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The rheological and filtration properties of black seed (*Nigella Sativa* L.) ester as a base fluid in drilling fluid

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Abstract

The unceasing utilization of diesel oil, the regular base fluid, as the base fluid of drilling mud has brought about severe ecological worries and regulation because of its poisonousness, non-biodegradability, and similarity issues. Notwithstanding, drilling more complex wells is turning out to be progressively significant in satisfying the world's need for oil and gas, bringing about increased commercial synthetic-based fluid importation. In light of the ecological worries associated with the usage of diesel-based drilling fluids as well as reducing commercial synthetic-based fluid imports, black seed oil (BSO) was utilized to make an ester for a new ecologically safe drilling fluid. This research explored the potential use of black seed ester in formulating oil-based muds with standard additives. It compares its qualities to that of diesel following a thorough mud check on the mud samples to evaluate the rheological properties and the impact of aging at test temperatures of 86°F, 120°F, and 150°F. Fourier transform infrared (FTIR) spectrometry technique affirmed that BSO ester is a natural compound. About the flow properties, the kinematic thickness at 40 °C of the biodiesel is 4.31 mm²/s and is higher than that of petroleum diesel, which is 3.52 mm²/s. Densities for the ester and diesel oil-based mud tests were 7.9 ppg, 7.8 ppg, 7.3 ppg, and 6.8 ppg, respectively, at 70/30, 75/25, and 80/20, oil–water-proportion (OWR), as utilized in the mud formulation. The prepared mud was aged for 24 h under static conditions to guarantee total hydration. Black seed oil (BSO) ester mud exhibited lower viscosity at all temperatures, aging conditions, and shear rates analyzed, making it more suitable for oil-well drilling fluids when compared with Diesel. BSO being a naturally occurring seed oil could be important for drilling contractors and service companies due to its good ecological acceptability and its applicability.

Keywords: Biodegradation, Black cumin seed oil, Diesel, Methyl ester/biodiesel, Oil-based mud, Rheological properties

Introduction

The first and most important step in the progress of petroleum exploration and production activities is drilling the oil and gas well and selecting drilling fluids, which additionally incorporates mixing and utilizing drilling fluids that must be safe to use, economically viable, and meet technical parameters [1, 2]. To meet the growing demand

for hydrocarbons, more complex and technical wells have been continuously drilled in difficult environments in recent years, bringing about a critical interest in multifunctional drilling fluids [3]. Concerns about the environment are driving the development and use of synthetic fluids [4]. Given that the marine environment is a pioneering prospect for hydrocarbon exploration [5, 6], there is a strong incentive to keep an economic-ecological balance in this process [7].

The base fluid is a major component of drilling mud. It comes in various forms, and the drilling conditions determine its application [8]. Oil-based fluids, the most commonly used base components as the external phase in oil-based drilling muds, OBM, are made of diesel or mineral oil (petroleum-based oils), mineral oil, and low toxicity mineral oil which are used due to their viscosity, low flammability, and low rubber solvency [9, 10]. The recycling and release of these materials are, nevertheless, subject to severe environmental regulations [11–13]. SBMs are oil-based mud variants that combine the desirable operating characteristics of OBM with the lower toxicity and environmental impact of water-based mud [8]. Its base fluid is made of non-water-soluble organic compounds, and neither the base fluid nor the additives are petroleum-based [14].

The environmental movement to use biodegradable oils has been forward-thinking [4, 15, 16]. The approval to discharge drilling cuttings directly into the sea, which is an unacceptable practice for OBMs and some water-based fluids in many areas, has been the primary motivator [17]. The findings by [6] show that *Jatropha*-based drilling mud outperformed the conventional diesel-based mud in terms of thermal stability when compared. Udeagbara et al. [18] show that castor oil-based drilling mud has a lower pH value, plastic viscosity (PV), and gel strength (GS) than diesel-based mud. According to the rheological and lubricity test results, neem oil biodiesel-based drilling muds are comparable to conventional diesel-based drilling muds [12].

Chrysothamnus albidum seeds are the least toxic and biodegradable, making them more environmentally friendly [19]. Adesina et al. [7] used *Jatropha* oil to create an environmentally friendly oil-based mud. Viscosity modifiers and reducers were used. It outperforms diesel-based mud in terms of efficiency and frictional pressure losses.

Walnut and soya bean-based drilling mud perform comparably to commercial synthetic oil-based drilling mud purchased from industry, as demonstrated by [20]. The soya and walnut cakes' filtration volumes and cake thicknesses suggested that they had superior filtering qualities than Diesel OBM. Adesina et al. [20] discovered that walnut and soya bean-based muds perform similarly to industrial synthetic OBMs. Bean plants fed synthesis-based mud grew faster and were less toxic than beans fed diesel OBMs.

Li et al. [21] discovered that waste cooking oil had a higher viscosity at low temperatures, limiting its use in deep water or cold environments. Sauki et al. [15] demonstrated that ester can be used as an alternative drilling fluid because its rheological properties are not significantly different from those of sarapar-based mud, which is commonly used in the drilling industry. Adewale et al. [22] proposed using additive chemistry in its formulation. Amorin et al. [5] discovered that using appropriate and combined antioxidants revealed the potential geothermal stability of the vegetable oils (esters) to withstand the first tier of HPHT environments. *Calophyllum* oil has the potential to be a drilling mud base oil, according to [23]; however, plant base mud would require more pump pressure and cost to start flowing than commercial mud. According to [24],

thinner must be used in the mud formulation with rubber seed oil to improve its cutting suspension ability. Oseh [25] show that the rheology, filtration properties, electrical stability, thermal stability, and shale swelling inhibition performance of the almond seed oil-based drilling mud are comparable to that of the diesel OBM and its low branching degree and lack of aromatic compounds contribute to its high biodegradability.

The novelty of this research was to evaluate the potential of locally derived vegetable oil for the improvement of mud rheology and filtration in comparison with some other vegetable and conventional oils. In this study, the continuous phase was black seed oil (BSO), vegetable oil extracted from its seeds that was trans-esterified into an ester and used to create synthetic-based drilling mud for use in drilling operations. Because these properties are so important to overall drilling mud performance, the BSO ester mud was tested for basic properties, rheology, filtration characteristics, electrical stability, and biodegradability. The results were discussed, as was their performance in comparison to previous work on synthetic-based mud variants.

Materials and methods

Materials

Matured black seeds were obtained from Oje market in Ibadan, Nigeria, and thoroughly cleaned to get rid of any foreign objects like garbage and dirt and were sun-dried.

The base oil for the diesel-OBM formulation was commercial-grade diesel fuel obtained from a local supplier (Bovas filling station, Ibadan, Nigeria). The conventional diesel also satisfies American Society for Testing Materials (ASTM) Grade II diesel standards (ASTM D975, Grade II-DS500). All of the chemicals used (n-hexane, sodium hydroxide (NaOH), sulphuric acid (H_2SO_4), and methanol) were of analytical grade, and the equipment used was from the University of Ibadan's Petroleum Engineering Laboratory.

Useful apparatus and their model

The following apparatus was used in the experiment:

Manual Press Model 304, MAXWXKING, was used in carrying out black seed oil extraction; Water bath WB series standard model, 12 l, WB-12 was used to heat BSO after extraction; a universal Hydrometer (HYDROMETER-1000–2000, OMSONS) was used to measure the specific gravity of the base oils; Seta Compact Cloud and Pour Point Cryostat – 94,100–4 was used to carry out the Cloud and pour point analysis; OFITE Emulsion Tester – 131–50 was used for emulsion stability test, 78–1 Magnetic Stirrer Hot Plate for heating and stirring BSO before and after transesterification; and Bruker ALPHA-II-FTIR-Spectrometer analyzed the functional groups present in the ester.

Seta flash Series3 Flashpoint Tester – 33,200–3 characterized the flash and fire point of the ester; Baroid Mud balance – E037-01 was used to determine the mud density; OFITE Filter Press – 140–30 deduced the filtration properties at high temperature and pressure; OFITE Multimixer – 9B was employed to mix the mud components into a single mix; and OFITE 8-Speed Rotational Viscometer – 800 analyzed the rheological properties of the mud samples.

Procedures for the production of BSO ester

The flow process used to utilize the production and properties performance of BSO ester as a base fluid for drilling is shown in Fig. 1.

Oil extraction

Twenty-eight kilograms of black seeds were sun-dried for 48 h. Then, it was sterilized for 120 min between room temperature and 50 °C under air pressure to create substantially sterilized black seeds. They were manually pressed using a hand press (Manual Press Model 304, MAXWXKING) to extract the oil with low heat and no chemicals. The resulting cold-pressed black seed oil, BSO was then produced at temperatures ranging from room temperature to 60 °C and pressures ranging from 0 to 70 bars.

Filtration was carried out on the oil to remove shafts and sludge. Cold pressing is an effective process for obtaining black seed oil from its seeds. Cold pressing technology is beneficial [26] because it is environmentally friendly and an organic method. It keeps the healthy BSO’s linoleic acid and oleic acid and important fatty acids as well as their natural enzymes, higher level of nutrients, and other beneficial elements. The oil extraction yield was calculated based on the weight of obtained oil after pressing and the weight of black cumin seed as follows:

$$\text{Extraction yield\%} = \frac{\text{Extracted oil amount (g)}}{\text{Seed weight (g)}} \times 100 \tag{1}$$

Esterification process of black seed oil (from alcohol and carboxylic acid)

According to previous studies, a transesterification technique was employed to manufacture the ester [25–27]. Figure 2 shows the transesterification flow process of BSO ester. BSO was transformed into fatty acid methyl ester (FAME) using methanol. Because the product and glycerol can be separated simultaneously during the process, using methanol in the transesterification procedure has advantages. The by-products of

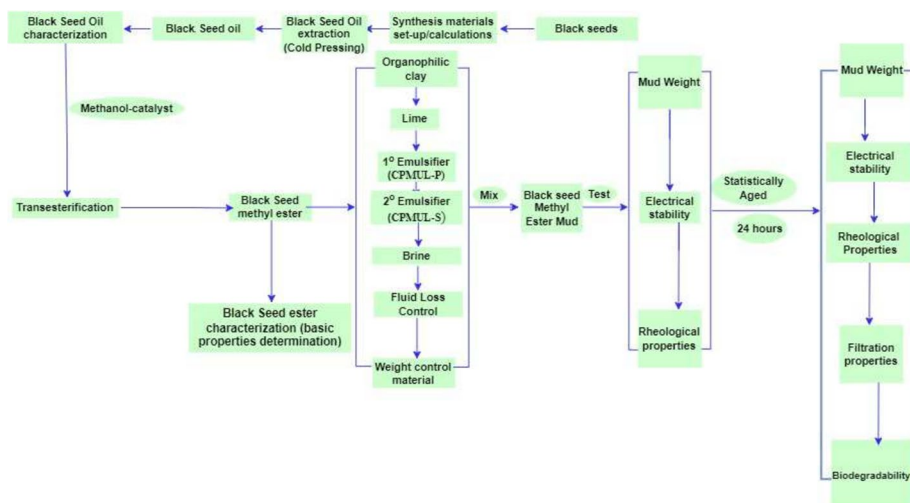


Fig. 1 Flow process used to utilize the production and properties performance of BSO ester as a base fluid for drilling

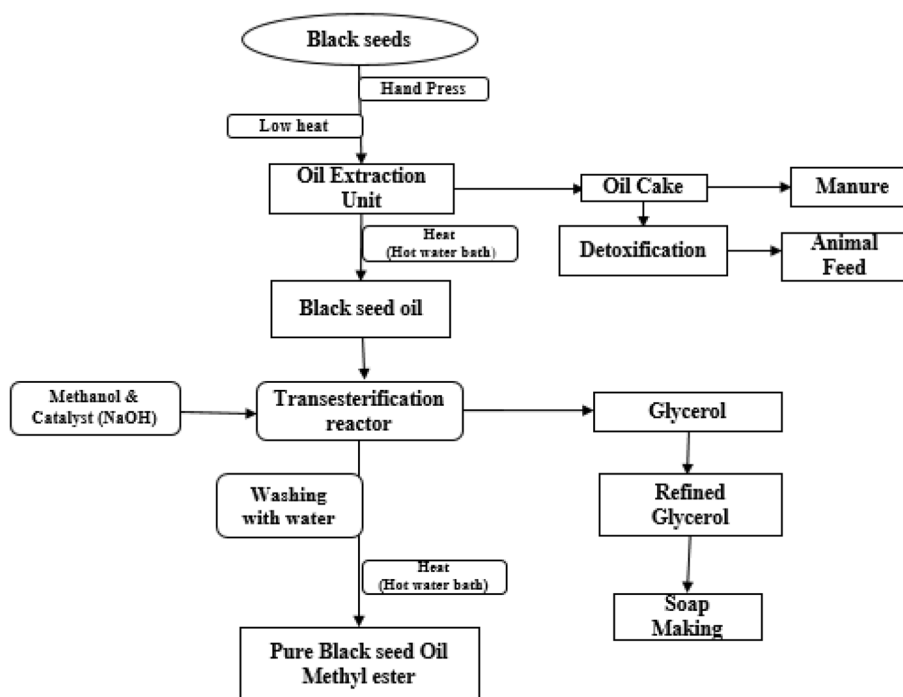


Fig. 2 Transesterification process of black seed oil into black seed ester

transesterification are raw glycerol and BSO ester [28]. Using an alkali catalyst, BSO was trans-esterified to produce the esters used in this study. A 140°F temperature was used for the experiment [29].

In a 2000cm³ beaker with a magnetic stirrer, 4 Litres of black seed oil, and 5.5 Litres of methanol were combined. Before being added to the oil, 10 g of NaOH was dissolved in methanol and vigorously stirred for 60 min at 60 °C at 150 rpm (revolutions per minute). As seen in Fig. 3, the mixture was left in place to let gravity deposit the glycerol at the beaker’s bottom. After the separation was finished, the upper ester layer (BSO methyl ester) was transferred to a new beaker. To remove any remaining methanol and water from the catalyst neutralization reaction, the ester was then heated to 230°F in the water bath [28]. Fatty acid methyl ester content was calculated using the EN-14103 method. The formula below is used to compute the % yield of final biodiesel after eliminating glycerol, catalyst, contaminants, methanol, and water. The mass of biodiesel is the sum of the masses of oil and alcohol, less the masses of glycerol and unreacted compounds.

$$\text{Yield of BSO ester (\%)} = \frac{\text{Mass of methyl ester obtained (g)}}{\text{Mass of the BSO Biodiesel used (g)}} \times 100 \quad (2)$$

Water washing

Here, excess catalyst, glycerol, and other impurities that might encourage further reaction are removed from the BSO ester by washing it with water at a concentration of 30% of the ester volume. After gently swirling the water for 10 min into the ester, it was allowed to settle for 6 h. Figure 4 depicts the decantation and secure storage of the BSO’s crystal-clear upper-layer ester derivatives in a glass bottle.

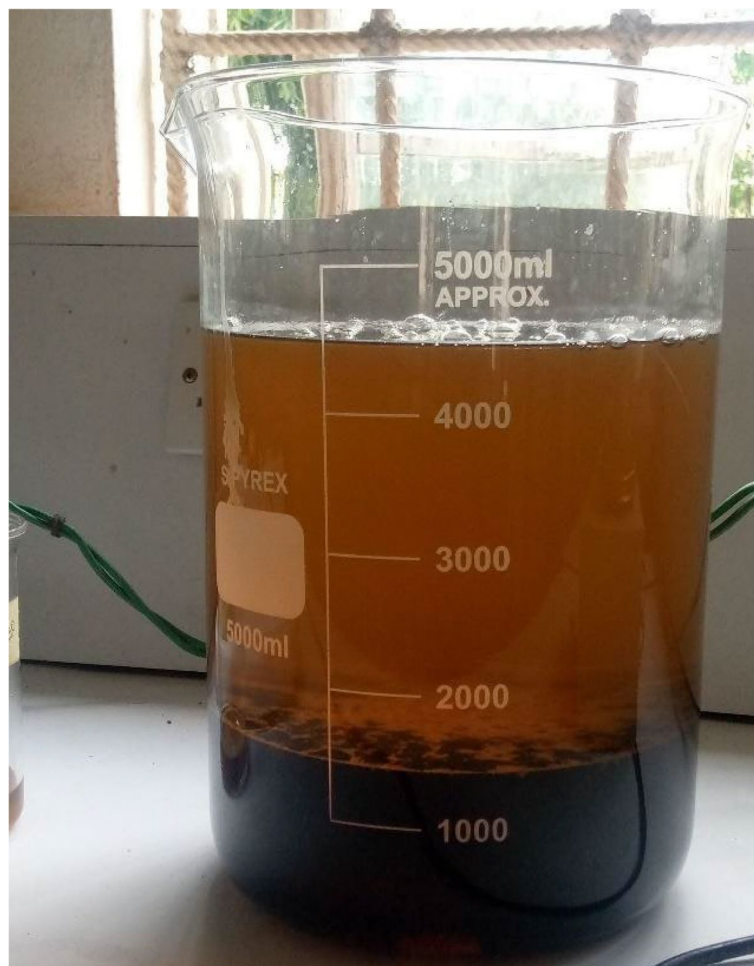


Fig. 3 Phase separation of the BSO ester and its glycerol

Functional analysis of BSO ester

This entails analyzing the produced BSO ester qualitatively using FTIR. The FTIR (ALPHA-II-FTIR-Spectrometer) was used to record the IR spectrum of the BSO sample.

Characterization of BSO ester and the control sample

The samples' density, specific gravity, kinematic and dynamic viscosity, cloud point, pour point, and flash point were all determined.

Mud testing and property evaluation

Formulation of muds

Drilling muds formed of inverted emulsion are produced using BSO ester and diesel oil in line with previous research [20, 22, 25]. The muds were developed to maintain the component proportions of each base fluid sample, except the oil–water ratio, or OWR to accommodate for variations in OWR concentrations throughout the circulatory system. Four separate mud samples were created using varied component

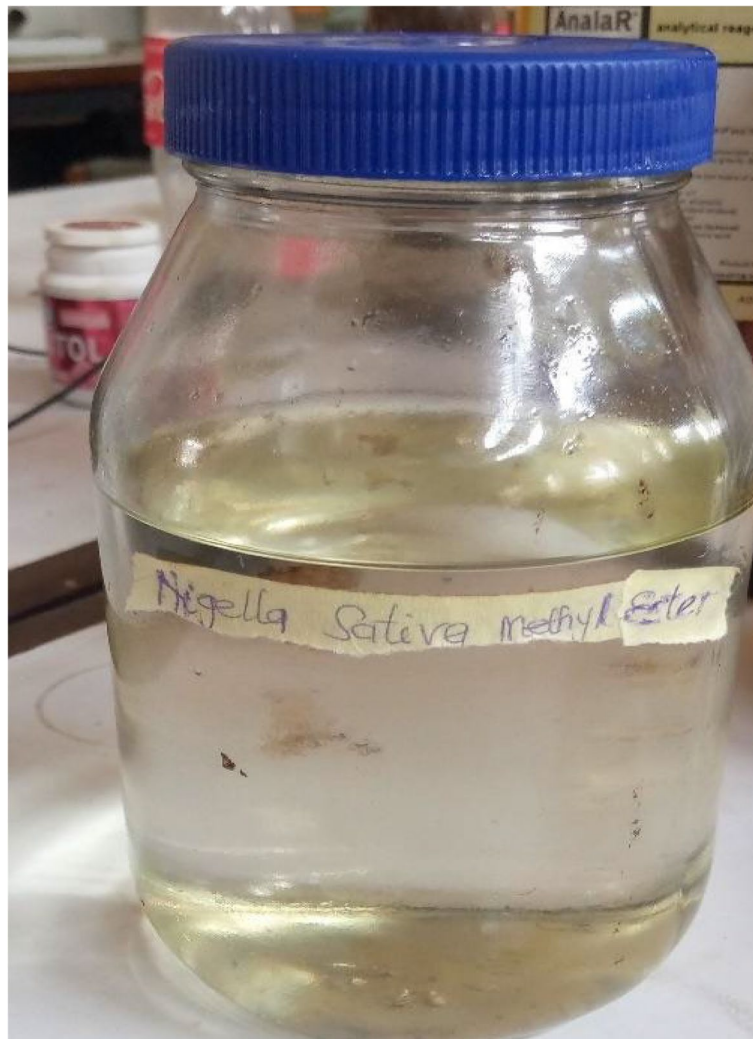


Fig. 4 Crystal clear BSO ester after washing

ratios, $A = (70:30 \text{ OWR})$, $B = (75:25 \text{ OWR})$, $C = (80:20 \text{ OWR})$, and $D = (75:25 \text{ OWR})$, each of which was successively mixed in the OFITE Multimixer as indicated in Table 1. Samples A, B, and C are BSO ester muds, whereas sample D is a diesel-based mud that was developed with API Recommended Practices 13B-2 and ISO 10416:2008. The resulting emulsion was well mixed before barite was added. The four mud samples were allowed to age statically at room temperature for 24 h before their properties were recorded [30].

Mud tests

Mud density, rheological properties, high-pressure high temperature, filtration properties, and toxicity/biodegradability were all tested on the mud samples with API Recommended Practices 13B-2.

Table 1 BSO ester drilling mud formulations for a 350 ml laboratory barrel

| Mixing order | Additives | Unit | BSO A = (70:30 OWR) | Samples BSO B = (75:25 OWR) | BSO C = (80:20 OWR) | Diesel D = (75:25 OWR) |
|--------------|--|------|--|---|---|--|
| 1 | Base oil | mL | 207 | 222 | 238 | 222.3 |
| 2 | Organophilic Clay | g | 1 | 1 | 1 | 1 |
| 3 | Lime | g | 0.5 | 0.5 | 0.5 | 0.5 |
| 4 | Primary Emulsifier (CPMUL-P) | g | 10 | 10 | 10 | 10 |
| 5 | Secondary Emulsifier (CPMUL-S) | g | 6 | 6 | 6 | 6 |
| 6 | Brine | mL | 110.3 mL H ₂ O + 48.7 g CaCl ₂ | 94.5 mL H ₂ O + 41.7 g CaCl ₂ | 78.8 mL H ₂ O + 34.7 g CaCl ₂ | 92.75mLH ₂ O + 43.51 g CaCl ₂ |
| 7 | Fluid Losscontrol (Drilling Gilsonite) | g | 5 | 5 | 5 | 5 |
| 8 | Barite | g | 116 | 120 | 118 | 130 |

Mud density

The weight of the mud was calculated using a Baroid mud balance. The dry mud cup in the mud balance, which was positioned on a level, flat surface, was added after the mud samples had been stirred with the multi-mixer for 5 min. Through the hole in the cup, some of the mud is allowed to exit, letting the compressed air escape. Readings were taken, and the weight of the mud was calculated in “ppg”.

Mud pH

By measuring the pH meter’s potential, a pH meter was used to determine the pH of the oil samples. The pH meter probe was calibrated with buffer solutions, rinsed, and blotted dry before it was inserted into the BSO ester and diesel mud samples. Measurements were taken and recorded.

Electrical stability (ES) properties

Using the Fann electrical stability meter, the ES of the BSO ester muds and diesel OBM was assessed at premix, at three test temperatures (86°F, 120°F, and 150°F), as well as after the mud samples had been statically aged. After being thoroughly cleaned with distilled water, the electrode probe was dried off and used to stir each of the mud samples. The readings were taken and recorded in volts.

Cloud and pour point tests

Seta Compact Cloud and Pour Point Cryostat are used to measure both the cloud and pour points of fluid samples. Two of the tester compartments are filled with 1/2 Litres of methanol each, and the testers’ test tube is filled with black seed ester and diesel oil samples up to the upper mark and fitted with a thermometer cork with a thermometer into

the oil samples in the test tube through the cork. The outer black insulating gasket and disc on the glassware are placed into the cryostat compartment and power to the corresponding compartments is switched on. The test tube at periodic intervals is brought out to check the cloudiness of the oil samples. A thermometer is placed in a test tube that is tilted on a flat surface. The pour point of an oil sample is the temperature at which the first drop of oil forms. A thermometer is placed in a test tube that is tilted on a flat surface. The pour point of an oil sample is the temperature at which the first drop of oil forms.

Flash point test

A flash point tester, Seta Flash Series 3 is used to measure the flash point of fluid samples. The flash point tester power is switched on, and the test temperature is set to 150°F because it is vegetable oil. A syringe is loaded with the sample and injected into the sample cup through the filler orifice. The gas supply is switched on and lighted while the pilot and test jet flame is set at 4 mm. At the set temperature, a warning beep sounds. The shutter is opened and closed over 5 s. A flash is detected at the flash point of the sample and recorded as the flash point of the sample. With the opening and closing of the shutter, the temperature when a fire is ignited also is recorded. The sample cup is allowed to cool to room temperature and the steps above are repeated for the diesel oil sample.

Rheological and filtration properties

The rheological properties and flow characteristics of the 4 invert emulsion drilling muds (IEDMs) were investigated using an OFITE Model 800 viscometer with 8 accurately regulated speeds and a broad-ranging shear rate from 3 (Gel), 6, 30, 60, 100, 200, 300, and 600 RPM under a temperature of 250 °C and differential pressure of 500 psi. The calibration of the viscometer was done in accordance with API recommended practice 13B-2. The speed of the viscometer in terms of RPM was adjusted using a regulator knob and the deflection readings were taken accordingly as shown on the light-enabled dial panel. The samples were examined for homogeneity at the test temperatures. The deflection readings and the RPM values were subsequently used as inputs for the estimate of rheological properties of the drilling muds, such as mud's apparent viscosity, (AV), plastic viscosity (PV), and yield point (YP) at varying temperatures (86°F, 120°F, and 150°F) after aging using Eqs. (3– 5). The gel-strengths (GS) were tested at 10 s and 10 min [25].

$$\text{Apparent viscosity (AV)} = \theta_{600}/2(\text{mPa.s}) \quad (3)$$

$$\text{Plastic viscosity (PV)} = \theta_{600} - \theta_{300}(\text{mPa.s}) \quad (4)$$

$$\text{Yield point (YP)} = (\theta_{300} - \text{PV})(\text{Pa}) \quad (5)$$

The API filtrate volume was measured using the API Fann filter press. The cake thickness formed was measured thereafter. The mud cell was then pressurized to 600 psi with 100 psi back pressure to provide 500 psi differential pressure while measuring the

volume of filtrate recovered over 30 min for high-pressure high-temperature (HPHT) processes. To avoid results being distorted by the hot filtrate evaporating, back pressure is used.

Toxicity/biodegradability

On the grounds of the university's laboratory, growing bean seedlings were used to test the four mud samples for toxicity and biodegradability. After 5 days of growth, 100 ml of each of the three BSO ester mud and diesel oil drilling mud samples were exposed to bean seedlings [2, 20]. The number of days they lived and the effects they had on soil life and plant growth were all noted.

Results and discussion

BSO was transesterified to produce BSO ester, which showed a 58% decrease in viscosity. As a result of the long-chain fatty acid content of the oil being converted to alkyl esters and a decrease in the quantity of bound and free glycerol, the oil's triglycerides dropped after transesterification. In terms of clarity, color, and performance, the BSO ester appeared superior as well [28, 31].

Base fluid characterization

Table 2 shows some of the basic (fuel) properties of BSO ester and diesel. A universal hydrometer, (HYDROMETER-1000–2000, OMSONS) was employed for measuring the specific gravity of liquids based on the concept of buoyancy, and water whose density is approximately 1 g/ml was used as the reference fluid. The formula used to calculate the specific gravity of the oil at 86°F is:

$$\text{Specific Gravity, S.G.} = \frac{\text{Density of oil}}{\text{Density of water}} \quad (6)$$

The density of diesel and BSO ester at 86°F is 0.85 g/ml and 0.95 g/ml, respectively. The BSO ester has a specific gravity of 0.95, demonstrating that this ester can be used to create SBMs with a variety of high densities. Low cloud points in BSO SBF are a crucial factor in determining storage stability, particularly at low temperatures where they will prevent phase separation and instability.

Table 2 Basic (fuel) properties of BSO ester and diesel

| Properties | BSO ester | Diesel |
|---|----------------|----------------|
| Colour | Clear Liquid | Brown Liquid |
| Appearance Form | Viscous Liquid | Viscous Liquid |
| Kinematic Viscosity (40°C) (mm ² /s) | 2.70 | 3.52 |
| Dynamic Viscosity (cp) | 2.57 | 2.98 |
| Density (g/ml) at 15°C | 0.95 | 0.85 |
| Specific Gravity | 0.95 | 0.85 |
| Flash Point | 163 | 78 |
| Cloud Point | -28 | -7 |
| Pour Point | -15 | -18 |

Compared to diesel, which has a flash point of 70 °C, the BSO ester has a much higher flash point of 163°F, ensuring greater transportation fire safety, and capacity storage than diesel and is within the operational range while significantly higher than the minimum limits of the API specifications [8]. The extracted biodiesel has a much higher pour point than diesel even though both have a low operating temperature. Another significant fuel property that displays advantageous ester properties is their ability to be used in any climate without causing cold flow issues [13]. Because both base oils' pour points are lower than the surrounding air, their mud can be pumped out of the mud pit. Samples of the base oil, BSO ester, and diesel oil all have similar properties and adhere to API standards.

BSO ester compositional analysis

Figure 5 reveals FTIR spectra of BSO ester at the mid-infrared region (MIR; 4000–400 cm^{-1}). The functional groups responsible for IR absorption in BSO ester are shown in Table 3. Regarding the number of peaks, there are more than five peaks, indicating that BSO ester is an organic compound that contains an oxygen-related group, such as alcohol or phenol. A peak at 2200 cm^{-1} should be an absorption band of $\text{C}\equiv\text{C}$. High intensity at 1650 to 1600 cm^{-1} , producing double bonds or aromatic molecules. No triple bond region (2000–2500 cm^{-1}) was detected, informing no $\text{C}\equiv\text{C}$ bond in the material [32]. Conjugation present or absent in the MIR region.

Mud pH

The obtained pH values of the mud samples are within the pH level ranging from 8.5 to 10 as shown in Fig. 6. Hence, the pH values of the base oil samples are comparable and give a proper yield that will prolong the life of drilling equipment by neutralizing corrosion tendencies and problems with minimal well bore stability [26, 27].

Mud density

The mud weight of BSO ester mud decreases as the OWR rises as shown in Fig. 7, with sample A having the highest mud weight. The density of both base fluids increased from premix to 10 ppg at varying barite content as shown in Fig. 8. The density of A, B, and C muds shows decreasing a trend as oil content is increasing because water density is higher than ester-based mud density [14].

The findings demonstrated that BSO ester mud densities were significantly higher than diesel, resulting in lower formulation costs. These results will lead to improved bore clearing and a heightened ability to increase weight and maintain column or hydrostatic pressure while suspending cuttings in the mud.

Electrical stability (ES) properties

At three test temperatures of 86°F, 120°F, and 150°F, Fig. 9 displays the ES data for BSO ester and diesel mud samples. The results of the ES tests demonstrated that the BSO ester mud has acceptable ES values and compares favorably to the diesel mud. Samples A and B had the higher ES value above 400 V at 86°F and after static aging. As the temperature of observation increases, the ES values for all mud samples drop, which causes them to separate and break apart, especially in static conditions. Sample C's ES values are below the required 400 V after static aging [5, 11]. The more stable emulsion is indicated

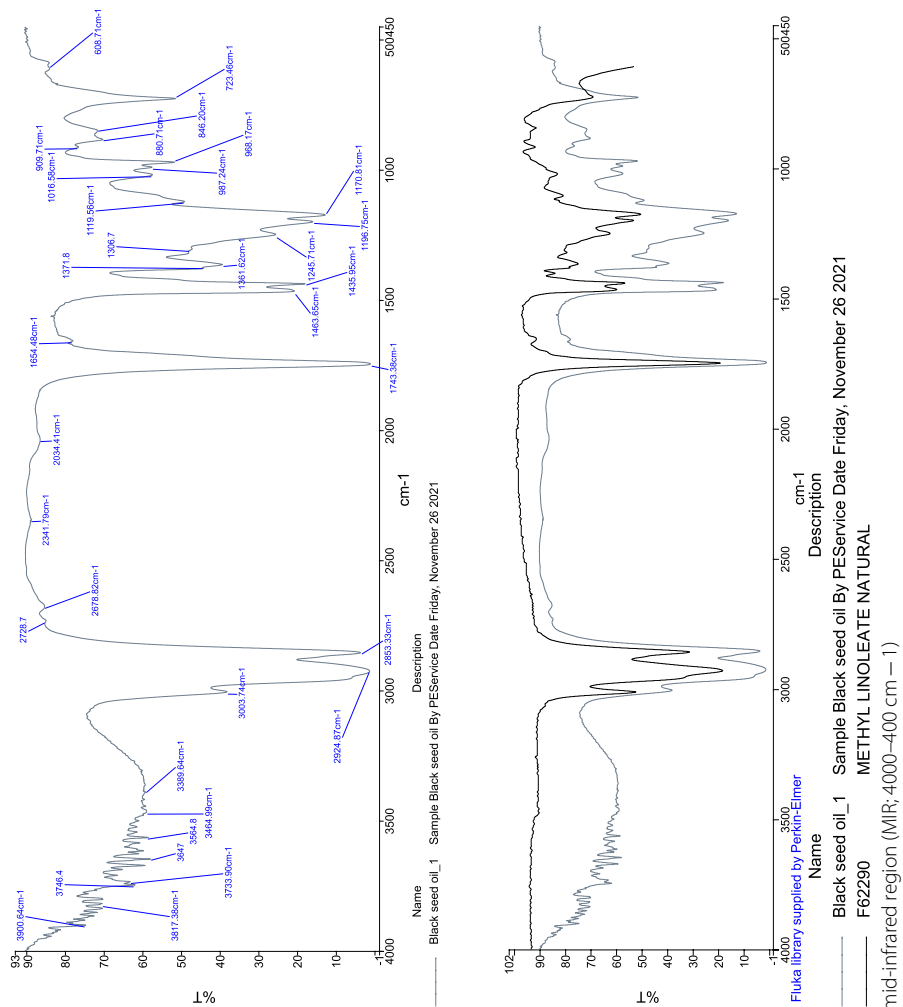
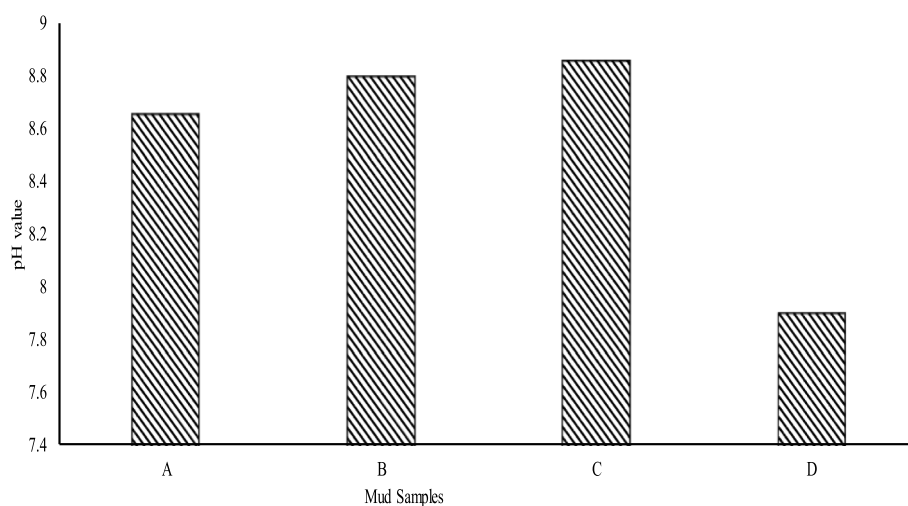


Fig. 5 FTIR spectra of BSO ester at the mid-infrared region (MIR; 4000–400 cm⁻¹)

Table 3 FTIR Analysis of BSO ester

| Functional Group | Strength of band | Peak Value (cm ⁻¹) | Bond |
|---------------------------------|------------------|--------------------------------|--------------|
| Ester group | Strong | 1743.38 | C=O stretch |
| Alcohol group, hydrogen bonding | Broad and Strong | 3389.64 | O-H stretch |
| Primary Amine | Medium | 3464.99 | N-H stretch |
| Alkene, Aromatic group | Weak to Medium | 3003.74 | =C-H stretch |
| Alkene | Weak to Medium | 1654.48 | C=C stretch |
| Alkane | Strong | 723.46 | -C-H stretch |
| Alkane | Medium to Strong | 2924.87 | -C-H stretch |
| Alkane | Medium | 2853.33 | -C-H stretch |
| Acid | Medium | 1252.22 | C-O stretch |

**Fig. 6** pH values of samples of mud

by A's higher ES values. Sample D peaked at its emulsion values at room temperature after 24 h of static aging after which it began to break again at increased temperature.

Rheological properties of drilling muds

Figures 10, 11, 12, and 13 show the data on AV, PV, YP, and GS, respectively, before and after static aging tests for (86 °F, 24 h), and the effect of temperature on the shear stress versus shear rate profile of the mud is also shown. As a result of their lower viscosity at all temperatures, aging conditions, and shear rates examined [22], BSO esters will offer less resistance to fluid flow, resulting in a turbulent flow at low pump pressure, which will result in good hole cleaning and effectively suspend drilling mud cuttings when circulating.

Plastic viscosity

BSO ester mud have a lower PV and an increase in PV value with aging, and a subsequent decrease with increasing temperature. Before static aging, the PV of the BSO ester is minimally lower at premix, i.e., before the addition of barite as shown in Fig. 10, and increased significantly after barite was added due to the fatty acid chain

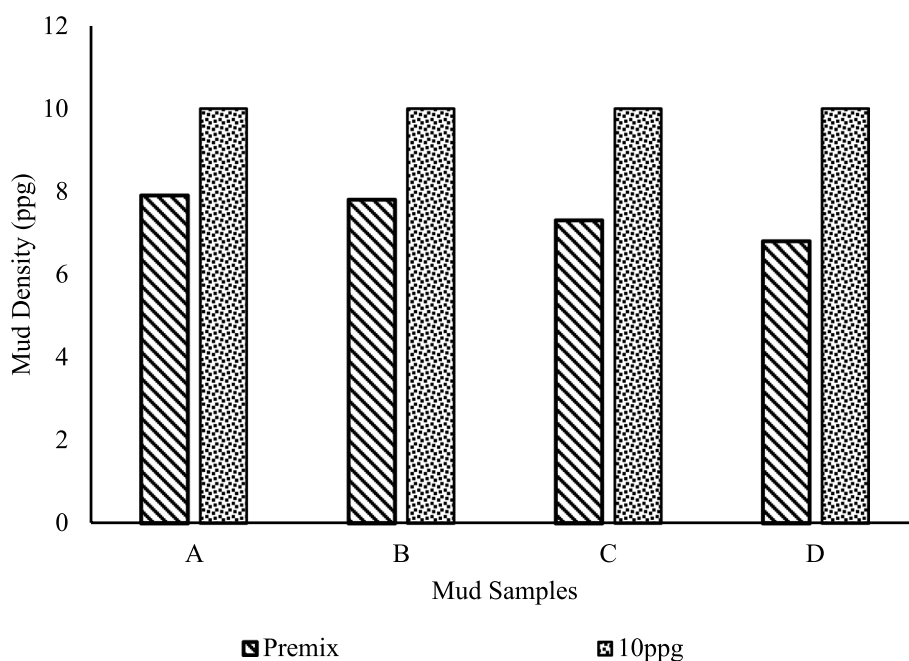


Fig. 7 Mud sample density at premix and 10 ppg

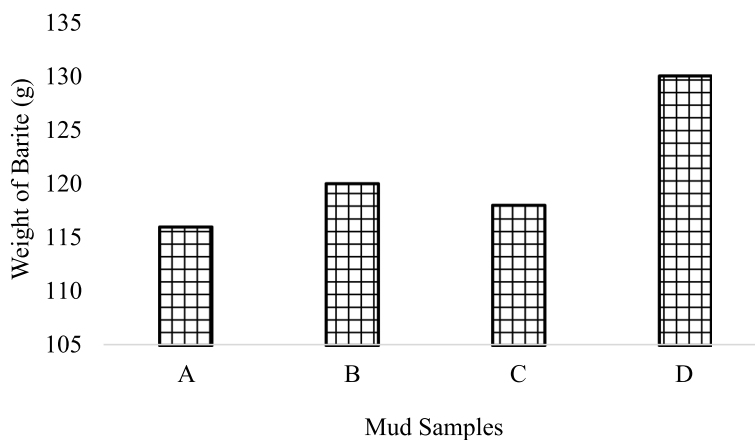


Fig. 8 Barite weight used to obtain samples with a mud density of 10 ppg

length and the existence of carbon double bonds. After static aging, the viscosities of all the mud samples slightly reduced as the temperature increased which indicates that the fluids are temperature sensitive. For example, between 86°F at 24 h and 120°F at 24 h of static aging, the PV of the BSO ester, sample A decreased by 52.2% from 23 to 11cp which could be due to solid sag, while that of the diesel decreased by 27.3% from 33 to 24cp [6]. While sample C did not fall within the range at 86°F after 24 h of static aging, all the mud samples met API recommendations for optimum performance drilling fluid range for the PV which is below 35cp indicating an increase in the rate of penetration during drilling operations as well as provide superior hole quality.

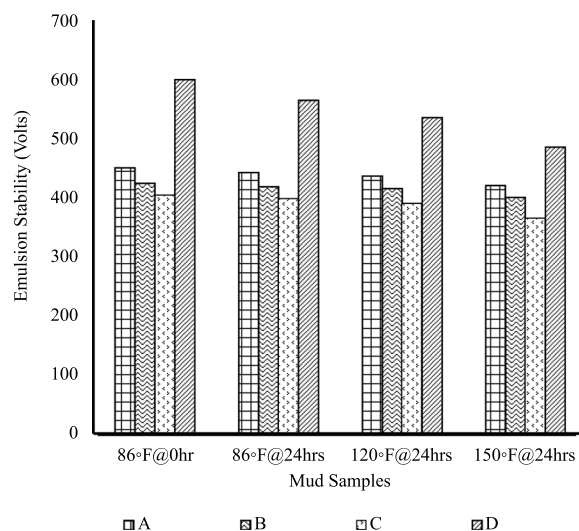


Fig. 9 Electrical stability (ES) of mud samples

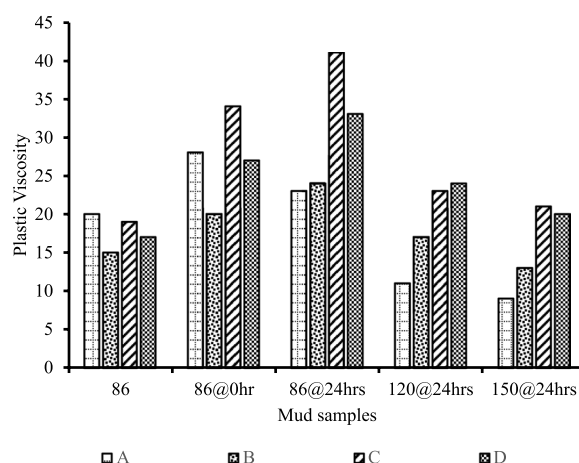


Fig. 10 Plastic viscosity (PV) of mud samples

Apparent viscosity

The AV of the mud samples, as determined by dial readings (cp) against viscometer speeds at various temperatures and aging conditions, is shown in Fig. 11. It shows that the viscosity of BSO ester mud is mildly sensitive to temperature. As temperature increases, the strength of intermolecular forces diminishes. This accounts for the observed decline in the viscous property. The OWR rose and the mud’s AV was decreasing because a further rise in shear rate would cause the water droplets to cluster and organize themselves into strings, which would reduce the viscosity of the fluid [1, 33]. The comparison of the two-based mud reveals that the mud made from BSO ester has satisfactory apparent viscosity.

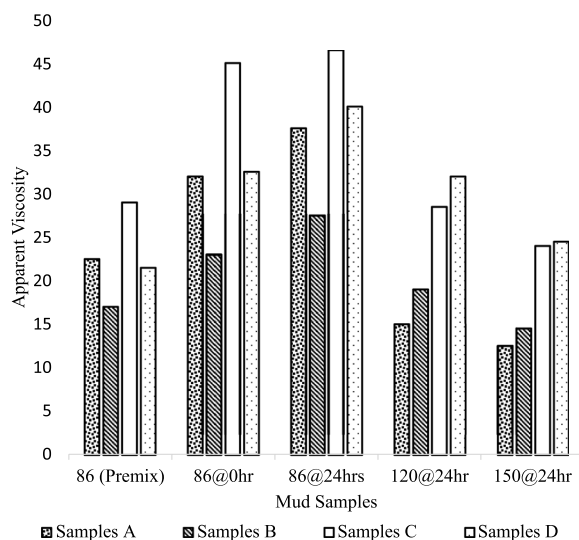


Fig. 11 Apparent viscosity of the mud samples

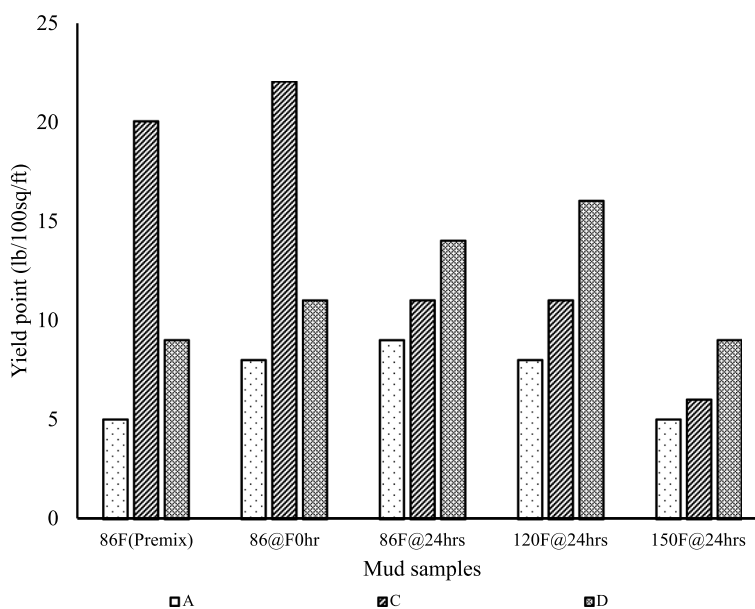


Fig. 12 Yield point (YP) of mud samples

Yield point

The yield point values of BSO ester mud and diesel-based mud are shown in Fig. 12. The yield value of the mud samples decreased as the temperature increased due to the reduction in the density of the samples. Only sample C's yield value decreases with an increase in temperature from 22lb/100ft² until it reaches 11 lb/100ft² at 86°F after 24 h of static aging and started decreasing with increasing temperature slightly to 6 lb/100ft² at 150°F, though it falls within the API's permitted range for the YP is between 15 and 25 lb/100ft². The yield point values of samples A and B are both lower than those of samples C and D. As the OWR rises, the yield point of BSO ester-based mud falls; in other words, raising the oil phase lowers the yield point. The content of colloidal sizes

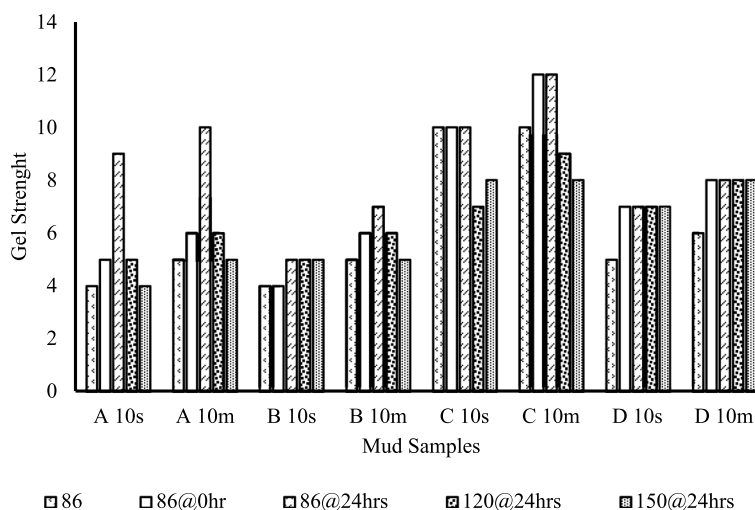


Fig. 13 Gel strength (GS) of mud samples

Table 4 API recommended range of drilling fluids properties. API specification for mud (American Society of Mechanical Engineers, 2005)

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

of organophilic clays interacting with the BSO ester molecules was the reason for the unimproved YP of the ester mud [27, 34], although a lower YP, on the other hand, would represent the effectiveness of the drilling mud’s performance.

Gel strength, GS

From 86 to 150°F, as well as before and after static aging, there are no discernible differences in the gel strengths of the formulated mud samples, and both mud samples have good gel structures, and they show advantageous rheological properties. Figure 13 demonstrates that the gel strengths of all the mud samples are within the operational recommended range presented in Table 4 except in a few cases before at premix, at 86°F, and 150°F. They are temperature dependent and their gel strengths tend to reduce with increasing temperature as mirrored in sample C. These reductions indicate a decrease in the flocculation of clay particles, which shows a slightly higher value of the petrol-diesel than the biodiesel [27]. Both base fluid samples maintained their thixotropic behavior suggesting advantageous suspension efficiency of drilled cuttings.

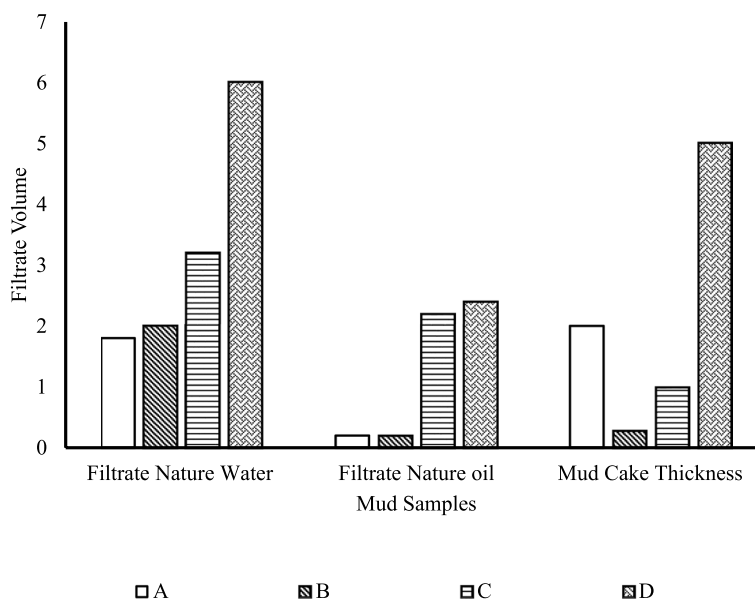


Fig. 14 HPHT static filtration properties of mud samples

Filtration properties of mud samples

The filtrate volume and filter cake thickness results after static aging of the BSO ester are not similar to that of the diesel. The API specifies a filtrate loss volume of less than 4.0 ml and a filter cake thickness of less than 2.0 mm (Table 4). As shown in Fig. 14, mud sample D has the largest filtrate volume of 5.4 ml, while mud sample A has the smallest volume of 2 ml. Low fluid loss is an important characteristic of effective drilling fluids and is required for borehole integrity. Samples B and C show good mud cake thickness of 0.28 mm and 1.0 mm, respectively, in the range. Because sample B has less water than A, cuttings are less likely to retain oil, which makes cuttings disposal easy, affordable, and good for wellbore stability [7, 35]. The properties of BSO ester-based mud are satisfactory as displayed in Figs. 14 and 15, per the filtration results. The goal of good drilling fluids is to build a thin filter cake on the borehole wall as shown in sample A in Fig. 15. By retaining less oil on drilled cuttings, the low OWR showed that BSO ester mud is more viable with low fluid loss, which enhances wellbore stability.

Mechanism of interaction of how filtration is minimized

The apparent viscosity of BSO muds and the solid particle distribution shows how BSO improves the filtration property of drilling fluids. The filter cake was created at a

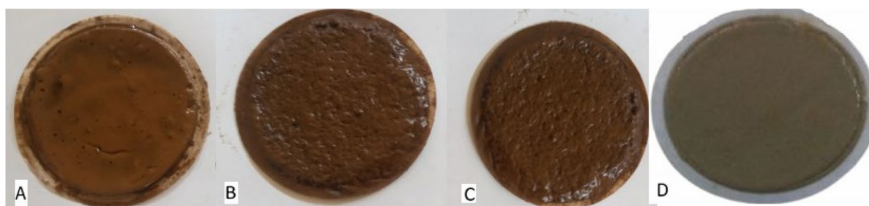


Fig. 15 Filter cake photographs of HTHP static filtration. Note: **A** BSO @ 70:30 OWR. **B** BSO @ 75:25 OWR. **C** BSO @ 80:20 OWR. **D** Diesel @ 75:25

temperature of 250 °C and a pressure difference of 500 psi. A low solid concentration in the filter cake leads to a low filtering loss, as demonstrated in BSO mud samples, particularly sample A, according to [36]. Filtration loss additives with deformable particles to fill tiny gaps and strengthen the seal, according to [37], can aid in the formation of a desired filter cake [18]. As a result, the distribution of solid particles has a significant impact on the filter cake formation process [38]. These solid particles include barite, organophilic clays, emulsion droplets, and additives for filtration control loss.

Toxicity/biodegradability

The rate of growth and number of days survived by the bean seedling was recorded in Fig. 16. It took the bean plant submerged in sample D, diesel base mud, 5 days to survive before it withered. In mud samples A, B, and C, the bean plants persisted for many days as shown in Fig. 16, respectively. According to observations, earthworms and tiny insects were found in the BSO-based mud, but there was no sign of any living organisms in the diesel oil-based mud sample. BSO esters caused the seeds to survive the longest, indicating low toxicity, while diesel caused the seeds to survive for the shortest amount of time, indicating high toxicity. Green materials or plant materials containing oxygen inside their structure degrade faster, implying that BSO-based mud is less poisonous and less detrimental to the environment [7].

Comparison with other (vegetable based synthetic-based muds)

This experiment’s findings typically contrasted with those of other vegetable oils in the literature. The rheological properties outperform prior works using the same OWR, while the physicochemical properties obtained from this study are in an agreement with earlier work on the fundamental characteristics of vegetable oil. Tables 5 and 6 compare the emulsion and rheological characteristics of some prior investigations into groundnut biodiesel, soya bean, methyl palm oil with BSO ester-muds, and diesel-based mud. They are all comparable.

Rheological model of the mud samples

Figures 17, 18, 19, and 20 display the rheogram of mud samples A, B, C, and D. The flow curves of the mud sample made with BSO ester, a synthetic oil, are shown in

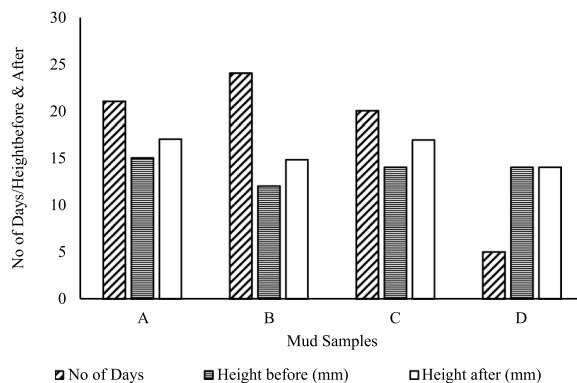


Fig. 16 Result of bean seedling exposure to mud samples (biodegradability)

Table 5 Emulsion stabilities and rheological properties of vegetable oils

| Vegetable Oil | Groundnut Biodiesel mud | Soya bean Biodiesel mud | Palm oil methyl ester mud (Sauki, 2015)[15] | Waste cooking biodiesel mud (Li, 2015)[21] | Black seed Biodiesel mud (This study) |
|---|-------------------------|-------------------------|---|--|---------------------------------------|
| pH | 8.9 | 9.2 | - | - | 7.3 |
| ES | 870 | 1030 | - | 1540 | 398 |
| MW (ppg) | 8.3 | 8.3 | 10.7 | 8.56 | 10 |
| PV (CP) | 53 | 49 | 15 | 13.5 | 41 |
| YP (lb/100ft ²) | 29 | 23 | 21 | 10 | 11 |
| GS (10 s/10 m) (lb/100ft ²) | 8/9 | 7/9 | 8/16 | 9/10.5 | 10/12 |
| AV | 67.5 | 60.5 | 25.5 | - | 46.5 |
| 6 rpm | 10 | 9 | - | - | 10 |

Table 6 Emulsion stabilities and rheological properties of vegetable oils at 70/30 OWR

| Vegetable Oil | Groundnut Biodiesel mud | Soya bean Biodiesel mud | Palm oil methyl ester mud | Black seed Biodiesel mud (This study) |
|---|-------------------------|-------------------------|---------------------------|---------------------------------------|
| pH | 9.5 | 9.4 | - | 8.65 |
| ES | 706 | 815 | - | 442 |
| MW (ppg) | 8.6 | 8.5 | 10.7 | 10 |
| PV (CP) | 75 | 65 | 26 | 23 |
| YP (lb/100ft ²) | 35 | 33 | 43 | 9 |
| GS (10 s/10 m) (lb/100ft ²) | 11/13 | 9/10 | 18/24 | 9/10 |
| AV | 92.5 | 81.5 | 47.5 | 37.5 |
| 6 rpm | 13 | 11 | | 9 |

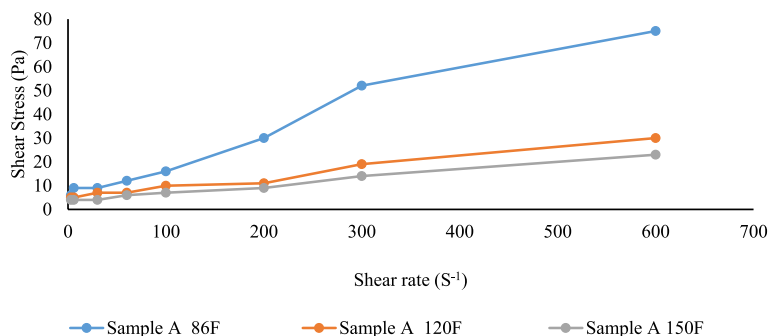


Fig. 17 Shear stress–shear rate of synthetic oil-based mud at 70:30 OWR, 86°F, 120°F, and 150°F temperatures

Fig. 17. The influence of the OWR 70:30 and the temperature at 86°F, 120°F, and 150°F were investigated. Shear thinning of the mud sample was observed in the lower shear rate range of 10 s⁻¹ to roughly 300 s⁻¹. Figure 18 shows the flow curves of a mud sample made with BSO ester; the impact of an OWR 75:25 and three different temperatures—86°F, 120°F, and 150°F were examined. The plant oil has an appreciable effect on mud shear thinning, with shear rates ranging from 10 s⁻¹ to around 200 s⁻¹. The flow curves of the mud sample made using BSO seed oil are shown in Fig. 19,

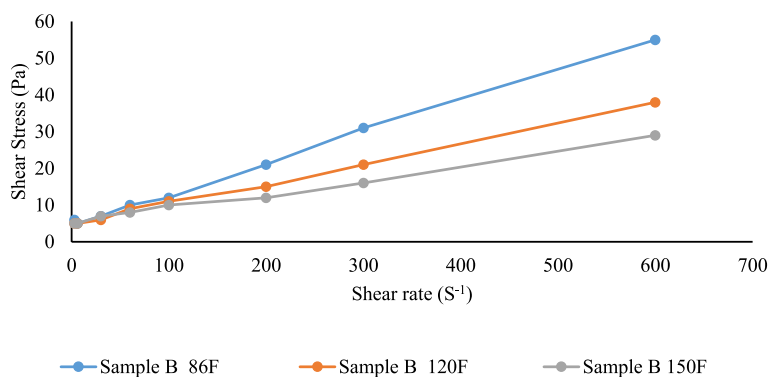


Fig. 18 Shear stress–shear rate of synthetic oil-based mud at 75:25 OWR, 86°F, 120°F, and 150°F temperatures

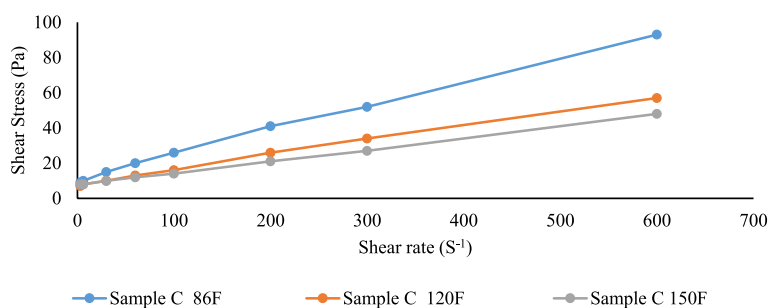


Fig. 19 Shear stress–shear rate of synthetic oil-based mud at 80:20 OWR, 86°F, 120°F, and 150°F temperatures

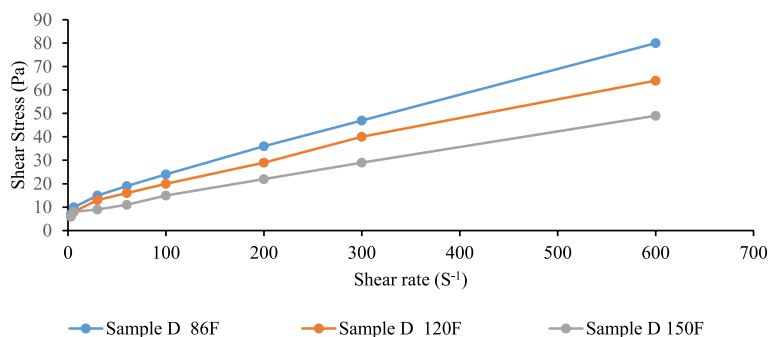


Fig. 20 Shear stress–shear rate of diesel oil-based mud at 75:25 OWR, 86°F, 120°F, and 150°F temperatures

where the impact of an OWR 80:20 and three different temperatures at 86°F, 120°F, and 150°F was studied. The plant oil exhibited the mud shear thinning effect, which happened at a shear rate between 10 s⁻¹ and 300 s⁻¹.

Figure 20 shows the flow curves of a mud sample made with diesel oil. The impact of OWR 75:25 and three different temperatures at 86°F, 120°F, and 150°F, respectively, was examined. The conventional oil exhibited a mud shear thinning effect, with shear rates varying from 10 s⁻¹ to around 200 s⁻¹. The linearity of the plot causes the viscous flow curves in Figs. 17 and 20 to show a similar non-Newtonian flow behavior throughout a broad range of shear rates. This demonstrates that when BSO ester is

used as a drilling base fluid in a drilling operation, the mud can function similarly to mud formulated with diesel [27, 31, 39].

The mud shear thinning effect exhibited by the BSO ester muds is due to the density of the synthetic base fluid [20]. The yield stress, or the crossing point with the y -axis, is not visible in the figures because of the minimal shear rate, which is roughly 10 s^{-1} and increases more linearly with increasing trend of temperature. Until the shear stress surpasses a certain value called the yield point [40], the fluid initially resists flowing. Shear stress and shear rate have a relationship that is linear after the fluid starts to flow [41]. This trend was also observed in the work of [21, 31].

For an extensive range of shear rates, the shear stress–shear rate relationship continues to follow non-Newtonian flow behavior. The potential of BSO ester as a continuous phase in the mud formulation is further demonstrated by the fact that the shear stress increased with an increase in OWR. In contrast, the shear viscosity steadily decreased with temperatures. This resulted from the BSO ester's improved viscosity and comparable with that of diesel oil and also demonstrated that the mud formulation met the API criteria for acceptable flowing properties [18].

Cost comparison of biodiesel with diesel-based muds

The cost of a biodiesel-based solution is comparable to the cost of a diesel-based solution. A cost comparison found that while the initial cost of biodiesel-based drilling fluid is greater, it provides a more convenient and cost-effective disposal option than oil-based drilling fluid [2]. Because of the high-specific gravity and kinematic viscosity, biodiesel is more economical when used for mud formulation because fewer weighting agents and rheology modifiers are required to achieve the proposed mud weight, rheology, and gelling characteristics of the muds [11] and optimize drilling economics [12, 21].

The economic evaluation of the inverse emulsion mud suggests reduced formulation and waste management costs [25], implying lower waste disposal costs [20]. Although the initial cost of biodiesel is greater than that of diesel, the economic study revealed that it has lower disposal costs than diesel. Furthermore, owing to the rapid increase of worldwide biodiesel production, different supportive regulations, and its cheap treatment cost [24, 27], methyl ester-based mud is economically and technically viable for oil and gas drilling operations.

The compositions of drilling fluids will vary depending on the needs of the drilling operation. Holes must be drilled through various forms of lithology in a formation that requires multiple types of drilling fluid density [42]. Under oil field conditions, BSO is synthesized in the same way as diesel-based drilling mud is given in the form of raw materials created at the drilling site. During well planning procedures, there is always a specified logistics solution in the drilling schedule for transferring raw materials to the drilling site [23]. Further research will focus on the technological aspects of preparing it in sufficient quantities maybe as a single unit for biodiesel solutions.

Conclusions

The results of the testing show that BSO ester has a good possibility of being a technically feasible alternative for diesel OBM. Its qualities demonstrated that it was a good alternative to its synthetic versions, diesel, and prospective drilling options are mud

manufactured with BSO esters; as a result, its usage is essential for drilling contractors and service firms due to its high environmental acceptability. Furthermore, the following findings were reached:

1. The performance of BSO ester mud was enhanced by the transesterification reaction.
2. Because of its exceptional ecological acceptability, good emulsion stability, high flash point, high thermal stability, and great biodegradability, the extracted biodiesel may be employed as a continuous phase of synthetic-based mud.
3. Fewer weighting agents and no rheology modifier were required to achieve the 10 ppg mud weight while still meeting the API criteria of the base oil.
4. The BSO ester has higher application applicability, rheological, and filtering capabilities in mud formulations.
5. According to the biodegradability assessment results, BSO ester-based mud is less harmful to plant growth than diesel oil-based mud. This reveals that the BSO ester mud sample is safer for plants and microbes than the diesel mud sample.

Recommendations

1. Identifying the optimal alcohol-to-oil ratio for transesterification to improve the characteristics of BSO.
2. Using local additives to improve the rheology of BSO mud samples.
3. Future research might investigate the stability of BSO ester mud at high temperatures, as well as its performance in the presence of nanoparticles in BSO ester mud.
4. Additional research will be conducted in the technological areas for making it in the needed amounts, possibly using a single unit for biodiesel solutions.

Abbreviations

| | |
|----------------|--|
| BSO | Black seed oil |
| FTIR | Fourier transform infrared (FTIR) spectrometry |
| HPHT | High pressure high temperature |
| OBF | Oil base fluid |
| OBM | Oil-based mud |
| OWR | Oil to water ratio |
| ppg | Pounds per gallon |
| SBM | Synthetic-based mud |
| WBM | Water-based mud |
| API | American Petroleum Institute |
| ASTM | American Society for Testing and Materials |
| GS | Gel strength |
| θ_{600} | Dial reading at 600 rpm |
| θ_{300} | Dial reading at 300 rpm |

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Authors' contributions

DO gathered the materials and analyzed and interpreted the experiment data regarding the performance evaluation of *Nigella sativa* Lester drilling mud. AA supervised the research work and was a major contributor to writing the manuscript. The authors read and approved the final manuscript.

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