of the mud samples using Bingham plastic and Power law models at 49°C

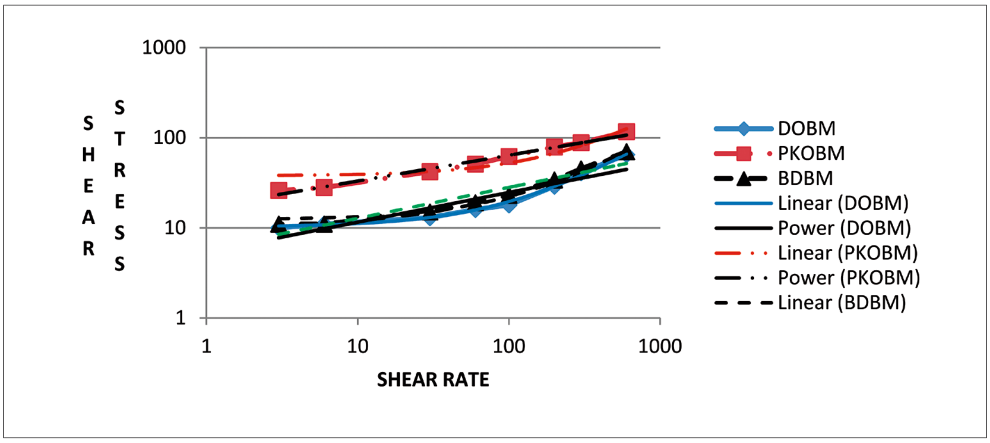


Figure 11: comparison of the flow profile of the mud samples using Bingham plastic and Power law models at 66°C

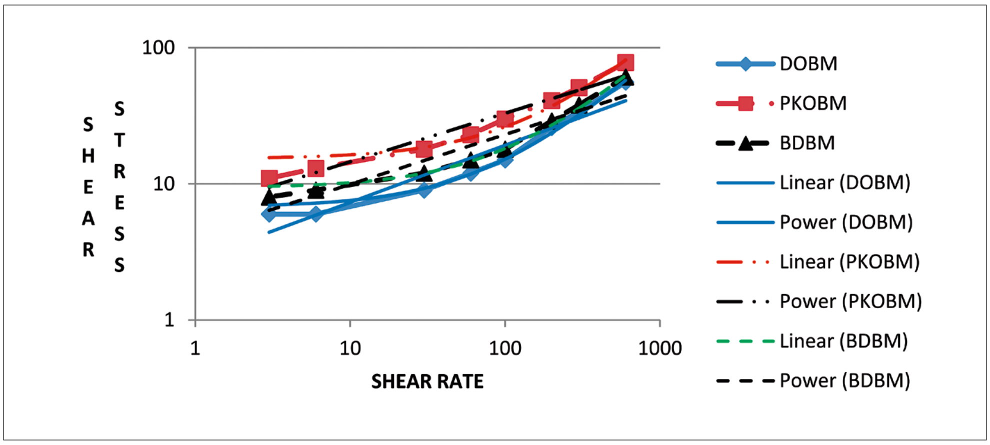


Figure 12: comparison of the flow profile of the mud samples with Bingham plastic and Power law models at 80°C

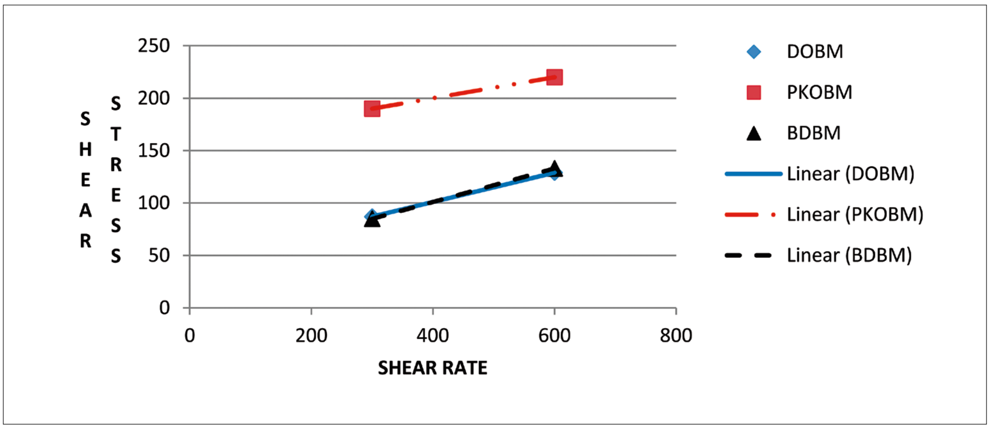


Figure 13: Plot of Bingham plastic shear stress-shear rate at 49°C

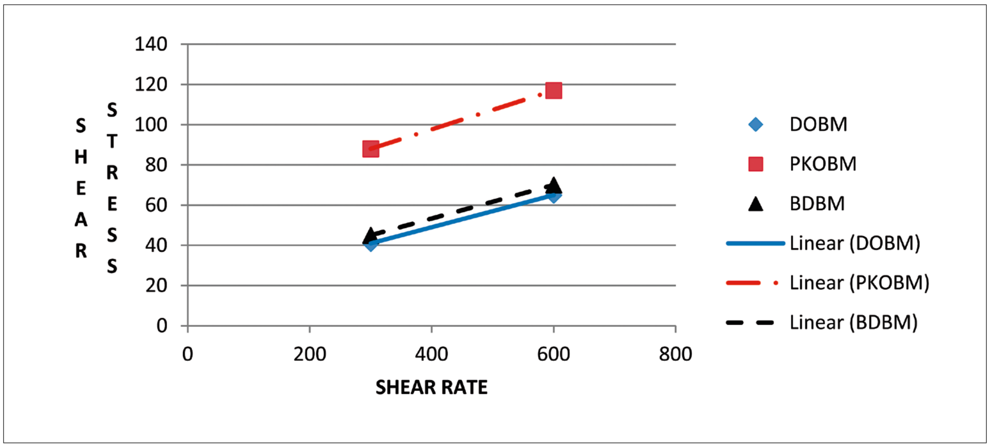


Figure 14: Plot of Bingham plastic shear stress-shear rate at 66°C

3.5 Emulsion and Thermal Stability of the Mud Samples

The emulsion characteristics study showed that diesel and PKO biodiesel based mud have good and comparable emulsion stability as shown in **Figure 9**. The thermal stability of the drilling fluids is usually a measure of the 1-minute shear strength of the mud systems before and after they were subjected to a temperature of 120°C in an oven for 6 hours and allowed to cool to room temperature.

The Emulsion Stability (ES) obtained agreed with the stated typical minimum ES values of 300 V to 800 V for 960 to 1440 kg/m^3 mud and 750 V to 2000 V for higher densities in the work of **Ali, et al. (1987)** and **Growcock**

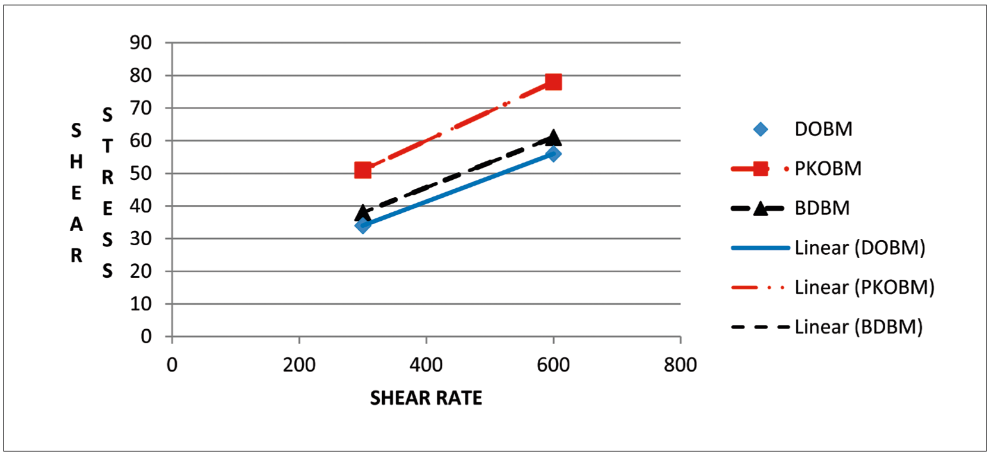


Figure 15: Plot of Bingham plastic shear stress-shear rate at 80°C

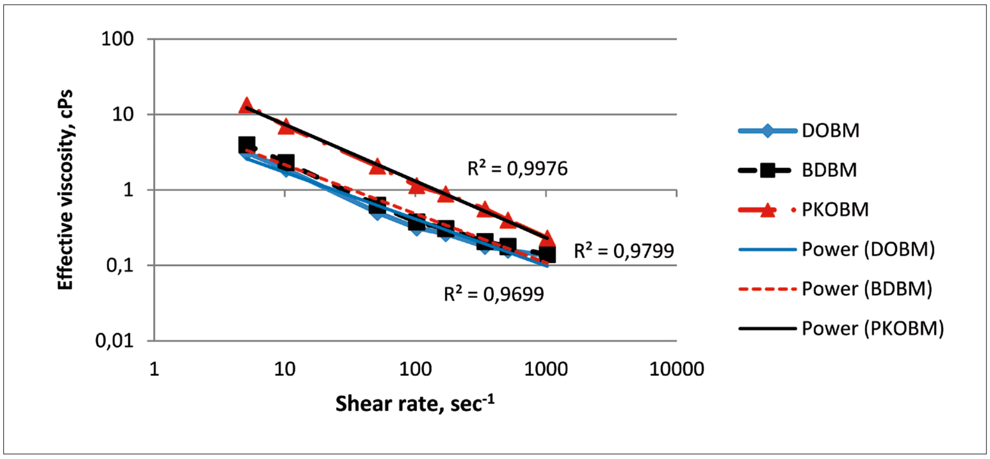


Figure 16: Plot of the effect of shear rate on effective viscosity at 49°C

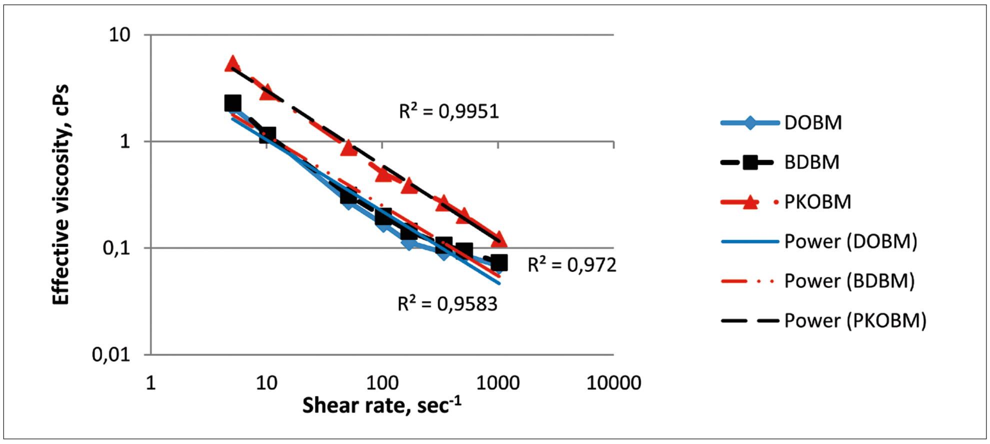


Figure 17: Plot of the effect of shear rate on effective viscosity at 66°C

et al., (**1997**) and also basic guidelines given by **Lyons and Plisga** (**2004**). When mud is subjected to an elevated temperature for long periods of time and allowed to cool, it thickens. The diesel and biodiesel mud systems show comparatively the same thermal stability as shown in **Figure 5**. The shear strength before and after the mud systems were subjected to a temperature of 120°C for 6 hours showed that the DOBM and BDBM were thermally stable with less significant changes in 1 minute shear strengths before and after ageing of 0.83 kg/m^2 and 0.98 kg/m^2 for DOBM and 0.88 kg/m^2 and 0.98 kg/m^2 for BDBM. PKOBM was less stable with

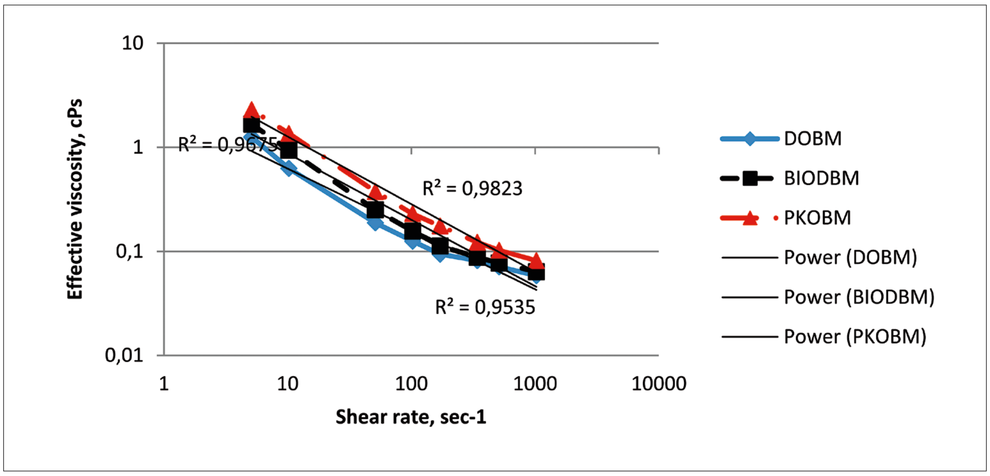


Figure 18: Plot of the effect of shear rate on effective viscosity at 80°C

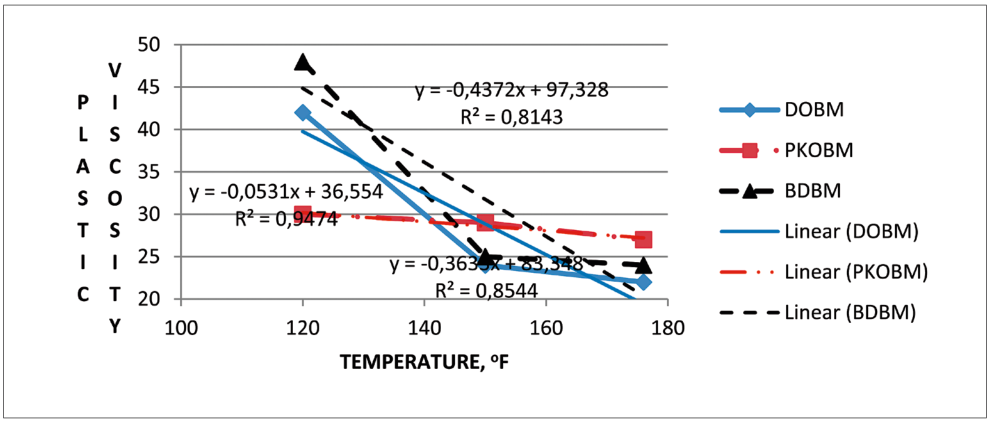


Figure 19: Effect of temperature on Plastic viscosity of Mud samples

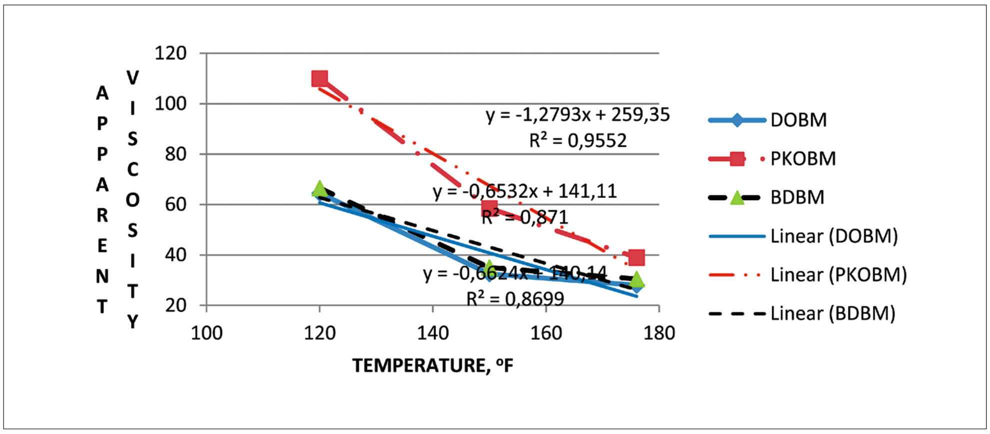


Figure 20: Effect of temperature on apparent viscosity of Mud samples

1.27 kg/m^2 and 1.71 kg/m^2 before and after ageing respectively.

3.6 Summary of Rheological Properties

A summary of the rheological properties obtained from the three mud samples is presented in **Table 7**. From the results obtained, the flow curves of all the mud samples exhibit yield point and viscosity thinning that are consistent with drilling fluids. The flow index (n-value) having a value less than 1 indicates the non-Newtonian behaviour of the mud sample, lower n-values give better hole cleaning since the fluid velocity increases over a larger area of the annulus.

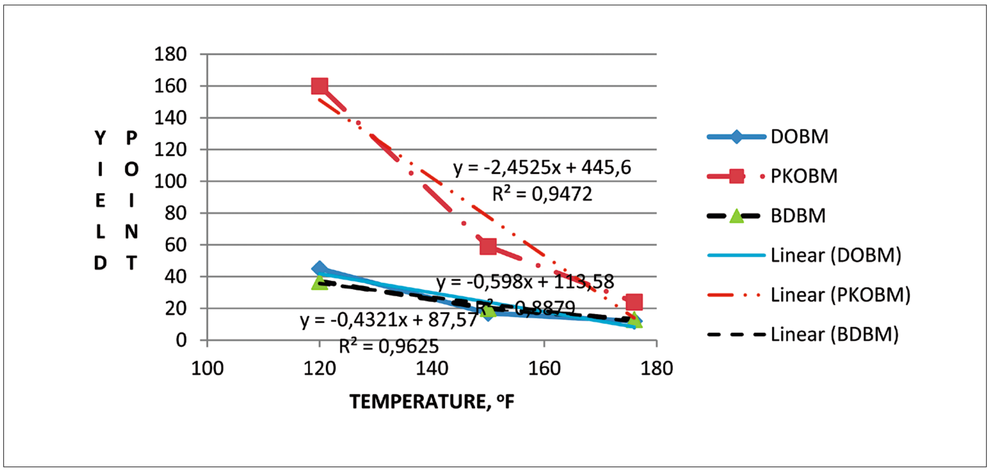


Figure 21: Effect of temperature on plastic yield point of Mud samples

3.7 Toxicity Measurements of Mud Samples

3.7.1 Toxicity of the Base Fluids (Water Soluble Fraction, WSF)

From **Tables 8**, **9** and **10**, it could be inferred that the survival rates in the water-soluble fractions (WSF) of palm kernel oil and palm kernel oil biodiesel were higher than that of diesel oil mud. The estimated 96-hour median lethal concentration (LC_{50}) using Microsoft Excel Forecast Function, showed that, the palm kernel oil water soluble fractions are slightly toxic with a 96 hour median lethal concentration (96-hour LC_{50}) of 9546.76 ppm and PKO biodiesel base fluid showed toxicity with a 96-hour LC_{50} of 6886.36 ppm. Diesel oil is highly toxic with a 96-hour LC_{50} of 2684.21 ppm. It can be seen why the diesel oil based drilling fluid has some setbacks due to environmental regulations.

Table 8: Average Mortality Rate of Diesel Base Oil Water Soluble Fractions (WSF) on Catfish (D. magna).

Time	Average survival on two trials / concentration (ppm)			
	0	2000	5000	10000
12 hours	10	10	9	8.5
24 hours	10	8.5	5	2.5
48 hours	10	3	0	0
72 hours	9.5	0	0	0
96 hours	9.5	0	0	0

Table 9: Average Mortality Rate of Palm Kernel base oil Water Soluble Fractions (WSF) on Catfish (D. magna).

Time	Average survival on two trials / concentration (ppm)			
	0	2000	5000	10000
12 hours	10	10	10	10
24 hours	10	10	10	10
48 hours	10	10	10	10
72 hours	9.5	9	8	8
96 hours	9.5	8	6	6

Table 10: Average Mortality Rate of PKO Biodiesel base oil Water Soluble Fractions (WSF) on Catfish (D. magna).

Time	Average survival on two trials / concentration (ppm)			
	0	2000	5000	10000
12 hours	10	10	10	10
24 hours	10	10	10	8
48 hours	10	9.5	9	8
72 hours	9.5	9.5	9	6.5
96 hours	9.5	7	5.5	4

3.7.2 Toxicity of the Drilling Fluids (Suspended Particulate Phase, SPP)

As shown in **Tables 11**, **12** and **13**, the Suspended Particulate Phase (SPP) of all the mud samples were non-toxic. A substance with a 96 hour-LC_{50} greater than 10000 ppm is considered non-toxic as established in the literature. Palm kernel oil based mud SPP with 96hLC_{50} of 29000 ppm and PKO biodiesel based mud, 15000 ppm are far less toxic when compared with that of diesel based mud (8877.36 ppm). Palm kernel oil is generally considered non-toxic and contains no aromatic compound based on the above evaluations.

Table 11: Suspended Particulate Phase (SPP) Toxicity Test of the Diesel Oil Based Mud Sample

Time	Average survival on two trials / concentration (ppm)			
	0	2000	5000	10000
12 hours	10	10	10	9
24 hours	10	10	10	9
48 hours	10	9.5	8	7.5
72 hours	9.5	8	7	6
96 hours	9.5	8	7	4.5

Table 12: Suspended Particulate Phase (SPP) Toxicity Test of the Palm Kernel Oil Based Mud Sample

Time	Average survival of two trials / concentration (ppm)			
	0	2000	5000	10000
12 hours	10	10	10	10
24 hours	10	10	10	10
48 hours	10	10	10	10
72 hours	9.5	9	10	8
96 hours	9.5	9	8.5	8

Table 13: Suspended Particulate Phase (SPP) Toxicity Test of the PKO Biodiesel Based Mud Samples

Time	Average survival on two trials / concentration (ppm)			
	0	2000	5000	10000
12 hours	10	10	10	9
24 hours	10	10	10	9
48 hours	10	9.5	8.5	9
72 hours	9.5	8.5	8.5	7
96 hours	9.5	8.5	8	6.5

3.8 Statistical Analysis

Chi-square statistical analysis was based on 0.05 level of significance as can be seen in **Table 14**. The mortality rate of diesel oil WSF was dependent on its concentration but did not show any significant dependence of mortality rate on the concentration of both palm kernel oil and PKO biodiesel WSF. In the case of Suspended Particulate Phase (SPP), the mortality rate of test spices was independent of the concentration of the SPP of all the mud samples.

Table 14: Results of Chi-square Test

Mud samples	Diesel	Palm kernel oil	PKO Biodiesel
Degree of freedom	12	12	12
Tabulated chi square	21.03	21.03	21.03
Calculated chi square for WSF	36.59	0.97	1.36
Calculated chi square for SPP	0.43	0.17	0.23

4. Conclusion

This study has shown a remarkable improvement by transesterification of vegetable oils as base fluid in the formulation of invert emulsion drilling mud. The transesterification of vegetable oils to form biodiesel reduces the initial kinematic viscosity (23.41 mm^2/s) and pour point (13°C) of the oil to 11.39 mm^2/s and 6°C respectively. However, its values fall outside the API specifications for drilling mud base fluid, kinematic viscosity, >2.24 mm^2/s and pour point, -9°C. Commercial biodiesels with values within the API range are available in the market. The biodiesel based mud (BDBM) compared favourably technically, far less toxic and more economical than the diesel based mud (DOBM). The biodiesel based mud (BDBM) required lesser quantity of barite (184.6 g) as against 194.4 g for diesel based mud to achieve the same mud weight of 1378 kg/m^3. The rheological properties of the BDBM compared favourably with that of DOBM mainly at higher temperature of 80°C where the PV, YP, 10 sec Gel and 10min Gel are 24 cp, 0.63 kg/m^2, 0.59 kg/m^2 and 0.68 kg/m^2 respectively as against 22 cp, 0.59 kg/m^2, 0.44 kg/m^2 and 0.54 kg/m^2 respectively for DOBM. **Figures 10, 11** and **12** compare the three mud samples on shear stress versus shear rate showed that they conform with both Bingham Plastic and Power Law models at temperatures of 49°C, 66°C and 80°C. The trend of the mud samples in **Figures 13** to **15** using Bingham Plastic models at temperatures of 49°C, 66°C and 80°C agree with the works of **Mohammed** (**2016**) and **Onuh et al.** (**2019**). It can be observed in **Figures 16** to **18**, that the lower the Shear rate, the higher the effective viscosity at the reference temperatures. The plastic viscosity was decreasing with an increase in temperature in **Figures 19** to **21**, typical of a drilling fluid and in line with the findings of **Amani and Al-Jubouri** (**2012**), **Jaffal et al.,** (**2016**) and **Igwilo et al.** (**2017**). The 30-minute filtration test at 120°C and 3450 kPa condition shows all oil filtrate for all the samples with 3.6 ml for DOBM and 3.2 ml for BDBM. Mud cake thickness of DBM and BDBM are the same, 0.5/32 in respectively. Biodiesel mud formed a more stable emulsion (1274 volts) compared to diesel mud (1169 volts). Diesel based mud shows a superior thermal stability. When aged for 6 hours at 120°C in a static condition. The 1-minute shear strength of DBM, before and after ageing, are 17 and 20 respectively while that of biodiesel based mud are 18 and 25. This study has shown that biodiesel base fluid, although relatively toxic with a 96-hour LC_{50} of 6886.36 ppm, is far less toxic when compared to diesel, 96-hour LC_{50} 2684.21 ppm. The biodiesel mud is practically non-toxic with a 96-hour LC_{50} of 15000 ppm. This study has shown that biodiesel can be considered an alternative to DOBM due to its environmental effects though there is need for field pilot test. The biodiesel used in this study was produced in a laboratory with its limitations. The quality of biodiesel can be improved in subsequent studies to match commercial grade.

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SAŽETAK

Eksperimentalno ispitivanje sintetizirane isplake na bazi biodizela

Kao bazni fluid za pripremu isplake na bazi ulja najčešće se upotrebljava dizelsko ulje. Dizelsko ulje ima negativan utjecaj na okoliš i sve je veća potreba za ekološki održivijim alternativama koje se s tehničkoga aspekta mogu usporediti s dizelskim baznim uljem. U novije se vrijeme istražuje mogućnost upotrebe biljnih ulja kao baznoga ulja u isplakama. U ovome istraživanju eksperimentalno su pripremljene isplaka (gustoće 1378 kg/m3) na bazi palmina ulja (ulje iz palminih koštica) (PKOBM) i isplaka na bazi biodizela dobivenoga iz palmina ulja (BDBM). BDBM je dobiven transesterifikacijom biljnoga (palmina) ulja. Zatim je napravljena usporedba karakteristika PKOBM-a i BDBM-a i konvencionalne isplake na bazi dizelskoga ulja (DOBM). Isplake su uspoređene na temelju reoloških i filtracijskih svojstava, sposobnosti stvaranja isplačnoga obloga, stabilnosti emulzije, toplinske stabilnosti te akutne toksičnosti. Rezultati dobiveni istraživanjem upućuju na to da isplake PKOBM i BDBM pokazuju tipično Herschel-Bulkley (modificirani eksponencijalni (*power-law*) model) reološko ponašanje pri temperaturama od 49 °C, 66 °C i 80 °C. BDBM je pokazao usporediva reološka svojstva uz bolji kapacitet iznošenja krhotina nego što bi na to upućivao niži indeks protoka. U testu gubitka filtrata isplake BDBM je pokazao nešto manji gubitak filtrata u odnosu na DOBM, dok je PKOBM imao veći gubitak filtrata, koji je iznosio 4,4 ml. Ispitivanja isplačne obloge (*filter cake*) i termičke stabilnosti nisu pokazala znatnije razlike između DOBM-a i BDBM-a, dok je PKOBM imao najmanje poželjne performanse. U usporedbi stabilnosti emulzija BDBM je pokazao najstabilniju emulziju (destabilizacija emulzije pri naponu od 1274 V), a PKOBM najmanje stabilnu emulziju (destabilizacija pri 739 V), dok u slučaju DOBM-a do destabilizacije emulzije dolazi pri naponu od 1169 V. Temeljem provedenih istraživanja zaključeno je da bi se BDBM mogao koristiti kao ekološki održiva zamjena za isplaku na bazi dizelskoga ulja (DOBM).

Ključne riječi:
biodizel, reološka svojstva, gubitak filtrata, stabilnost emulzije, termalna stabilnost, akutna toksičnost

Author's contribution

Ugochukwu I. Duru, George O. Nduwuba and **Princewill M. Ikpeka** designed the experimental work. While **George O. Nduwuba** performed the experiment, **Ugochukwu I. Duru and Ifeanyichukwu M. Onyejekwe** supervised the experiment at Department of Petroleum Engineering Laboratory. The four authors **(U.I. Duru, G.O. Nduwuba, I.M. Onyejekwe** and **P.M. Ikpeka)** analysed the experimental results obtained and participated in writing the final draft. The first draft was written by **Ugochukwu I. Duru** and **George O. Nduwuba.**