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## **Performance Evaluation of a Benign Oil-Based Mud from Non-Edible Sweet Almond Seed *prunus amygdalus dulcis* Oil**

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### **Abstract**

Oil based mud formulated with diesel is commonly used to drill unforgiving formations, such as shale, high pressure high temperature (HPHT), deep water and ultra-deep water, and to improve wellbore stability. However, the major shortcoming of this mud lies in its adverse environmental impact. They are non-biodegradable and highly toxic, thus leading to high cost of wastes treatment. A benign and biodegradable OBM was formulated from sweet almond seed oil (SASO) using Soxhlet Extraction Method. The SASO base oil was converted to SASO methyl ester through the process of transesterification. The possibility of applying this biodiesel-based drilling mud (BBDM) for drilling especially under HPHT and shale formations were examined. The results indicated that the rheology, filtration characteristics, electrical stability, thermal stability and shale swelling inhibition of the BBDM are comparable with those of the diesel OBM. The biodiesel has a flash point of 169 °C and is significantly higher than that of the diesel of 78 °C; indicating its ability to provide better fire safety than the diesel. The results also confirm that the biodiesel is non-toxic because it has significantly higher lethal concentration 50% (LC<sub>50</sub>) and effective concentration 50% (EC<sub>50</sub>) than those of the diesel. After 28 days' period of biodegradation tests, the BBDM displayed 83% aerobic biodegradation with *Penicillium sp.*, while the diesel OBM exhibited 25.2%. The low branching

degree and absence of aromatics in the BBDM are the reasons for its higher biodegradation. The selected biodiesel is a naturally occurring seed oil; therefore, its use could be essential for drilling contractors and service companies owing to its great environmental acceptability.

**Keywords:** Biodegradation, Biodiesel, Diesel, Sweet almond seed oil, Rheology, shale inhibition

## Introduction

One of the major components during the drilling of wellbore for oil and gas is the drilling fluid. They are indispensable and play crucial roles in the drilling process. These roles include, but are not limited to the following: (1) cooling and lubricating the drill bit and drill string to minimize wear; (2) creating and ensuring an overbalanced drilling condition to control the formation pressure; (3) transmitting downhole information to the surface for interpretation in the form of signals from the drilled well; (4) sealing off permeable formation by creating a strong, thin and low permeable mud cake at the borehole of the permeable formations; (5) circulating drilled cuttings and other solids particles from the wellbore to the surface (Fink, 2015; Ismail *et al.*, 2016; Fattah and Lashin, 2016; Kafashi, *et al.*, 2017; Gbadamosi *et al.*, 2018a; Oseh *et al.*, 2018;; Hakim *et al.*, 2018; Boyou *et al.*, 2019). Therefore, for any successful drilling operation, high-working performance of circulating drilling fluid is required.

The working-performance of drilling fluid relies on its rheology, filtration control additives and its shale inhibition characteristics (Priyanko *et al.*, 2018; Saboori *et al.*, 2018; Oseh *et al.*, 2018; Yeu *et al.*, 2019). Drillers are often faced with numerous problems as well gets deeper, hotter and longer than they have ever been. These problems range from the potential destruction of the mud properties under such great depths and temperatures, as well as higher operational costs (Abduo *et al.*, 2016; Smith *et al.*, 2018; Gbadamosi *et al.*, 2018b; Oseh *et al.*, 2019a, b). As drilling petroleum wells becomes increasingly challenging, drillers must apply advanced drilling fluid technologies with innovative methods to optimize well economics. This could involve developing a high performance drilling fluid additives especially the continuous phase additives to preserve the degradation of fluid's rheology with changes in temperature, shear and pressure which are typically observed during fluids circulation downhole at deeper drilling depths.

Over the years, two major drilling muds are commonly used to drill wells in the oil and gas industry. The drilling muds are water-based muds (WBMs) and oil-based muds (OBMs). WBMs are used for the less-demanding drilling of conventional vertical wells at medium depths, while OBMs are adjudged to have higher cuttings transport capacity and drilling efficiency than WBMs ((Ismail *et al.*, 2015; Sayindla *et al.*, 2017). OBMs are better suited for greater drilling depths, directional or horizontal well drillings which place greater stress on the drilling apparatus than WBMs (Ismail *et al.*, 2015; Li *et al.*, 2016a, b; Sulaimon *et al.*, 2017; Sayindla *et al.*, 2017). Thus, OBMs has been the drilling fluid of choice to improve wellbore stability and to drill in shale, HPHT, deep water and ultra-deep water formations (Wang *et al.*, 2012; Sulaimon, *et al.*, 2017; Sayindla *et al.*, 2017; Li *et al.*, 2018).

In addition, OBMs are preferable in drilling highly difficult sensitive formations because they provide excellent lubricity, lower filter cake permeability, better wellbore stability and higher rate of penetration than WBMs (Ismail *et al.*, 2011; Sulaimon *et al.*, 2017). However, application of OBMs formulated with petrol-diesel or mineral oil as the external phase has been seriously restricted by increasingly strict environmental laws in recent years. This is largely due to their toxic effluents and adverse effects on the environments. To resolve this issue, ester-based muds (EBMs) were designed. EBMs are designed with biodegradable and low toxic esters (Ismail *et al.*, 2011; Sulaimon *et al.*, 2017). They show operational performance comparable to those of diesel OBMs and mineral OBMs, and have less adverse effects on the ecosystem. EBMs faced their first field trials in Norwegian offshore and were successful in many ecologically sensitive formations, such as North Sea (Peresich *et al.*, 1991), Australia (Eckhout *et al.*, 2000), and Gulf of Mexico (Burrows *et al.*, 2001).

However, many esters used today to design EBMs are obtained from chemicals. As a result, they have higher drilling costs than diesel OBMs and mineral OBMs, making their complete use for drilling fluids restricted (Ismail *et al.*, 2011; Sulaimon *et al.*, 2017; Li *et al.*, 2018). Furthermore, EBMs has many drilling challenges, such as wellbore instability at challenging environments and poor temperature resistance (Li *et al.*, 2016a, b). Control of their viscosities and their susceptibility to hydrolysis in acidic or alkaline medium have also increased their disadvantages (Li *et al.*, 2016a, b). Operators are exploring for a lower cost and more technically dependable with high-properties performance ester source for non-aqueous based drilling muds to improve drilling operations.

As a response to improve the performance of synthetic OBMs, biodiesel was introduced as a substitute to diesel OBMs and mineral OBMs. Biodiesel simply can be referred to as the mono-alkyl esters of long-chain fatty acids obtained from renewable oils or fats from plant or animal source (Wang *et al.*, 2012; Sun *et al.*, 2013; Li *et al.*, 2016a, b; Yadav *et al.*, 2017). In terms of chemical configuration, biodiesel can function as the external phase of drilling mud as it provides all the environmental and technical advantages of conventional EBMs (Ismail *et al.*, 2011; Li *et al.*, 2016b; Sulaimon *et al.*, 2017). Biodiesels are produced from sources, such as edible sugars and starches, non-edible plant materials, algae and other microbes which are non-toxic and biodegradable (Li *et al.*, 2016a, b; Wang *et al.*, 2017).

A wide-range of edible and non-edible oils have been used to produce biodiesels from plants and animal sources (Li *et al.*, 2016a, b; Sulaimon *et al.*, 2017). Animal oils and fats, lard, tallow, waste-cooking oils, chicken fats, yellow grease and insect oils have all been used to produce biodiesels (Balat and Balat, 2010; Amin *et al.*, 2010; Wakil, 2015; Li *et al.*, 2016a, b; Li *et al.*, 2018). The cost of producing biodiesel is between 60–90% of the over-all cost of the feedstock (Giwa *et al.*, 2012). Presently, more than 95% of world's biodiesel production is derived mainly from edible and conventional vegetable oils despite the increase in price and competition arising from edible and non-edible uses (Wakil *et al.*, 2015).

Non-edible oils, waste oils or waste cooking oils are expected to help minimize food shortage and the cost of feedstocks used for biodiesel production. Therefore, utilising non-edible oils as base oils to formulate biodiesel-based drilling mud (BBDM) or invert emulsion drilling mud (IEDM) can provide better wastes management of oil rig drilling effluents and protection of the ecosystem (Zhong *et al.*, 2016). Recent increasing demand for more efficient feedstock resources coupled with fast growing production technology have caused biodiesel to become competitive economically (Zhong *et al.*, 2016; Li *et al.*, 2018). These have spurred researchers to attempt in formulating a high-properties performance, lower production cost, eco-friendly BBDM, though none seem to be a complete success.

Different studies conducted on synthetic OBMs either using glycerine ester or mono-alkyl ester of fatty acids for drilling fluids shows that biodiesel has comparable properties with petrol-diesel (Li *et al.*, 2016a, b; Wang *et al.*, 2017; Fakharany *et al.*, 2017a, b). Li *et al.*, (2016a, b) reported the development of biodiesel from waste cooking oil. They stated that the biodiesel has comparable basic properties with that of the petrol-diesel. The biodiesel has high flash point, acceptable elastomeric material property, remarkable biodegradability and non-toxicity. Sulaimon *et al.*, (2017) esterified and transesterified Malaysian crude palm oil to palm methyl ester (PME) to formulate IEDM. The formulated PME mud samples were tested at 300 °F and found to possess comparable properties with conventional diesel OBM. The PME passed the acute toxicity test by showing more than 50% survival rates of *Poecillia reticulata* marine species.

Paswan *et al.*, (2016) evaluated the use of Jatropha oil for the formulation of IEDM and reported that the Jatropha oil based IEDM had lower coefficient of friction, thereby indicating a higher lubricating property of the mud than that of petrol-diesel OBM. Furthermore, the Jatropha base oil used to formulate the IEDM had a lower formation damage effect and high shale recovery performance compared with the diesel OBM; thus, showing a great prospect in the development of biodiesel based drilling muds (Fakharany *et al.*, 2017a). Fakharany *et al.*, (2017b) also reported comparable properties of biodiesel formulated from Jatropha and soybean oil with those of petrol-diesel. They reported higher flash point and better electrical stability. Thus

far, IEDM formulated from biodiesel exhibits desirable and sterling operational properties comparable to diesel OBM.

As a type of mono-alkyl ester, this study proposed the use of almond seed oil to function as an external phase of BBDM substituting for conventional petrol-diesel OBMs and mineral OBMs. Almond seeds are one of the oil-bearing plants for the production of biodiesel and they belonged to the *Rosaceae* family. (Ogunsuyi and Daramola, 2013; Giwa and Ogunbona, 2014). Sweet almond seed oil (SASO) (*Prunus amygdalus "dulcis"*) are reported to contain approximately 51% lipid, 19% protein, 18% carbohydrate and 12% fibre (Giwa and Ogunbona, 2014). The oil content of sweet almond seed is about 35–60% (Giwa and Ogunbona, 2014; Akubude and Nwaigwe, 2016).

The non-edible oil derived from sweet almond seed wastes have been utilized in bioenergy production, used as absorbents to remove metals and dyes, and also for production of activated carbons dyes (Akubude and Nwaigwe, 2016). A study conducted revealed that plants wastes and by-products are economical alternatives to advance biodiesel production (Wang, 2013; Nwosu *et al.*, 2008). Almond seed shells are used as roughage to feed cattles and its hulls are used to feed livestock (Akubude and Nwaigwe, 2016). Almond seed wastes are obtained at low-cost from various processing industries and their wastes can be potent for biodiesel production (Ogunsuyi and Daramola, 2013). Therefore, non-edible seed oil from almond seed could be desirable for biodiesel production as the continuous phase in BBDM just like the *Jatropha* seed oil (Paswan *et al.*, 2016).

Development of BBDMs with non-edible oils is very scarce (Paswan *et al.*, 2016; Ogunsuyi and Daramola, 2013), and the application of conventional edible oils (vegetable oils) are prevalent (Balat and Balat, 2010; Agwu *et al.*, 2015). A few studies were undertaken for the past two decades to develop IEDMs and the external phase for this fluids are edible oils or conventional food vegetable oils. These base oils are not described in details and they include soybean oil (Agwu *et al.*, 2015), melon seed oil (Giwa *et al.* 2010), sun-flower oil (Balat and Balat, 2008), groundnut oil (Dosunmu and Ogunrinde, 2010), rapeseed oil (Demibras, 2007) and palm oil (Izah and Ohimain, 2013). An increasing utilization of these food oils for biodiesel productions can lead to increase cost of production, reduced availability and possibility of increased price of these oils. This study therefore used a non-edible seed oil from sweet almond seed as the continuous phase to formulate a BBDM. A SASO BBDM system was designed, characterized and its basic properties, rheology, filtration characteristics, electrical stability, thermal stability, shale inhibition, eco-toxicity, and biodegradability were evaluated. The potentials of SASO could be important to advance engineering design and basic research.

## Experimental details

### Materials

Pure fatty acid methyl esters, methanol, sodium hydroxide (NaOH) pellet, Sodium sulphate ( $H_2SO_4$ ), powder, potassium hydroxide (KOH) and n-hexane were all obtained from Sigma Aldrich (Merck, Germany). For the base oil, matured sweet almond seeds were obtained from Sabongari market, Kano, Nigeria. The edible portions (flesh) were removed to obtain a hard-stony shells containing the seeds oil. These hard-stony shells were cracked and carefully peeled to remove the seeds. 2 kg of the seeds were collected and sun-dried for 48 hours and oven-dried for 2 hours at 100 °C. The dried seeds were crushed into powder using blending machine. The resultant powder was stored in a cool dry conditions in preparation for oil extraction. The flow process used to produce the BBDM and determination of its properties is shown in Figure 1.



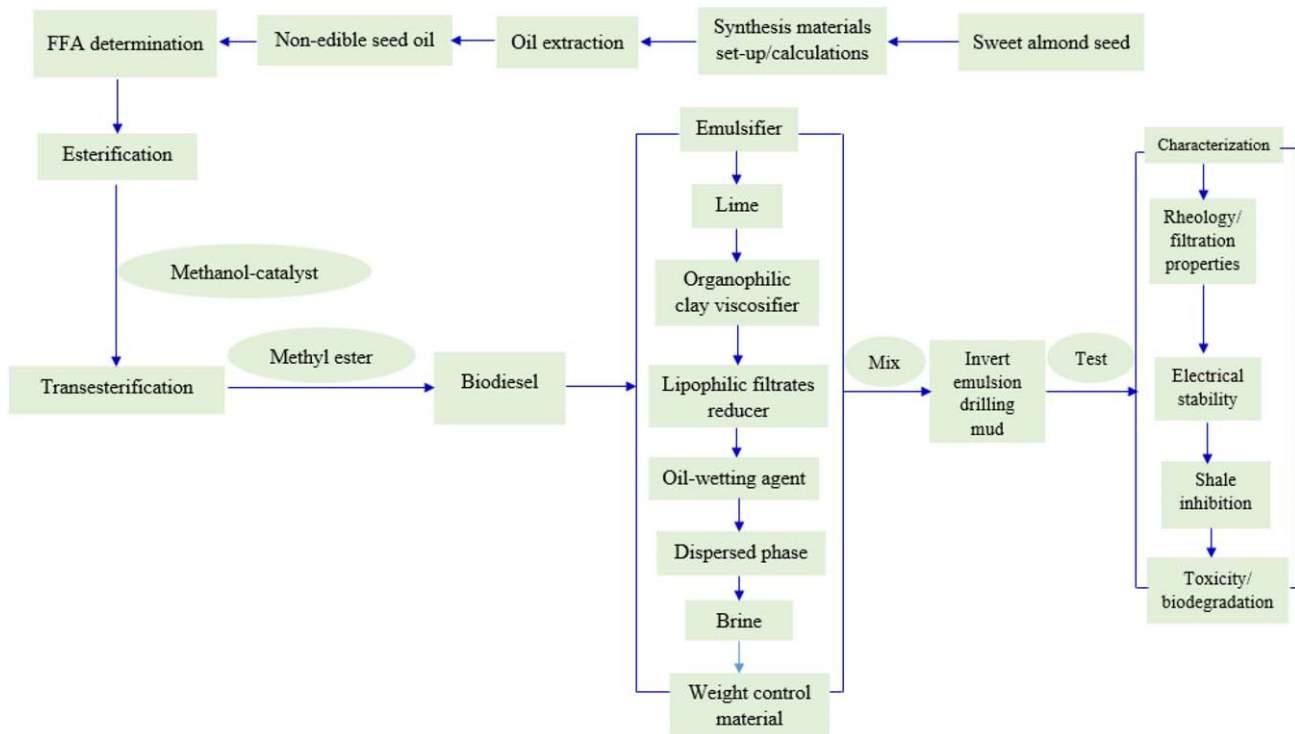


Figure 1—Flow process used to utilize the production and properties performance of non-edible almond seed oil as a biodiesel for drilling fluid

## Oil extraction

2 kg of the sweet almond seeds were collected and sun-dried for 48 hours and oven-dried for 2 hours at 100 °C. The dried seeds were crushed into powder using blending machine. The resultant powder was stored in a cool and dry conditions in preparation for oil extraction. Solvent extraction method with n-hexane was used to extract 1 kg of the lipid components of the milled sweet almond seeds at 60 °C for 6 hours until the extraction was finished. Rotary vacuum evaporator at 60 °C under controlled pressure was used to remove the solvent from the oil. Oil yield was determined from difference in weight of the dried seeds sample before and after extraction. The free fatty acids content of the extracted oil was determined. As shown in Figure 1, fatty acid methyl ester (FAME) was produced from SASO by two steps transesterification process using methanol. The use of methanol in the transesterification process is beneficial because the product glycerol can be separated at the same time during the transesterification process. The end products of the transesterification process are raw glycerol and the raw biodiesel.

## Free fatty acids determination

The conversion rate of free fatty acids (FFAs) in the esterification with  $\text{H}_2\text{SO}_4$ -catalyst reaction was estimated with the titration method (Urrutia *et al.*, 2016). FFA was determined with the aid of titration. 2 g of the SASO was added into a beaker and 10 ml of ethanol was introduced into the beaker. Three drops of chlorophyll indicator were added into the solution to start up the titration. Prepared solution of potassium hydroxide (KOH)-in-ethanol (EtOH) was added into the burette dropwise. The solution was continuously stirred during titration. The volume of KOH used was monitored when the colour of the solution changes from colourless to pink. The FFA content of the extracted oil was 2.46% and the acid value was 4.9 mg KOH/g before the esterification treatment. Further analysis on the sweet almond seed oil methyl ester (SASOME) was carried out using two steps transesterification method.

### Esterification of sweet almond seed oil

Two steps transesterification method was used to produce the biodiesel according to a previous research (Sulaimon *et al.*, 2017). The methods were carried out to avoid the problems of saponification associated with alkaline-catalyzed transesterification and slow reaction time typically associated with the acid-catalyzed transesterification. Due to the high FFAs content which was 4.90% (> 1%), H<sub>2</sub>SO<sub>4</sub>-catalyzed esterification process was implemented to reduce the FFAs content to a level below 1%. A high FFA content that is above 1% will result in soap formation which may weaken the efficiency of the catalyst, cause gel formation, and leads to increase in viscosity. It could also make glycerol separation to be difficult.

The reaction for the esterification process was conducted as follows: An aluminum tray containing water was heated at 60 °C using electric heater. 200 ml sweet almond seed oil was added into 250 ml three-necked bottom flask mounted with reflux condenser. The flask was placed in the tray on the electric heater with magnetic stirrer and temperature regulator. 2 g of sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) was mixed with 43.96 g methanol and used as catalyst in the three-neck bottom flask. The mixture was transferred into a separating funnel and left to cool for 3 hours after being heated for 60 minutes at 60 °C. The mixture was transferred and cooled for 3 hours in a separating funnel. The methanol upper brown layer was removed, while the yellow oil with SASOME lower layer was kept and washed with warm water of 1000 ml at 60 °C until it attained a pH 7. The water was then removed and the oil was cooled in the separating funnel at room temperature. Afterward, electric magnetic stirrer was used to stir the oil for 20 minutes at 100 °C. The remaining oil was SASOME which still contain FFA that was reduced through the second step transesterification using KOH catalyst. Table 1 shows the parameters used in the biodiesel esterification.

Table 1—Parameters for SASO esterification

| Parameters                              | Units | Values | Computation  |
|---|-------|--------|--|
| Pressure                                | psi   | 14.7   | -  |
| Temperature                             | °C    | 60     | -  |
| Time                                    | hour  | 1      | -  |
| Oil : Methanol (ratio)                  | -     | 1 : 6  | Mass of non-edible SASO = 200 g<br>Molecular weight of SASO = 874.5 g/mol<br>Mole of SASO = 0.2287<br><br>Density of methanol = 0.792 g/cm <sup>3</sup><br>Molecular weight of methanol = 32.04 g/mol<br>Mole of methanol = 6 × 0.2287 = 1.3722<br>Mass of methanol = 32.04 × 1.3722 = 43.96 g |
| H <sub>2</sub> SO <sub>4</sub> catalyst | wt.%  | 1      | 200 g × 0.01 = 2.0 g   |

### Transesterification process

180 g of SASO was added into three-necked 250 ml bottom flask equipped with reflux condenser. The flask was then placed in the tray and heated using electric heater with magnetic stirrer and temperature regulator. 1.80 g of KOH concentration was mixed with 39.57 g methanol and used as catalyst in the three-neck bottom flask. The mixture was vigorously stirred for 1 hour at 350 rpm (revolutions per minute) and at 60 °C. The mixture was transferred and cooled for 3 hours in a separating funnel. After the reaction, the obtained products were centrifuged to separate the SASO methyl esters from glycerol. The glycerol at the lower layer was removed by gravity while the upper layer was a mixture of SASO methyl ester and methanol. Vacuum distillation of the mixture using rotating evaporator was used to remove the methanol, and the SASO methyl ester was analyzed after purification. The extracted biodiesel yield of 78.3% was separately collected, washed with warm water and stored prior to characterization and for drilling mud formulation. After the two steps transesterification methods, the FFAs content reduced to 0.39% and the acid value reduced to 0.78 mg KOH/g. In order to avoid error, 3 tests were carried out for the SASO sample

and the average value of the 3 tests were recorded for further examination. Table 2 shows the parameters used in the biodiesel transesterification.

**Table 2—Parameters for SASO transesterification**

| Parameters             | Units | Values | Computation  |
|------------------------|-------|--------|--|
| Pressure               | psi   | 14.7   | -  |
| Temperature            | °C    | 60     | -  |
| Time                   | hour  | 1      | -  |
| Oil : Methanol (ratio) | -     | 1 : 6  | Mass of non-edible SASO = 180 g<br>Molecular weight of SASO = 874.5 g/mol<br>Mole of SASO = 0.2058<br><br>Density of methanol = 0.792 g/cm <sup>3</sup><br>Molecular weight of methanol = 32.04 g/mol<br>Mole of methanol = 6 × 0.2058 = 1.2350<br>Mass of methanol = 32.04 × 1.2350 = 39.57 g |
| KOH catalyst           | wt. % | 1      | 180 g × 0.01 = 1.80 g  |

### Biodiesel characterization

The produced SASOME was tested following the standard biodiesel test methods. The acid values were tested experimentally according to American Society for Testing Materials (ASTM) and European Committee for Standardization (ECN EN) specifications. The Cetane number was determined empirically. The free glycerol, total glycerol and fatty acid methyl ester were also measured according to ASTM and EN specifications, using 6890 N Agilent Gas Chromatography (Santa Clara, CA, USA) mass spectrometry with a flame ionization detector.

### Mud preparation and testing

Table 3 shows the two IEDMs formulations following past researches (Li *et al.*, 2016a, b; Sulaimon *et al.*, 2017; Elkatatny *et al.*, 2018). A standard of 1.0 g of material is added to a 350 ml laboratory barrel for the two IEDMs formulations. The muds were designed to achieve a density of 12 ppg and oil-water ratio (OWR) of 80/20. The SASO base oil was mixed with the mud additives using Hamilton beach multi-mixer in ascending order as shown in Table 3. Similar procedure was used to formulate the diesel OBM from petrol-diesel.

**Table 3—Invert emulsion drilling mud formulations for 350 ml laboratory barrel**

| Additive                       | Function   | SASO mud | Diesel OBM |
|--------------------------------|--|----------|------------|
| Diesel (ml)                    | External phase   | 300      | 300        |
| EZ-MUL (g)                     | Emulsifier and oil wetting agent                                 | 26.2     | 26.2       |
| Invermul (g)                   | Primary emulsifying agent  | 19.2     | 19.2       |
| Lime (alkaline) (g)            | Emulsion stabilizer, alkaline and fatty acid neutralizing agent. | 10.5     | 10.5       |
| Geltone II (g)                 | Viscosifier  | 3.5      | 3.5        |
| Oil-soluble resin (g)          | Filtration control agent   | 6.0      | 6.0        |
| GLO OBM WETT 1000 (g)          | Dispersant and wetting agent                                     | 1.74     | 1.74       |
| Water (ml)                     | Dispersed phase  | 75       | 75         |
| Brine (CaCl <sub>2</sub> ) (g) | Provide shale stability and forms alkaline aqueous phase         | 26.5     | 26.5       |
| Barite (g)                     | Weighting agent  | 296.7    | 296.7      |

### Rheological and filtration properties tests

The rheological properties of the drilling mud formulations were first tested at different temperatures (50, 80, 120 and 150 °C) from 5.11 to 1021 (1/s) shear rates before they were aged in a 4-roller oven at 50 °C. They were tested again to determine the effect of increasing temperature on the newly-formulated IEDMs. Fann model 50SL-HT rheometer was used to test the designed drilling muds before thermal aging (BTA) and after thermal aging (ATA). The gel-strengths (GS) were tested at a low shear rate of 5.11 (1/s) at different temperatures. The API filtrate volume (BTA) was measured using API Fann filter press, while the HPHT filtrate volume (ATA) was measured using Fann HT4700 HPHT filter press at the four different temperatures conditions. The cake thickness formed was measured thereafter.

### Electrical stability tests

The electrical stability (ES) of non-aqueous based drilling fluids is a property related to the stability and oil-wetting ability of its emulsion. Fann electrical stability meter was used to measure the ES of the two IEDMs at test temperatures of 50 °C and 120 °C following the API recommended procedure for field testing oil-based drilling fluids (API 13B-2, 2005). The electrode probe was carefully cleaned, kept dry and circulated in the tests mud samples. Barite and clays were not included in the prepared tests samples in order not to weaken the emulsion. Test samples were placed in a glass beaker and regulated to the test temperature. The samples were hand-stirred using the electrode probe for 10 seconds to obtain uniform sample composition and temperature. The electrode probe surfaces were fully covered by the sample and was stabilized and undisturbed during the measurements. It was positioned in a way of not having any contact with bottom or sides of the glass beaker. Automatic voltage ramp was used to measure the ES of the emulsion and the ES values were obtained when the ramp stopped displaying voltage breakdown. The measurements were repeated and the average values obtained were recorded.

### Shale swelling tests

Figure 2 shows the experimental approach for shale swelling inhibition tests. Before the commencement of drilling operation, it is crucial to understand the compatibility of drilling fluids with the wellbore. The approach to examine the compatibility of shale swelling is to interact the shale with the drilling fluids. Shale sample was obtained from Dange formation, Sokoto, Nigeria and X-ray diffraction (XRD) analysis was employed to select the shale sample with the most amount of smectite/montmorillonite clay mineral.

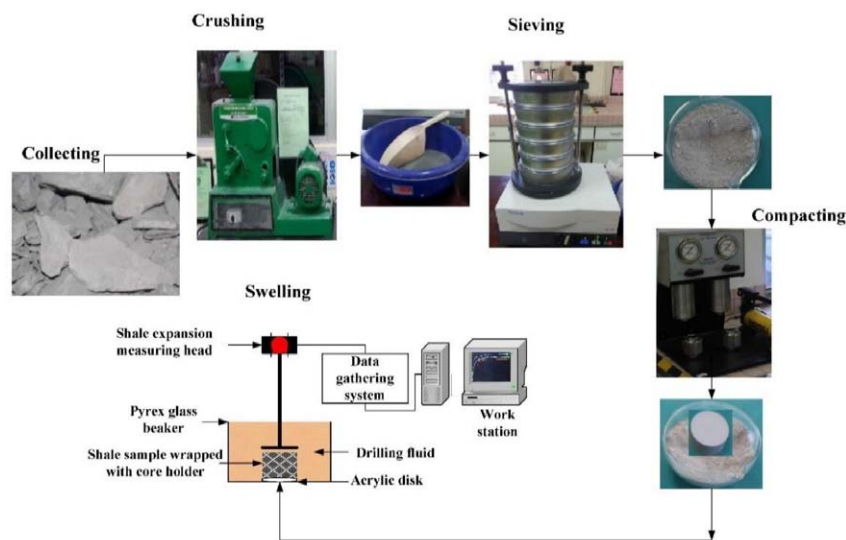


Figure 2—Experimental approach for shale swelling inhibition test



Two shale plugs were prepared from the selected shale cuttings. Linear swell meter (LSM) Fann compactor, model 2100 was used to compact the shale plugs for 90 minutes at 10000 psi. The prepared shale plugs preserved in the desiccator were adjusted in a core holder before being fixed under the measuring head of the Fann LSM as shown in Figure 3. Designed IEDMs were tested for shale swelling inhibition for 36 hours after aging in a 4-roller oven for 16 hours at 50 °C. Shale swelling results were reported in percentage and millimeters of expansion based on initial length. However, a limitation of the test was that the moisture content of the shale plugs kept in the desiccator for 3 hours was not accounted for.



Figure 3—Experimental (a) set up; (b) test of SASO and diesel mud samples with LSM

### Ecotoxicology and biodegradability tests

For the ecotoxicity test, acute toxicity bioassay was conducted using four different marine species for 96 hours following the specifications of Organization for Economic Co-operation and Development for Respirometric Test Procedures (OECD, 301 D, 2001). The marine species are *Poecilia latipinna*, *Tilapia guineensis*, *Moina mongolica* and *Artemia*. The SASO BBDM and diesel OBM were added separately into the test chemicals prepared at a specification of 10, 000 ppm concentration. The fishes were randomly distributed in batches of ten into the 10, 000 ppm concentration. Mortality was recorded at the end of 96 hr for both BBDM and diesel OBM.

The aerobic biodegradation tests of the SASO mud and diesel OBM were conducted using mineral salts medium in a shake flask experiment according to the OECD, 301 D (2001) for Respirometric Test Procedures. The test was achieved with isolated microorganisms for 28 days with aniline ( $C_6H_5NH_2$ ) as the control sample. The compositions of the mineral salt medium used were: 320 ppm KCl (potassium chloride); 400 ppm  $MgSO_4 \cdot 7H_2O$  (magnesium sulphate); 1400 ppm  $K_2HPO_2$  (dipotassium phosphate); 700 ppm  $KH_2PO_4$  (potassium dihydrogen phosphate); 14000 ppm agar ( $C_{14}H_{24}O_9$ ) and 400 ppm  $NaNO_3$  (sodium nitrate). Two commonly used drilled cuttings isolated *Staphylococcus sp.* bacteria and *Penicillium sp.* fungi were used for the measurement. 140 ml concentration of the mineral salt medium was introduced into 250 ml of six different conical flasks and 15 ml each of SASO mud and diesel OBM were added.

*Staphylococcus sp.* and *Penicillium sp.* were prepared using two different conical flasks. Each of the isolate was suspended in a 2 ml of mineral salt medium and incubated at ambient temperature for 28 days on a rotary shaker operated at 110 rpm. The percent biodegradability was calculated from the ratio of biochemical oxygen demand (BOD) and chemical oxygen demand (COD) after 28 days. A plot of percent degradation (BOD/COD) against time was shown in results and discussions section. The anaerobic biodegradability test of the mud samples was achieved according to the test procedures of International Organization for Standardization (ISO 11734: R2017). The test was conducted for 60 days with palmitic ( $C_{16}H_{32}O_2$ ), as the control sample. The test procedures can be found elsewhere (ISO 11734: R2017).

## Results and discussions

### Chemical and fatty acid compositions of SASO biodiesel

Table 4 shows the chemical and fatty acid compositions of the extracted SASO biodiesel. It shows that oleic acid, linoleic acid and palmitic acid are the major fatty acids in the selected feedstock. The extracted biodiesel has high amount of monounsaturated fatty acids of 71.1%, which illustrates that the SASO fatty acid methyl ester could be a good fuel quality (Li *et al.*, 2016a, b). The content of the total saturated fatty acids of the studied biodiesel is 11.1% and the total unsaturated fatty acids is 88.9%, which is a beneficial attribute as a fuel property because it has high stability and cetane number (Moser and Vaughn, 2010).

Table 4—Chemical and fatty acid compositions of SASO biodiesel

| Material                    | Molecular formula                              | Molar mass (g/mol) | Density (g/cm <sup>3</sup> ) | Content (wt. %) |
|-----------------------------|--|--------------------|------------------------------|-----------------|
| SASO yield (%) (w/w) oil    | -  | -                  | -                            | 78.3±2.7a       |
| Acid value (mg KOH/g)       | -  | -                  | -                            | 4.9 b; 0.78 c   |
| Free fatty acid (FFA) (%)   | -  | -                  | -                            | 2.46b; 0.39 c   |
| Palmitoleic acid            | C <sub>16</sub> H <sub>30</sub> O <sub>2</sub> | 254.41             | 0.894                        | 0.3             |
| Palmitic acid               | C <sub>16</sub> H <sub>32</sub> O <sub>2</sub> | 270.45             | 0.853                        | 9.3             |
| Oleic acid                  | C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> | 282.47             | 0.895                        | 69.6            |
| Stearic acid                | C <sub>18</sub> H <sub>36</sub> O <sub>2</sub> | 284.45             | 0.870                        | 1.8             |
| Linoleic acid               | C <sub>18</sub> H <sub>32</sub> O <sub>2</sub> | 280.45             | 0.900                        | 18.1            |
| Linolenic acid              | C <sub>18</sub> H <sub>30</sub> O <sub>2</sub> | 278.43             | 0.914                        | 0.7             |
| Total saturated acids       | Cn:0   | -                  | -                            | 11.2            |
| Total monounsaturated acids | Cn:1   | -                  | -                            | 71.1            |
| Total polyunsaturated acids | Cn:2, 3  | -                  | -                            | 18.8            |
| Total unsaturated acids     | -  | -                  | -                            | 89.9            |

a Indicate average ± standard deviation of three tests;

b indicates initial acid values before treatment;

c indicates values obtained after pretreating the oil - indicate not specified.

### Basic fuel properties of SASO biodiesel and petrol-diesel

Table 5 shows some of the basic (fuel) properties of SASO biodiesel and diesel tested according to ASTM and ECN EN specifications. These properties are typically related to engine performance. They can also help to evaluate the performance of base oils in drilling muds.

Table 5—Basic properties of SASO biodiesel and diesel

| Properties                                       | SASO           | Diesel         | Test method | ECN EN14124  | ASTM D6751  |
|--|----------------|----------------|-------------|--------------|-------------|
| Colour   | Pale yellow    | Light brown    | –           | –            | –           |
| Appearance form                                  | Viscous liquid | Viscous liquid | –           | –            | –           |
| Kinematic viscosity (40 °C) (mm <sup>2</sup> /s) | 4.31           | 3.52           | ASTM D97    | 3.5–5.0      | 1.96–6.0    |
| Density(g/cm <sup>3</sup> ) at 15 °C             | 0.874          | 0.848          | ECN EN14214 | –            | –           |
| Flash point (°C)                                 | 169            | 78             | ASTM D6751  | 120 minimum  | 130 minutes |
| Pour point (°C)                                  | -9             | -18            | ASTM D97    | –            | –           |
| Cold filter plugging point (°C)                  | -5             | -4             | ASTM D6371  | –            | –           |
| Cetane number                                    | 57             | 52             | ECN EN14214 | 51 minimum   | 47 minutes  |
| Linolenic acid content (%mol/mol)                | 0.7            | None           | ASTM D6751  | 12.0 maximum | –           |

| Properties                        | SASO  | Diesel | Test method | ECN EN14124  | ASTM D6751   |
|-----------------------------------|-------|--------|-------------|--------------|--------------|
| Acid value (mg KOH/g)             | 0.13  | 0.006  | ASTM D6751  | 0.5 maximum  | 0.5 maximum  |
| Oxidative stability (hour/110 °C) | 6.0   | 23     | ASTM D6751  | 6 minimum    | 3 minimum    |
| Free glycerol (wt.%)              | 0.013 | None   | ASTM D6751  | 0.02 maximum | 0.02 maximum |
| Total glycerol (wt.%)             | 0.174 | None   | ASTM D6751  | 0.25         | 0.24         |
| Aromatic materials (wt.%)         | None  | 33.8   | ASTM D6751  | –            | –            |
| Water content (wt.%)              | None  | None   | ASTM D6751  | –            | –            |
| Sulphur (wt.%)                    | None  | 0.035  | ASTM D6751  | –            | –            |
| Sediment (wt.%)                   | None  | None   | ASTM D6751  | –            | –            |

The density of a fuel is related to the operational safety of the oil. The more stable the density of mono-alkyl esters, the higher is the capacity of the mud to maintain necessary hydrostatic pressure and transport drilled solids (Sulaimon *et al.*, 2017). According to the data shown in Table 5, the density of the petrol-diesel at 15 °C is 0.848 g/cm<sup>3</sup> and is lower than that of the extracted biodiesel of 0.874 g/cm<sup>3</sup>; therefore, the biodiesel is suitable to be used to prepare IEDMs with a broad-range of density if considered, which is out of scope of this study. The density of the extracted biodiesel also implies that the biodiesel is denser than the diesel at the same temperature, which is mainly due to the fatty acids content, free and bound glycerine content (Benjumea *et al.*, 2008). Thus, the designed biodiesel could lead to better mileage and better engine power than the diesel.

Flash point just like the density is connected to the operational safety of the oil (Wang *et al.*, 2012; Li *et al.*, 2016a). Flash point is the temperature at which fuel will ignite on exposure to spark or flame. Biodiesel having a higher flash point than diesel fuel holds the advantage of higher fire safety for transportation purposes. As shown in Table 5, the SASO biodiesel has a significantly higher flash point of 169 °C than the diesel fuel with 78 °C; demonstrating its capacity to supply better fire safety, transportation and storage than the diesel (Li *et al.*, 2016a). The SASO biodiesel value is within operational range and significantly higher than specified minimum limits by ECN EN 14214 and ASTM D6751 specifications.

Another important property that is used to characterize the quality of a diesel is the cetane number, which is important for good engine performance. As shown in Table 5, the cetane number of biodiesel is comparable to that of petrol-diesel. The cetane number of the biodiesel meets the specified limits stated by ECN EN 14124 and ASTM D6751 quality standards. The cetane number of the extracted biodiesel is 57 and is 9.6% higher than that of the diesel. The higher value of the cetane number of the extracted biodiesel is due its long chain molecular structure comprising of Carbons (C) and hydrogen (H) atoms, with almost no aromatics or branches in the centre (Hasan and Rahman, 2017). Therefore, the studied feedstock could have a better engine performance than the petrol-diesel.

Cold flow properties are another important fuel property that shows advantageous biodiesel properties. The cold filter plugging point indicates the lowest-temperature operability at which fuels can still pass through a certain filtration device at a given time when cooled under certain conditions. The pour point and cold filter-plugging point of the extracted biodiesel were similar to that of the diesel; they both have low operating temperature. The studied biodiesel could be used in any climate without cold flow problems due to their higher monounsaturated content (Verma *et al.*, 2016).

Oxidation stability is another important basic property of fatty acid methyl esters. It is indicative of the stability of fuels during prolonged storage (Kurnia *et al.*, 2016). As shown in Table 5, the oxidation stability at 110 °C of the biodiesel shows 6.0 hours and that of the diesel shows 23 hours. The oxidative stability of the diesel is better than that of the extracted biodiesel. The oxidative stability of both products are above the 3 hours' minimum limit stated by ASTM D6751. The biodiesel indicates good oxidation stability; in which it exhibits a half-year shelf life. The good oxidation stability of the extracted biodiesel is due to its

low content of methyl ester of linoleic acid (C18:2, 17.1%), since it has been reported that its oxidation rate is 12 times higher than that of methyl ester of oleic acid (C18:1) (Pantoja *et al.*, 2013).

There is no threat of sulphur and aromatic materials in the biodiesel as shown in Table 5. Aromatic materials in diesel fuels and mineral oil are important parameters connected to environmental issues. The presence of aromatic materials in diesel fuel and mineral oil is the principal reason of harmful ecological effects and health issues of these oils (Li *et al.*, 2016a, b). Besides, drilling muds having fluorescent aromatics are inclined to affect the analysis of logging data in drilling operations. The produced biodiesel is free of aromatics; hence, non-fluorescent and non-toxic and could be beneficial to be used as the continuous phase of BBDM.

In relation to the purity-related indicators, the biodiesel just like the diesel is free of water and sediment. Important acid values present in the biodiesel and absent in the diesel are free glycerol, total glycerol and free fatty acids. The low operating temperature and viscosity of biodiesel can be greatly affected with high values of these terms (Li *et al.*, 2016a, b). According to the specified limits of ECN EN 14214 and ASTM D6751 standards shown in Table 5, the SASO biodiesel shows acceptable values of free glycerol  $\leq 0.02$ , total glycerol  $\leq 0.24$  and acid value  $\leq 0.5$ .

In relation to the flow properties, the kinematic viscosity at 40 °C of the biodiesel is 4.31 mm<sup>2</sup>/s and is higher than that of petrol-diesel, which is 3.52 mm<sup>2</sup>/s. The kinematic viscosities of the extracted biodiesel and the diesel are within the allowable limits as shown in Table 5 (ECN EN14214, 2003; ASTM D6751, 2009). The biodiesel has a large proportion of monounsaturated (71.1%) and total unsaturated (88.9%) fatty acids; therefore, a higher kinematic viscosity could be expected. This suggests that at 40 °C, the diesel liquid is better than that of the biodiesel. Thus, additional test was carried out on viscosity of the extracted biodiesel before being used to formulate the BBDM. The apparent viscosities of biodiesel and diesel were tested at different temperatures using a Fann model 50SL-HT viscometer and the results are shown in Figure 4. It shows higher temperature-sensitivity of the extracted biodiesel viscosity than that of commercial grade petrol-diesel. Therefore, a balanced formulation was proposed to achieve good SASO BBDM that contain substantially flat viscosity versus temperature profile.

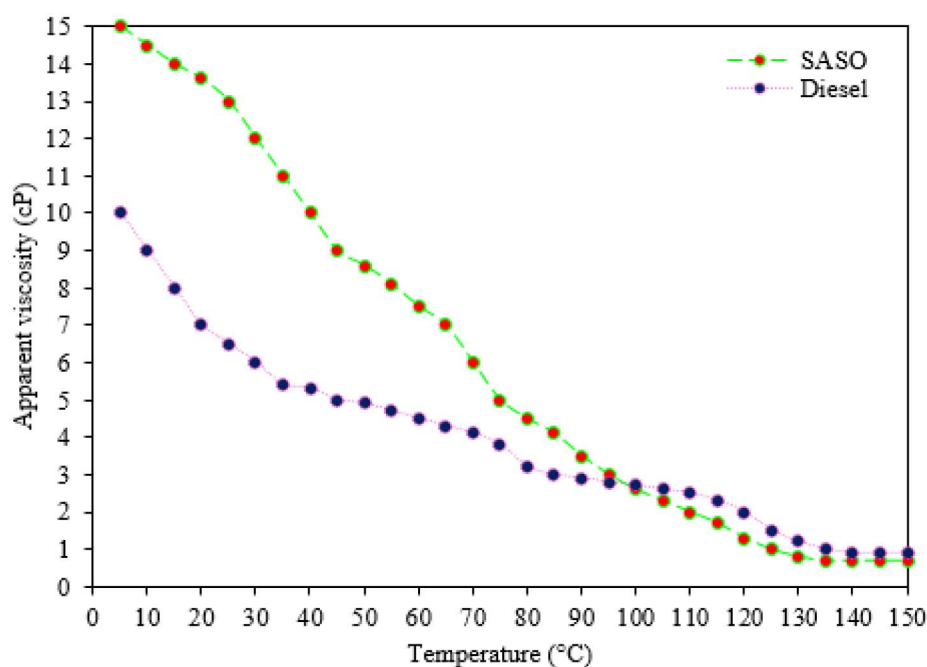


Figure 4—Temperatures versus viscosities profile of SASO biodiesel and petrol-diesel at shear-rate of 1021 (1/s).



## Effect of temperature on the rheological properties of drilling muds

**Shear stress versus shear rate.** Table 6 shows the optimum range of viscosity of drilling muds as recommended by American Society of Mechanical Engineers (ASME, 2005).

**Table 6—API specification for 150 °C before thermal aging (ASME, 2005)**

| Properties                        | Unit                  | Specified limits |
|-----------------------------------|-----------------------|------------------|
| Plastic viscosity (PV)            | cP                    | < 35             |
| Apparent viscosity (AV)           | cP                    | –                |
| Yield point (YP)                  | lb/100ft <sup>2</sup> | 15–25            |
| YP/PV                             | –                     | –                |
| Initial gel-strength (initial GS) | lb/100ft <sup>2</sup> | 6–10             |
| 10-min gel strength (10-min GS)   | lb/100ft <sup>2</sup> | 8–12             |
| Filtrates loss (FL)               | ml                    | < 4              |
| Filter cake thickness (FCT)       | mm                    | < 2              |
| Electrical stability (ES)         | volts                 | > 400            |

The effect of temperature on the rheological behaviour of the diesel OBM and the newly-developed BBDM before and after thermal aging tests are shown in Figures 5(a)-(d). It shows that the rheological trend of the BBDM with temperature variations are similar to those of the diesel OBM. The data also shows that the BBDM and diesel OBM behaves as a Bingham plastic fluid. From the observed gradient, the behaviour of all the mud samples were characteristics of shear-thinning because of their increasing shear stress at increasing shear-rate, which further exemplifies viscosity increment (Boyoun *et al.*, 2019; Oseh *et al.*, 2019b). Theoretically, temperature have effects on rheological behaviour of drilling muds as shown in Figures 5(c) and (d). It shows slightly lower values of rheology for the two mud samples than the values obtained before thermal aging (Figures 5(a) and (b)). According to the data shown in Figure 5(a), at high shear-rate of 1021 (1/s) pertaining to a temperature of 150 °C, the biodiesel had a shear stress of 60 lb/100ft<sup>2</sup> and decreased by 16.7% to 50 lb/100ft<sup>2</sup> after thermal aging (Figure 5(c)). Similar trend was observed at the same shear rate and temperature where the diesel OBM with 62 lb/100ft<sup>2</sup> (Figure 5(b)) decreased to 51 lb/100ft<sup>2</sup> by 17.7% (Figure 5(d)). This results exemplifies that the selected biodiesel has comparable rheology with petrol-diesel.

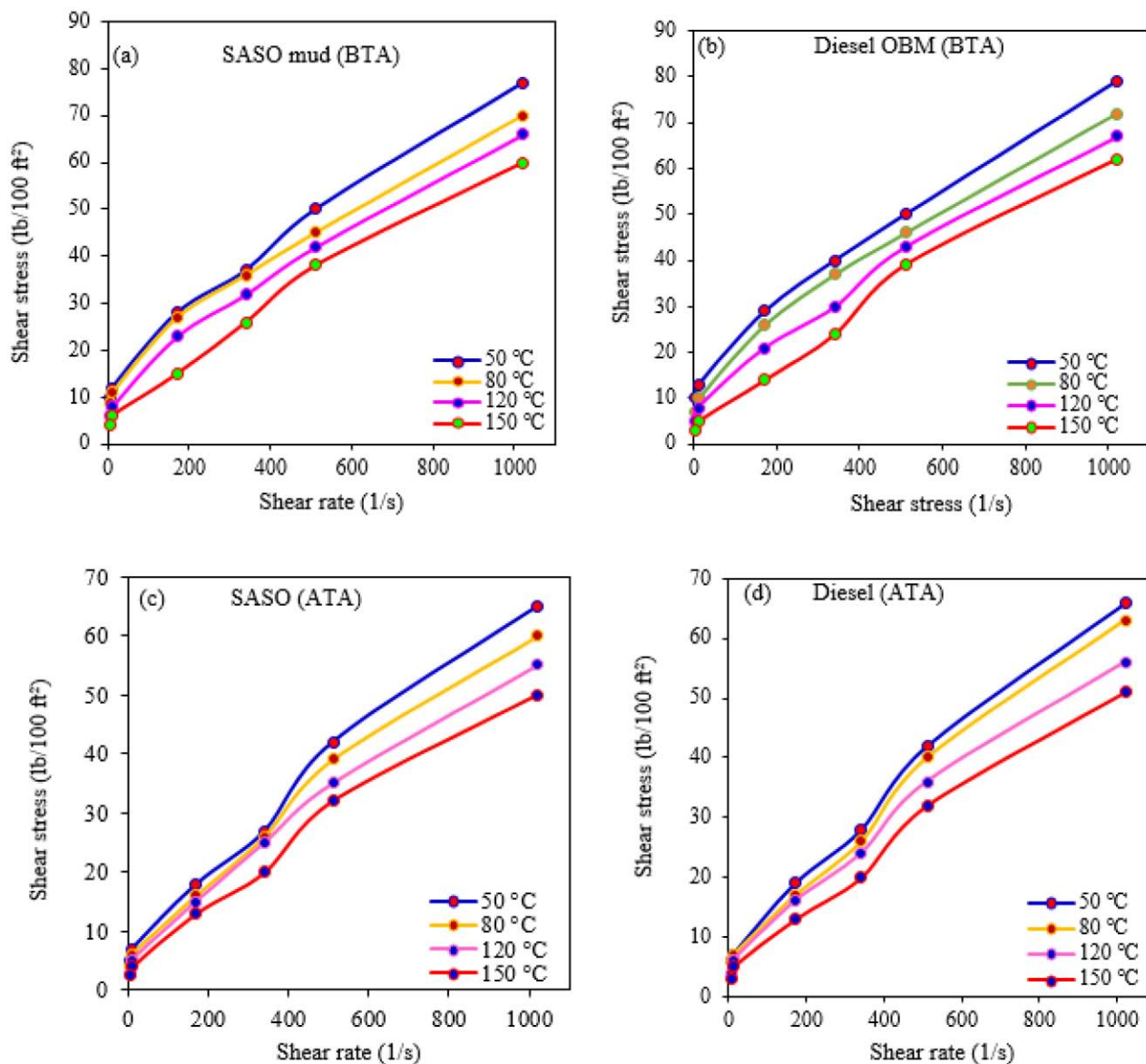


Figure 5—Temperature increment on shear stress vs shear rate of SASO mud and Diesel OBM before and after thermal aging tests. BTA = Before thermal aging tests; ATA = After thermal aging tests at 50 °C for 16 hours.

**Rheological behaviour of mud samples.** The effect of increasing temperature on the rheology of the biodiesel and diesel mud systems are shown in Figures 6(a) 50 °C, (b) 80 °C, (c) 120 °C and (d) 150 °C.

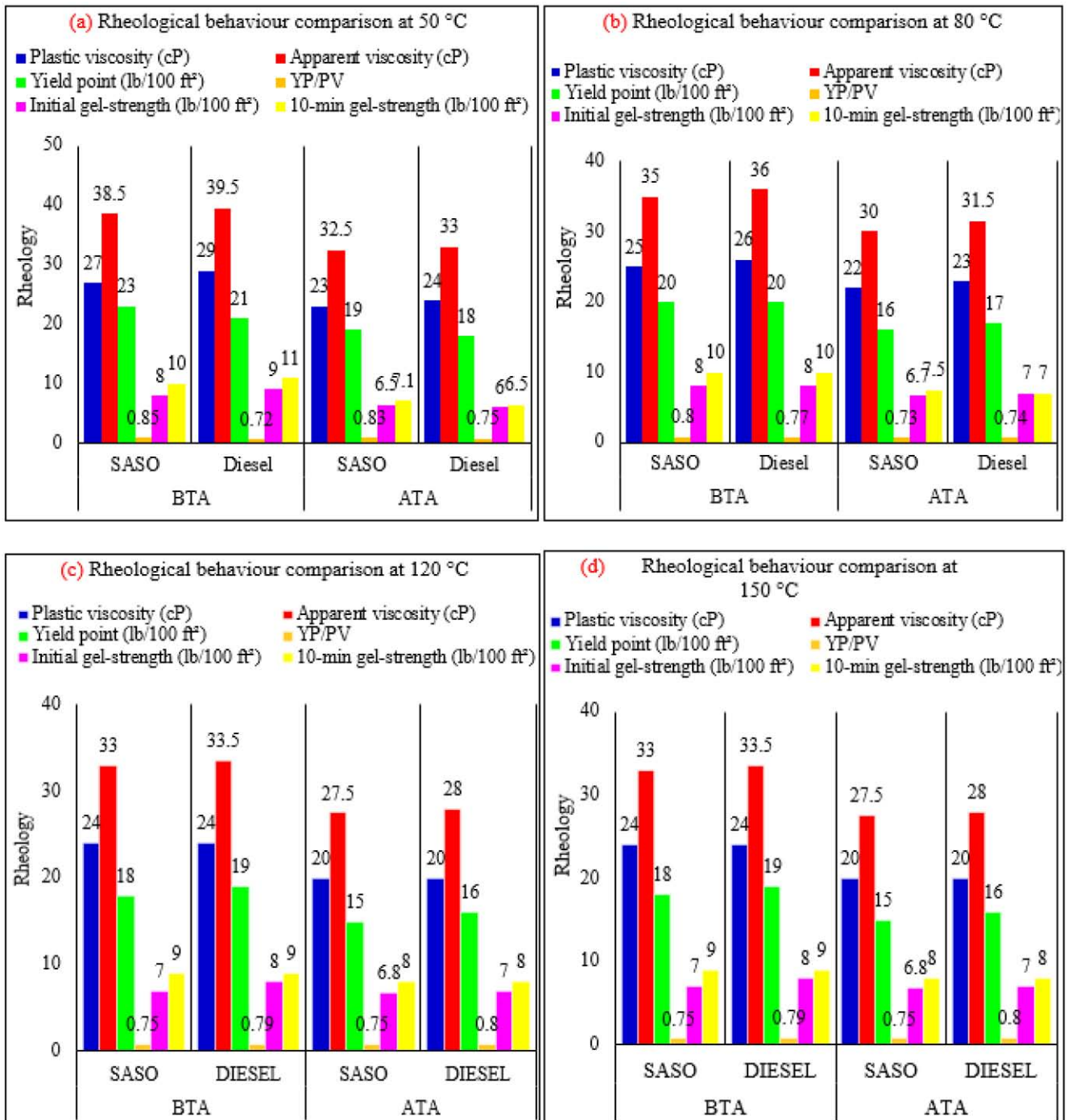


Figure 6—Effect of temperature increment on rheological properties of SASO mud and diesel OBM. BTA=Before thermal aging tests and ATA=After thermal aging tests.

**Apparent and plastic viscosities.** The API recommendations for optimum performance drilling fluid range for the PV is below 35 cP (Table 6). Before thermal aging, the AV and the PV of the biodiesel are minimally lower than those of the diesel as shown in Figures 6(a)-(d), which is attributed to the fatty acids chain length and the existence of carbon double bonds (C18:1; C18:2 and C18:3) (Knothe and Steidley, 2005) in the BBDM. After oven treatment, the viscosities of all the mud samples slightly reduced, which indicates that the fluids are temperature sensitive. For example, at 50 °C in Figure 6(a), the PV of the biodiesel decreased by 14.8% from 27 to 23 cP, while that of the diesel decreased by 17.2% from 29 to 24 cP. Similar trend was observed for the AV of the biodiesel and the diesel, which decreased by 15.6% and 16.5%, respectively.

The reduction in the viscosities of the two IEDMs could be due to the decrease effect of cohesive forces in shear stress at elevated temperature conditions. Van der Waal's intermolecular forces binds the liquid molecular networks and hinders their mobility. The thermal or kinetic energy will increase resulting in increase in liquid mobility once the temperature is high enough; thus, a weak binding force could result to cause decrease in the viscosity (Ahmad *et al.*, 2018).

**Yield point and YP/PV ratio.** The API recommendations for optimum fluid range for the YP is between 15-25 lb/100ft<sup>2</sup> (Table 6). The YP of all the mud samples are within operational range as shown in Figures 6(a)-(d). The results indicate comparable YP and a slightly better ratios of the biodiesel than those of the petrol-diesel. For example, from Figure 6(d), the biodiesel drilling mud has the same YP with the diesel OBM at 16 lb/100ft<sup>2</sup> before oven treatment. After oven treatment, the YPs of the biodiesel and the petrol-diesel were 12.5% and 18.8% lower lower, respectively, signifying that the diesel OBM may be more temperature sensitive. The content of colloidal sizes of organophilic clays interacting with the biodiesel molecules was the reason for the improved YP of the BBDM (Sulaimon *et al.*, 2017; Chilingarian *et al.*, 1983). This shows that the studied BBDM has good transport capacity of drilled cuttings, which might be stronger than that of the diesel OBM. Therefore, the extracted biodiesel could be suitable to be used to enhance cuttings transport capacity and improve hole cleaning efficiency.

**Gel strength.** The gel strengths of all the mud samples as shown in Figures 6(a)-(d) are within the operational recommended range presented in Table 6. Both IEDMs have good gel strengths, and just like the results of other rheological properties, the gel strengths show advantageous biodiesel rheological properties. The two fluids are temperature dependent and their gel strengths tends to reduce with increasing temperature. The reductions indicate a decrease in flocculation of clay particles, which shows a slightly higher values of the petrol-diesel than the biodiesel (Sulaimon *et al.*, 2017). Both IEDMs maintained their thixotropic behaviour suggesting advantageous suspension efficiency of drilled cuttings.

**Filtration properties.** The filtrate loss volume and filter cake thickness results before and after thermal aging tests are shown in Figures 7(a) filtrates volume and (b) filter cake thickness. The filtrate volume and filter cake thickness results of the biodiesel are similar to that of the diesel. The API recommendations for filtrate loss volume is below 4.0 ml and for filter cake thickness is below 2.0 mm (Table 6). The filtrate volume within the temperature of 50 °C to 80 °C before and after thermal aging tests are within the API recommendations limit. The filtrate volume at 120 °C and 150 °C are slightly above the API specified limit. From the reference point acknowledged by the current oil and gas industry, a filtrate loss volume below 10 ml in 30 minutes is typically eligible for drilling operations (Yang *et al.*, 2013). This infers that the two IEDMs can be a good filtration control agent. There are some instances where this set-limits are raised to 15 ml (Yang *et al.*, 2013). The studied biodiesel could have formed a tighter packing structure which results in synergistic interaction between the lipophilic clays (oil-soluble resins) and the surfactants to reduce fluid loss (Sulaimon *et al.*, 2017; Saboori *et al.*, 2018; Oseh *et al.*, 2019a, b).



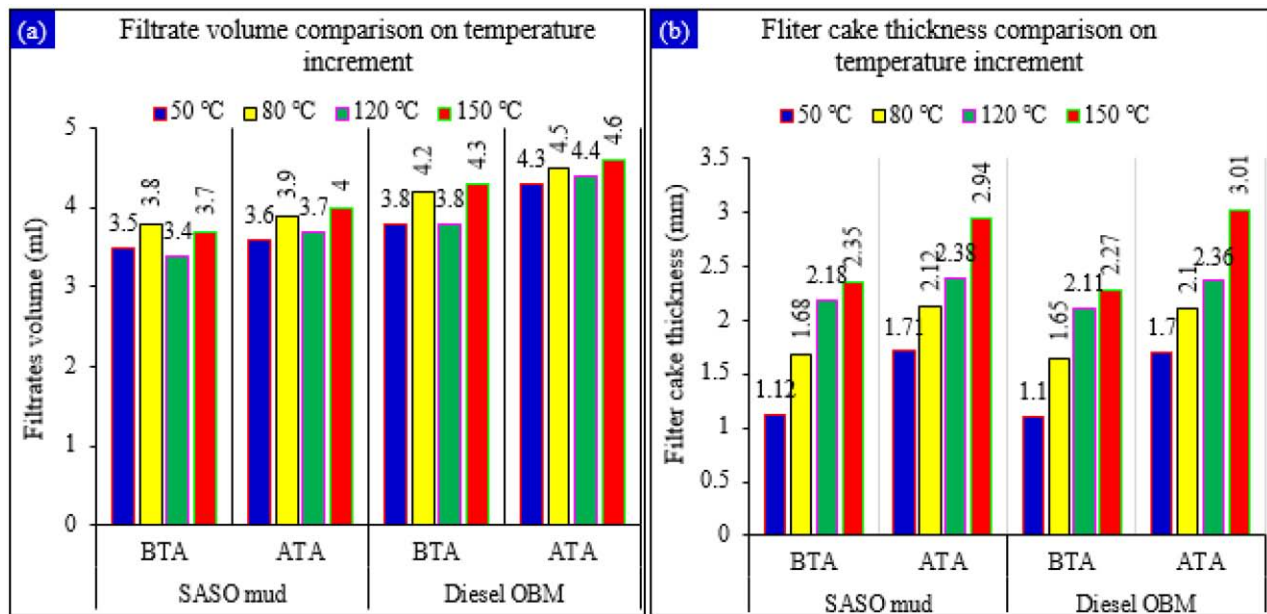


Figure 7—Effect of increasing temperature on (a) filtrate volume and (b) filter cake thickness of SASO mud and diesel OBM

**Electrical stability.** Figure 8 shows the electrical stability (ES) of the two IEDMs at two different temperatures before thermal aging. This property is crucial for non-aqueous drilling fluids. It describes the interface between oil and water while water is being displaced with OBM. The recommended API specification of ES for OBMs is greater than 400 volts (Table 6). The ES of the biodiesel is significantly above the specified limit at the two temperature conditions of 50 °C and 120 °C, and is significantly higher than that of petrol-diesel, which could be due to the ability of the emulsifier associated with the organophilic clay to increase its oil wetting (Growcock *et al.*, 1994). Therefore, the use of SASO as a base oil for BBDM may provide better emulsion stability than the petrol-diesel.

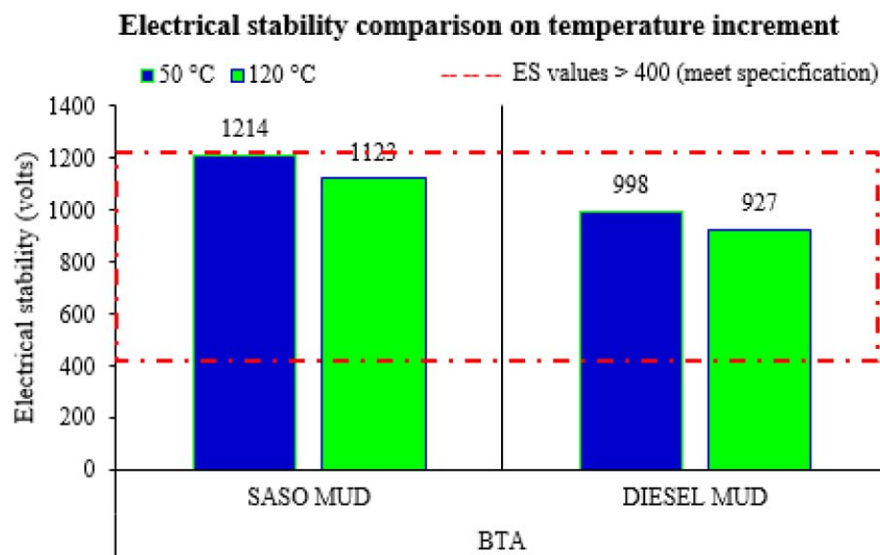


Figure 8—Comparison of SASO mud and diesel mud based on electrical stability

**XRD of Shale samples**

Figure 9 shows the results of three shale samples tested with XRD to select the most amount of smectites/montmorillonites clay minerals. It shows that shale Q was composed of the highest amount of smectites/montmorillonites, making it the most suitable candidate for the shale swelling tests. Most studies used

smectites/montmorillonites to examine shale swelling. These shale cuttings have a high amount of clay, which will make it easy for water molecules to absorb onto them to facilitate shale swelling.

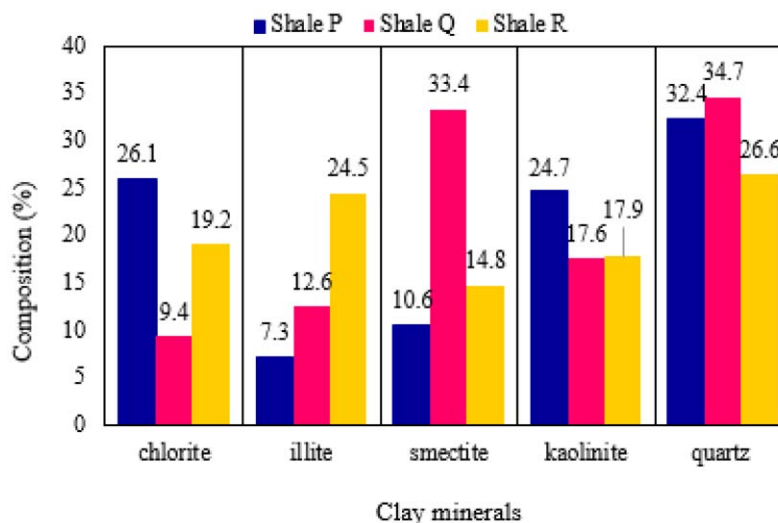


Figure 9—XRD tests results for SASO invert emulsion mud and diesel OBM

### Shale swelling test analysis

The LSM test results for SASO IEDM and diesel OBM are shown in Figure 10. The pellet immersed in SASO mud showed 9.9% of swelling. The BBDM exhibited better inhibition performance with less shale swelling than the petrol-diesel and lower swelling rate in the first 19 hours. The BBDM-immersed pellet started to swell slightly more, reaching swelling saturation point at 9.4%, while the diesel-immersed pellet attained swell-saturation point at 7.6% after 24 hours. The swelling of the immersed pellets for 36 hours showed that the petrol-diesel (8.8%) has slightly better shale swelling inhibition performance than the SASO BBDM (9.9%). The variation of the shale swelling inhibition performance could be due to the variation in molecular structures of the two IEDMs. Biodiesel molecules (made up of fatty acid methyl esters) have larger polarities than those of petrol-diesel because of the carbonyls groups in biodiesels. The obtained data showed that the interaction existing between shale and biodiesel is a little weaker than that between shale and petrol-diesel (Ismail *et al.*, 2014). It can be suggested that the selected biodiesel has a strong inhibition power which is a little weaker than the petrol-diesel.

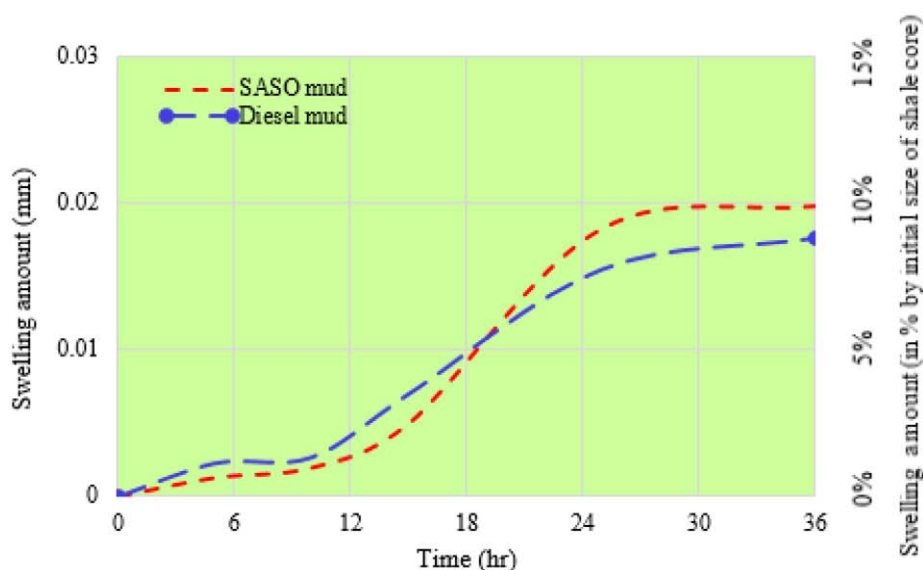


Figure 10—Shale swelling results of SASO BBDM and diesel OBM

### Ecotoxicity analysis

The results of ecotoxicity tests for the biodiesel and diesel are shown in Table 7 following the OECD 301D, (2001) specifications. The results confirm that the BBDM is non-toxic due to its significantly higher lethal concentration 50% ( $LC_{50}$ ) and effective concentration 50% ( $EC_{50}$ ) than those of the petrol-diesel, which could be due to the absence of sulphur and aromatic hydrocarbons in the extracted biodiesel (Li *et al.*, 2016a, b). The biodiesel also meets the specified required limits by OECD 301D, (2001) not met by the petrol-diesel.

Table 7—Ecotoxicities of biodiesel and diesel fuel (test procedure: OECD 301D, 2001).

| Test sample | Test species              | Test methodology | Test specification | Results       |
|-------------|---------------------------|------------------|--------------------|---------------|
| Diesel      | <i>Poecilia latipinna</i> | 72 hr $EC_{50}$  | 10, 000 ppm        | 384.6 ppm     |
|             | <i>Tilapia Guineensis</i> | 96 hr $LC_{50}$  | 10, 000 ppm        | 251.7 ppm     |
|             | <i>Moina mongolica</i>    | 96 hr $LC_{50}$  | 10, 000 ppm        | 498.3 ppm     |
|             | <i>Artemia</i>            | 96 hr $LC_{50}$  | 10, 000 ppm        | 279.2 ppm     |
| SASO        | <i>Poecilia latipinna</i> | 96 hr $LC_{50}$  | 10, 000 ppm        | 28, 287.4 ppm |
|             | <i>Tilapia Guineensis</i> | 96 hr $LC_{50}$  | 10, 000 ppm        | 37,841.5 ppm  |
|             | <i>Moina mongolica</i>    | 96 hr $LC_{50}$  | 10, 000 ppm        | 42, 234.8 ppm |
|             | <i>Artemia</i>            | 72 hr $EC_{50}$  | 10, 000 ppm        | 37, 083.6 ppm |

### Biodegradable analysis

The results of aerobic biodegradability of the BBDM and diesel OBM are shown in Figure 11 following the specifications by OECD 301D, (2001). BBDM showed 78% degradation with *Staphylococcus sp.* and 83% biodegradation with *Penicillium sp.* during 28 days' period of measurement, in contrast to those of petrol-diesel which reached biodegradation of 23.7% with *Staphylococcus sp.* and 25.2% with *Penicillium sp.*

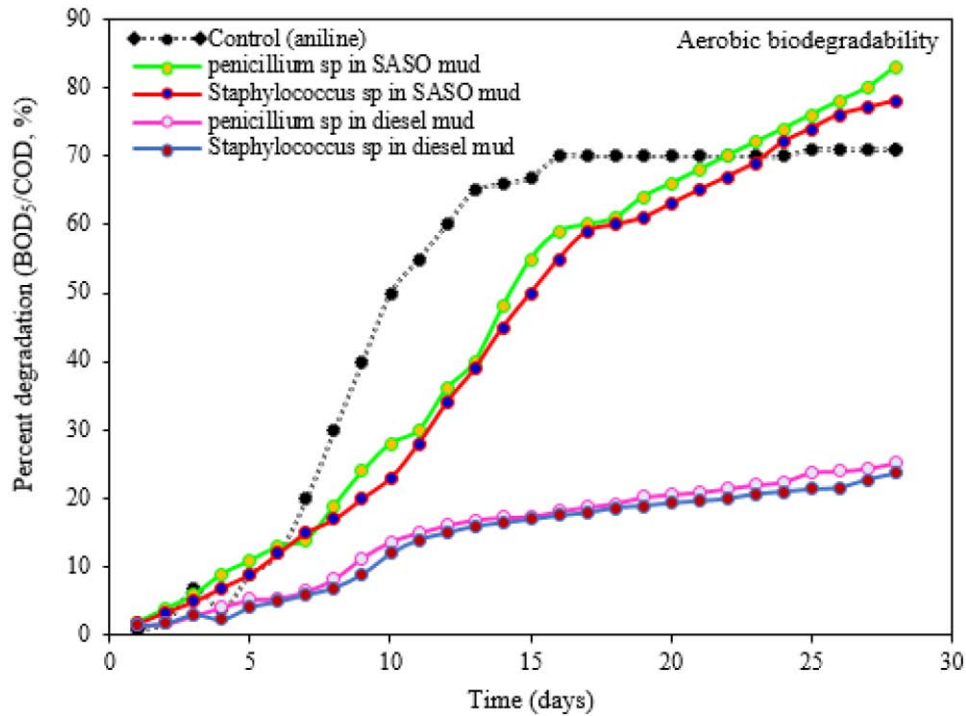


Figure 11—Aerobic biodegradation of BBDM and diesel OBM for 28 days according to OECD 301D (2001). BOD is the biochemical oxygen demand (mg/L) and COD is the chemical oxygen demand (mg/L).

The anaerobic biodegradation of the BBDM and the petrol-diesel are shown in Figure 12 following the specifications by ISO 11734. A total of 60% and 62% of the BBDM with *Staphylococcus sp.* and *Penicillium sp.*, respectively was biodegraded, whereas those of diesel OBM degraded by 29% and 31%, respectively, after 60 days. The molecules of the extracted biodiesel have low branching degrees in their structure and the biodiesel have no aromatic compounds. These enabled the biodiesel to biodegrade very fast, hence, its higher biodegradation than the petrol-diesel (Li *et al.*, 2016a, b). The proposed SASO biodiesel is eco-friendly oil with approval to discharge its effluents owing to its outstanding biodegradability and non-toxicity; Therefore, using the newly-developed biodiesel as a base oil to formulate BBDM may be operationally effective and could be economically viable.



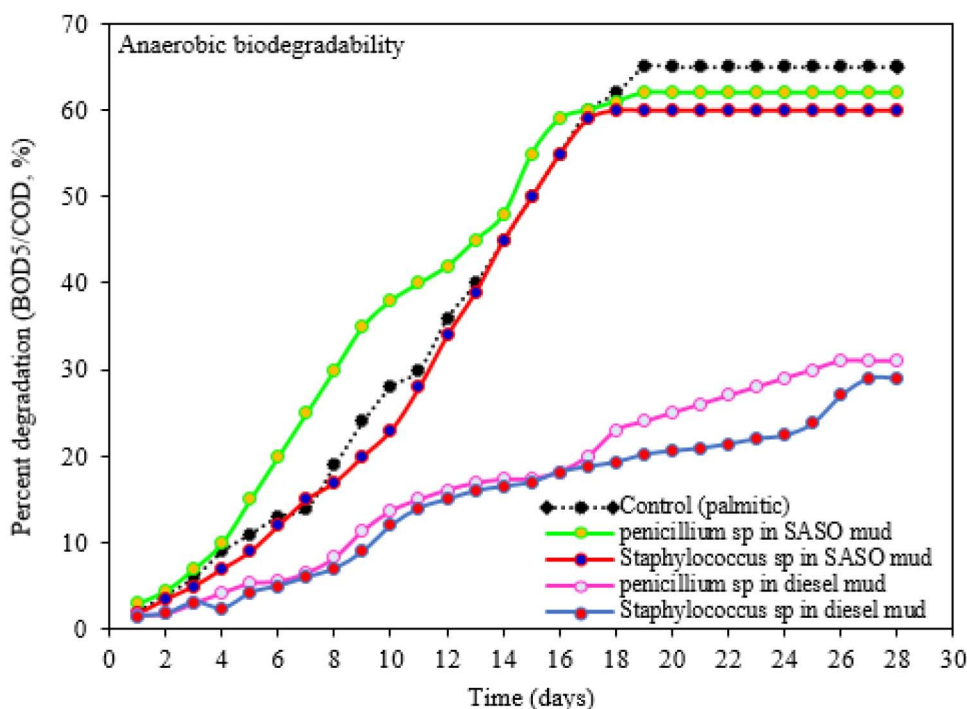


Figure 12—Anaerobic biodegradation of BBDM and diesel OBM for 60 days according to (ISO 11734: R2017). BOD is the biochemical oxygen demand (mg/L) and COD is the chemical oxygen demand (mg/L).

## Conclusions

The following conclusions are drawn from this study:

1. The newly-designed SASO biodiesel was extracted using two steps transesterification method. The extracted biodiesel can be used as a continuous phase of BBDMs due to its good ecological acceptability, good emulsion stability, high flash point, high thermal stability and excellent biodegradability.
2. The cold flow property, rheological behaviour, filtration properties and shale swelling inhibition of the newly-designed SASO biodiesel are all similar to those of diesel; therefore, both oils could be useful to advance engineering design and basic research.
3. The application of the newly-designed SASO biodiesel as a base oil for BBDMs could optimize drilling economics. SASO being a naturally occurring seed oil could be important for drilling contractors and service companies due to its good ecological acceptability.

## Acknowledgements

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## Nomenclature

|      |  |
|------|--|
| API  | American petroleum institute             |
| ASME | American society of mechanical engineers |
| ASTM | American society for testing materials   |
| ATA  | After thermal aging                      |
| AV   | Apparent viscosity                       |
| BBDM | Biodiesel based drilling mud             |

|                  |   |
|------------------|---|
| BOD              | Biochemical oxygen demand                             |
| BTA              | Before thermal aging                                  |
| COD              | Chemical oxygen demand                                |
| EBM              | Ester-based mud                                       |
| EC <sub>50</sub> | Effective concentration 50%                           |
| EN               | European nations                                      |
| ECN              | European Committee for Standardization                |
| ES               | Electrical stability                                  |
| FFA              | Free fatty acids                                      |
| GS               | Gel-strength  |
| HPHT             | High pressure high temperature                        |
| IEDM             | Invert emulsion drilling mud                          |
| ISO              | International organization for standardization        |
| LC <sub>50</sub> | Lethal concentration 50%                              |
| LSM              | Linear swell meter                                    |
| OBM              | Oil-based mud   |
| OECD             | Organization of economic co-operation and development |
| OWR              | Oil-water ratio                                       |
| PME              | Palm methyl ester                                     |
| PV               | Plastic viscosity                                     |
| SASO             | Sweet almond seed oil                                 |
| SASOME           | Sweet almond seed oil methyl ester                    |
| WBM              | Water-based mud                                       |
| XRD              | X-ray diffractometer                                  |
| YP               | Yield point   |

## References

- Abduo, M.I., Dahab, A.S., Abuseda, H., Abdulaziz, M.A., Elhossieny, M.S., (2016). Comparative study of using Water-Based mud containing Multiwall Carbon Nanotubes versus Oil-Based mud in HPHT fields. *Egyptian Journal of Petroleum*, **25**(4), pp.459–464.
- Agwu, O.E., Okon, A.N., Udoh, F.D., (2015). A Comparative Study of Diesel Oil and Soybean Oil as Oil-Based Drilling Mud. *JPE*, **24**, pp.1–9. doi: <http://doi.org/doi:10.1155/2015/828451>.
- Ahmad, H.M., Kamal, M.S., Al-Harhi, M.A., (2018). High molecular weight copolymers as rheology modifier and fluid loss additive for water-based drilling fluids. *J. Mol. Liq.* **252**, pp.133–143.
- Akubude, V.C., Nwaigwe, K.N., (2016). Economic Importance of Edible and Non-Edible Almond Fruit as Bioenergy Material: A Review. *American Journal of Energy Science*, **3**(5), pp.31–39 <http://www.openscienceonline.com/journal/energy>.
- Amin, R.A.M, Clapper, D.K., Norfleet, J.E. et al., (2010). Joint Development of an Environmentally Acceptable Ester-based Drilling Fluid. Presented at the Trinidad and Tobago Energy Resources Conference, Port of Spain, Trinidad, 27-30 June. SPE-132693-MS. DOI: [10.2118/132693-MS](https://doi.org/10.2118/132693-MS).
- API RP 13B-2., 2005. *Recommended Practice for Field Testing Oil-based Drilling Fluids*.
- Balat, M., Balat, H., (2010). Progress in biodiesel processing. *Applied Energy*, **87**(6), pp.1815–1835
- Benjumea, P., Agudelo, J, Agudelo, A., 2008. Basic properties of palm oil biodiesel–diesel blends. *Fuel*, **87**, pp. 2069–75.
- Boyou, N.V., Ismail, I., Sulaiman, W.R.W, Haddad, A.S., Hussein, N., Heah, T.H., Nadaraja, K., (2019). Experimental Investigation of Hole Cleaning in Directional Drilling by Using Nano-Enhanced Water-based Drilling Fluids. *Journal of Petroleum Science and Engineering*, **176**, pp.220–231.
- Burrows, K., Evans, J., Hall, J., et al., (2001). New low viscosity ester Is suitable for drilling fluids in deep water applications. Presented at the SPE/EPA/DDE Exploration and Production Environmental Conference, SAN Antonio, Texax, 26-28 February, SPE-66553-MS.
- Caenn, R., Darley, H.C.H., Gray, G.R., (2017). Introduction to Drilling Fluids. In: *Composition and Properties of Drilling and Completion Fluids*, pp.1–34.

- Chilingarian, G.V., Alp, E., Al-Salem, M., Uslu, S., Gonzales, S., Dorovi, R.J., (1983). Drilling Fluid Evaluation Using Yield Point-Plastic Viscosity Correlation. *Society of Petroleum Engineers*.
- Demibras, A., (2007). Importance of biodiesel as transportation fuel. *Energy Policy*, **35**, pp.4661–4670, <http://dx.doi.org/10.1016/j.enpol.2007.04.003>.
- Dosunmu, A., Ogunrinde, J., (2010). Development of Environmentally Friendly Oil Based Mud using Palm Oil and Groundnut Oil. SPE 140720. Paper presented at the 34th Annual International Conference and Exhibition, 31 July-3 August, Tinapa-Calabar, Nigeria. **28**, pp. 201–213.
- Eckhout, D., Dolon, S., Gozan, P., et al., (2000). Developmental process and field applications of a new ester-based mud system for ERD wells on Australia's Northwest shelf. Presented at the IADC/SPE Asia Pacific Drilling Technology, Kuala Lumpur, 11-13 September, SPE-62791-MS.
- European Committee for Standardization., 2003. *ECN EN14214, fatty acid methyl esters (FAMES) for diesel engines-requirements and test methods, Brussels*.
- Fakharany, T.E., Khaled, R., Mahmoud, A., (2017a). Formulating Environmentally Friendly Oil- Base Mud using Jatropa Oil. *Iarjset*. **4**, pp.1–6. doi:10.17148/IARJSET.2017.4101.
- Fakharany, T.E., Khaled, R., Mahmoud, A., (2017b). Formulating Environmentally Friendly Oil- Base Mud using Soybean Oil. *Iarjset*. **4**, pp.1–6. doi:10.17148/IARJSET.2017.4101.
- Fattah, K.A., Lashin, A., (2016). Investigation of mud density and weighting materials effect on drilling fluid filter cake properties and formation damage. *Journal of African Earth Science*. **117**, pp.345–357. doi:10.1016/j.jafrearsci.2016.02.003.
- Fink, J., (Ed.), (2015). Chapter 1 - Drilling muds, in: *Petroleum Engineer's Guide to Oil Field Chemicals and Fluids* (Second Edition). Gulf Professional Publishing, Boston. pp.1–61. doi:10.1016/B978-0-12-803734-8.00001-1.
- Gbadamosi, A.O., Junin, R., Abdalla, Y., Agi, A., Oseh, J.O., (2018a). Experimental investigation of the effects of silica nanoparticle on hole cleaning efficiency of water-based drilling mud. *Journal of Petroleum Science and Engineering*. **172**, pp.1226–1230.
- Gbadamosi, A.O., Junin, R., Oseh, J.O., et al., (2018b). Improving hole cleaning efficiency using nanosilica in water-based drilling muds. SPE-193401-MS. Nigeria Annual International Conference and Exhibition, 6-8 August, Lagos, Nigeria, **21**, pp.1–16.
- Giwa, S., Layeni, A., Ogunbona, C., (2012). Synthesis and characterization of biodiesel from industrial starch production byproduct. *Energy and Environmental Engineering Journal*, **1**, pp.45–51
- Giwa, S., Ogunbona, C., (2014). Sweet almond (*Prunus amygdalus "dulcis"*) seeds as a potential feedstock for Nigerian Biodiesel Automotive Project. *Rev. Ambient. Agua*. **9**, pp.445–458. <https://doi.org/10.4136/1980-993X>.
- Giwa, S.O., Chua, L.A., Adam, N.M., (2010). Investigating "Egusi"(*Citrullus Colocynthis L.*) Seed oil as potential biodiesel feedstock. *Energies*. **3**, pp.607–618.
- Hakim, H., Katende, A., Farad, S., Ismail, A., Nsamba, H., (2018). Performance of polyethylene and polypropylene beads towards drill cuttings transportation in horizontal wellbore. *Journal of Petroleum Science and Engineering*, **165**, pp.962–969.
- Hasan, M.M., Rahman, M.M., 2017. Performance and emission characteristics of biodiesel-diesel blend and environmental and economic impacts of biodiesel production: A review. *Renew. Sustain. Energy Rev.*, **74**, pp. 938–948.
- Ismail, A.R., Kamis, A., Foo, K.S., (2001). Performance of the Mineral Blended Ester Oil-Based Drilling Fluid Systems, *University Teknologi Malaysia*. <http://dx.doi.org/10.2118/2001-044>.
- Ismail, A.R. Jaafar, M.Z., Wan, R., Wan, S., Ariff, R., (2015). Shale Swelling Mitigation in Water-Based Drilling Fluids with Amine Additives. 8th International Conference on Geological and Geo-Resources Engineering, Penang, Malaysia, **35**, pp.23–31.
- Ismail, A.R., Aftab, A.A., Ibupoto, Z.H., Zolkifile, N., (2016). The novel approach for the enhancement of rheological properties of water-based drilling fluids by using multi-walled carbon nanotube, nanosilica and glass beads. *Journal of Petroleum Science and Engineering*, **139**, pp. 264–275.
- Ismail, A.R., Ismail, N.J., Jaafar, M.Z., et al., (2014). The application of biodiesel as an environmental friendly fluid to drill oil and gas wells. Presented at the 5th International Sriwijaya seminar on Energy and Environmental Science and Technology, Palembang, Indonesia, 10-11 Sept. **04**, pp. 1–9.
- ISO 11734/TC., (2017). ISO/DIS 10253, Water Quality-Marine algae growth inhibition test with *Skeletonema costatum* and *Phaeodactylum tricoratum*.
- Izah, S.C., Ohimain, E.I., (2013). The Challenge of Biodiesel Production from Oil Palm Feedstock in Nigeria. *Greener Journal of Biological Sciences*, **3**, pp.1–12.
- Kafashi, S., Rasaei, M., Karimi, G., (2017). Effects of sugarcane and polyanionic cellulose on rheological properties of drilling mud: An experimental approach. *Egypt. J. Pet.* **26**, pp.371–374. <https://doi.org/https://doi.org/10.1016/j.ejpe.2016.05.009>.

- Knothe, G., Steidley, K.R., 2005. Kinematic viscosity of biodiesel fuel components and related compounds. influence of compound structure and comparison to petrol diesel fuel components. *Fuel*, **84**, pp. 1059–1065.
- Kurnia, J.C., Jangam, S.V., Akhtar, S., Sasmito, A.P., Mujumdar, A.S., (2016). Advances in biofuel production from oil palm and palm oil processing wastes: A review. *Biofuel Res. J.*, **9**, 332–346.
- Li, W., Liu, J., Zhao, X. et al., (2018). Novel Modified Rectorite Provides Reliable Rheology and Suspend ability for Biodiesel Based Drilling Fluid. Presented at the SPE/IADC Middle East Drilling Technology Conference and Exhibition, Abu Dhabi, UAE, 29-31 January. SPE-190830-MS. DOI: [10.2118/190830-MS](https://doi.org/10.2118/190830-MS).
- Li, W., Zhao, X., Ji, Y. et al., (2016a). Investigation of Biodiesel-Based Drilling Fluid, Part 1: Biodiesel Evaluation, Invert-Emulsion Properties, and Development of a Novel Emulsifier Package. *SPE Journal*, **21**(5), pp.1755–1766. SPE-180918-PA. DOI: [10.2118/180918-PA](https://doi.org/10.2118/180918-PA).
- Li, W., Zhao, X., Ji, Y. et al., (2016b). Investigation of Biodiesel-Based Drilling Fluid, Part 2: Formulation Design, Rheological Study, and Laboratory Evaluation. *SPE Journal*, **21**(5), pp.767–1781. SPE-180926-PA. DOI: [10.2118/180926-PA](https://doi.org/10.2118/180926-PA).
- Moser, B.R., Vaughn, S.F., (2010). Evaluation of alkyl esters from *Camelina sativa* oil as biodiesel and as blend components in ultra-low-sulfur diesel fuel. *Bioresour. Technol.*, **101**, pp. 646–653. <http://dx.doi.org/10.1016/j.biortech.2009.08.054>.
- Nwosu, F.O., Dosumu, O.O., Okocha, J.O.C., (2008). The Potential of Terminalia Catappa (Almond) and Hyphaene Thebaica (Dum Palm) Fruits as Raw Materials for Livestock Feed. *African Journal of Biotechnology*, **7**(24), pp.4576–4580.
- OECD, Chemical Group., (2001). *Ready biodegradability: Closed Bottle Respirometric Test Procedures 301D, OECD revised guidelines for tests for ready biodegradability, Paris, France*
- Ogunsuyi, H.O., Daramola, B.M., (2013). Evaluation of Almond (*Prunus Amygdalus*) Seed Oil as a Viable Feedstock for Biodiesel Fuel. *International Journal of Biotechnology Research*, **1**(8), pp. 120–127.
- Oseh, J.O., Gbadamosi, A.O., Ogunyemi, A., Omotara, O.O., (2018). Transports of Different Cuttings Sizes in A Wellbore using Henna and Lignite Materials. *Journal of Engineering Research and Development (AJERD)*, **3**, pp.351–365.
- Oseh, J.O., Norddin, M.N.A., Farooq, F., Ismail, A.R., Ismail, I., Gbadamosi, A.O., Agi, A., (2019a). Experimental investigation of the effect of henna leaf extracts on cuttings transportation in highly deviated and horizontal wells. *Journal of Petroleum Exploration and Production Technology*, **5**, pp.1–18. <https://doi.org/10.1007/s13202-019-0631-z>
- Oseh, J.O., Norddin, M.N.A., Ismail, I., Ismail, A.R., Gbadamosi, A.O., Agi, A., (2019b). Effect of the surface charge of entrapped polypropylene at nanosilica-composite on cuttings transport capacity of water-based muds. *Applied Nanoscience*, DOI: [10.1007/s13204-019-01063-9](https://doi.org/10.1007/s13204-019-01063-9).
- Pantoja, S.S., Da Conceição, L.R.V., Da Costa, C.E.F., Zamian, J.R., Da Rocha Filho, G.N., (2013). Oxidative stability of biodiesels produced from vegetable oils having different degrees of unsaturation, *Energy Conversion and Management*, **74**, pp. 293–298.
- Paswan, B.K., Jain, R., Sharma, S.K., Mahto, V., Sharma, V.P., (2016). Development of Jatropha oil-in-water emulsion drilling mud system. *Journal of Petroleum Science Engineering*. **144**, pp.10–18. <https://doi.org/https://doi.org/10.1016/j.petrol.2016.03.002>.
- Peresich, R.L., Burell, B.R., Prentice, G.M., (1991). Development and field trials of a biodegradable invert emulsion fluid. Presented at the SPE/IADC drilling conference, Amsterdam, 11-14 March. SPE-21935-MS. pp.46–54
- Priyanko, P.G., Debashis, S., Shashi, D.B., Rupam, K., (2018). Biodiesel production from tea seed oil. AIP Conference Proceedings. **1998**, pp.1–14.
- Saboori, R., Sabbaghi, S., Kalantarias, A., Mowla, D., (2018). Improvement in filtration properties of water-based drilling fluid by nanocarboxymethyl cellulose/polystyrene core-shell nanocomposite. *Journal of Petroleum Exploration and Production Technology*. **21**, pp.43–52.
- Sayindla, S., Lund, B., Ytrehus, J.D., Saasen, A., (2017). Hole-cleaning performance comparison of oil-based and water-based drilling fluids. *Journal of Petroleum Science Engineering*, **159**, pp.49–57. <https://doi.org/https://doi.org/10.1016/j.petrol.2017.08.069>.
- Smith, S.R., Rafati, R., Haddad, A.S., Cooper, A., Hamidi, H., (2018). Application of aluminium oxide nanoparticles to enhance rheological and filtration properties of water-based muds at HPHT conditions. *Colloids and Surfaces A: Physio-chem. Eng. Aspects*, **537**, pp.361–371.
- Sulaimon, A.A., Adeyemi, B.J., Rahimi, M., (2017). Performance enhancement of selected vegetable oil as base fluid for drilling HPHT formation. *Journal of Petroleum Science Engineering*, **152**, pp.49–59. <https://doi.org/10.1016/J.PETROL.2017.02.006>.
- Sun, M.B., Qiao, J., Liu, B.F. et al., (2013). Research and Application of Biodiesel-based Drilling Fluid. *Drilling Fluid & Completion Fluid*, **30**(4), pp.15–18.



- Urrutia, C., Sangaletti-Gerhard, N., Cea, M., Suazo, A., Aliberti, A., Navia, R., (2016). Two step esterification–transesterification process of wet greasy sewage sludge for biodiesel production. *Bioresource Technology*. **200**, pp. 1044–104.
- Verma, P., Sharma, M.P., Dwivedi, G., (2016). Evaluation and enhancement of cold flow properties of palm oil and its biodiesel. *Energy Reports* **2**:8–13.
- Wakil, M.A., Kalam, M.A., Masjuki, H.H., Atabani, A.E., Rizwanul-Fattah, I.M., (2015). Influence of biodiesel blending on physicochemical properties and importance of mathematical model for predicting the properties of biodiesel blend. *Energy Conversion and Management*, **94**, pp.51–67.
- Wang, L., (2013). Properties of Manchurian apricot (*Prunus mandshurica* Skv.) and Siberian apricot (*Prunus sibirica* L.) seed kernel oils and evaluation as biodiesel feedstocks. *Ind. Crops Prod.*, vol. **50**, pp.838–843. <http://dx.doi.org/10.1016/j.indcrop.2013.08.072>.
- Wang, M., Sun, M., Shang, H. et al., (2012). Biodiesel-based Drilling Fluids. Presented at the IADC/SPE Asia Pacific Drilling Technology Conference and Exhibition, Tianjin, China, 9-11 July. SPE-155578-MS. DOI: [10.2118/155578-MS](https://doi.org/10.2118/155578-MS).
- Wang, S., Yuan, H., Wang, Y., Shan, R., (2017). Transesterification of vegetable oil on low cost and efficient meat and bone meal biochar catalysts. *Energy Conversion and Management*. **150**, pp.214–221.
- Yadav, M., Singh, V., Sharma, Y.C., (2017). Methyl transesterification of waste cooking oil using a laboratory synthesized reusable heterogeneous base catalyst: Process optimization and homogeneity study of catalyst. *Energy Conversion and Management*. **148**, pp.1438–1452. <https://doi.org/https://doi.org/10.1016/j.enconman.2017.06.024>.
- Yang, J., Xu, T.T., Wu, X.X., Pan, X.Y., Hou, R.X., (2013). The studies of biodiesel drilling fluids. *Drill Fluid Completion Fluid*. **30**, pp. 15–18.
- Yeu, W.J. Katende, A., Sagala, F., Ismail, I. (2019). Improving hole cleaning using low density polyethylene beads at different mud circulation rates in different hole angles, *Journal of Natural Gas Science and Engineering*, **61**, pp.333–343.
- Zhong, W., Xuan, T., He, Z., Wang, Q., Li, D., Zhang, X., Huang, Y.Y., (2016). Experimental study of combustion and emission characteristics of diesel engine with diesel/second-generation biodiesel blending fuels. *Energy Convers. Manag.* **121**, pp.241–250.