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Comparison of Performance Properties of Muds Formulated With Synthesized C_{16} and C_{16} Esters of Lauric Acid

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ABSTRACT: Drilling muds have relied on a range of base fluids, with mineral oil-based formulations dominating the landscape for decades. However, mineral oil-based muds contain a plethora of toxic aromatic compounds which are persistent in the environment. Hence, the objective of the paper was to synthesize and compare the performance properties of drilling muds formulated with C_{14} and C_{16} esters of lauric acid using appropriate standard procedures. Benchmarking of the esters with a reference synthetic base fluid indicated that the esters have suitable physicochemical properties for application as synthetic base drilling fluid. Their kinematic viscosities are within the API recommended range, ethyl laurate (EL) has a lower cloud point relative to the reference, and the two base fluids have higher flash point and electrical stabilities relative to the reference. The results obtained from comparing the rheology of muds prepared with ester products and that prepared with the reference fluid indicate that the muds prepared with ethyl, and n-butyl laurate have higher electrical stability than the mud prepared with the reference base fluid. The results also show that the muds prepared with the esters synthesized in this work displayed better rheology profiles than the mud prepared with the reference synthetic base fluid. However, ethyl laurate (EL) formulated mud had better thermal stability than n-butyl laurate (BL) at the temperature range studied. Through the investigation of these ester-based drilling muds, we showcased the potential of these esters to enhance drilling efficiency, minimize environmental impact, and optimize operational performance.

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The exploration and extraction of hydrocarbon reserves in the oil and gas industry demand cutting-edge technologies and innovative solutions (Aftab *et al.*, 2022). Among the critical components of this complex process, drilling fluid (also known as drilling mud) plays an important role in enabling efficient and safe wellbore drilling (American Society of Mechanical Engineers, 2004; Agwu *et al.*, 2015; Aliyu *et al.*, 2017). Traditionally, drilling muds have relied on a range of base fluids, with mineral oil-based formulations dominating the landscape for decades. However, despite the superior technical performance of mineral oil-based muds, especially in shale dominated drilling environment, they contain a plethora of toxic aromatic compounds which are persistent in the environment (Razali *et al.*, 2018). Regrettably, water-based fluids, which exhibit better environmentally friendly attitudes are unsuitable for drilling such shale formations because of their swelling tendencies that compromise well bore stability (Habib *et al.*, 2014; Lin *et al.*, 2014). So, in this era of increased environmental awareness and

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performance optimization, a significant paradigm shift is underway (Goshtasp, 2018). The search for alternative drilling fluids that will combine the technical efficiency of mineral oil-based fluids with environmental friendliness of its water - based counterpart for drilling mud formulation, led researchers to explore the suitability of synthetic base fluids (Chai *et al.,* 2015; Wai, *et al.,* 2015;) often referred to as designer fluids because they are prepared from readily available raw materials which can be customized to meet specific drilling needs (Orji, *et al.,* 2018). Esters, long recognized for their versatility in various industrial applications, have emerged as a fascinating alternative synthetic base fluid for formulating drilling muds (Anawe and Folayan, 2018; Razali *et al.,* 2018). Ester-based drilling fluid as a class of synthetic drilling fluids, was introduced in the early 1990's to address the environmental issues inherent with the application of petroleum-based drilling fluids in drilling operations (Carlson and Hemphill 1994; Nor *et al.,* 2014; Chai *et al.,* 2015;Chen *et al.,* 2020;). Over the years, they have proved to be better replacements to mineral oil-based fluids because of the absence of aromatic toxicants in their structural makeup. Thus, their toxicity is low and their biodegradability is high (Dardair et al., 2014; Aftab, *et al.,* 2022). It is true that synthetic ester-based fluids are costlier than mineral based fluids but, this is offset by the high cost of transporting drill cuttings from mineral based fluids to onshore facilities for the treatment protocols prescribed in order for them to meet various regulatory prescriptions before they are discharged into offshore environments (Abdul *et al.,* 2014; Onuh, *et al.,* 2019; Kumar *et al.,* 2020). Hence, the objective of the paper was to synthesize and compare the performance properties of drilling muds formulated with C14 and C16 Esters of lauric acid.

MATERIALS AND METHODS

Materials: The experimental work was divided into four different stages, beginning with the synthesis of the esters, and ending with mud rheology test as illustrated in Figure 1. The synthetic ester-base fluids used to formulate the mud samples were synthesized by esterifying lauric acid with ethanol and 1-butanol over sulphamic acid according to the method reported by Orji *et. al.,* (2018), while the reference fluid was supplied by Shell Nigeria Exploration and Production Company (SNEPCo). The base fluid samples were tagged as EL, BL, and RBF to represent ethyl laurate, butyl laurate, and reference base fluid respectively. Commercially available lime, brine, gilsonite, organophilic clay and barite were used in the mud formulation. Equipment used includes an H1 2211 pH/ORP meter (Hanna Instruments), Fann viscometer (Fann 35A Model), Hamilton beach mixer and cup.

FTIR Spectroscopic Analysis of the Ester Base Fluids: The functional groups present in the ester samples were investigated with a Fourier Transform Infrared (FTIR) system *prestige 21* (Shimadzu), and the infrared absorption spectra recorded in the region of 4000 – 400 cm^{-1}.

Determination of the Physical Properties of the Esters: The physical properties of the esters necessary for base fluid application include the pH, flash point, fire point, cloud point, specific gravity and viscosity. In order to determine the suitability of the synthesized esters as synthetic base fluids, these physical properties of the ester fluids as well as the reference synthetic base fluid (RBF) were measured following standard analytical procedures.

Mud Rheology Test: The ester products synthesized in this study were utilized in formulating two mud samples according to the American Petroleum Institute (API) recommended practice 13B. A reference mud with the RBF was also formulated with the same quantity of base fluid and additives, under the same experimental conditions. The formulated drilling muds consist of the ester or commercial base fluid, primary emulsifier, lime, brine, secondary emulsifier, gilsonite, and others, all made up to 350 ml volume. The summary of the quantities and functions of the various components is presented in Table 1. The plastic viscosity (PV), yield point (YP) and gel strength were determined from the record of the viscosity of the mud at different viscometer revolutions (from 3 to 600 rpm). The electrical stability (ES) of the mud was also investigated at 80 ºF in line with the standard test endorsed by API RP 13B-2 (American Petroleum Institute, 2014).

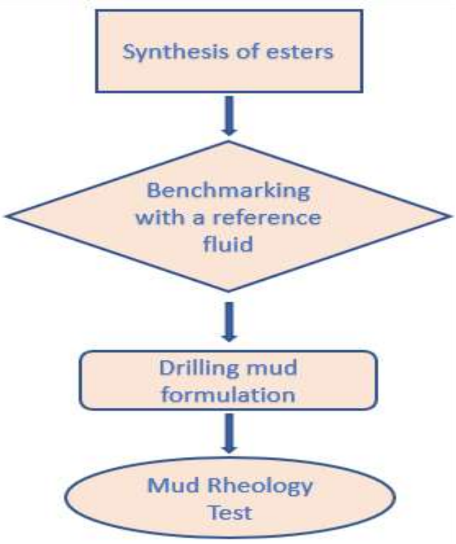

Fig 1: Flowchart of the Methodology Adopted in the Study

Table 1: Mud Formulation with the Synthesized Esters and the Reference

S/N	Component	Function of Additives	Specific Gravity (SG)	Quantity
1	Ester/RBF	Continuous Medium	0.87	210.00ml
2	Barite	Weighting Agent	4.20	76.00g
3	Brine	Salinity Source	Mixed	15.00ml
4	Primary Emulsifier	Emulsifier	0.92	3.00g
5	Gilsonite	Fluid Loss Control	2.60	7.00g
6	Organophilic Clay	Viscosifier	1.77	20.00g
7	Lime	Alkalinity Agent	2.13	12.00g
8	Secondary Emulsifier	Emulsifier	0.93	7.00ml

Effect of Temperature on Mud Rheology: The rheological properties of the drilling muds were investigated by measuring their PV, YP, 10 seconds gel strength, 10 minutes gel strength, and electrical stability. The PV, YP, 10 seconds, and 10 minutes gel strength were measured from 80 ºF to 200 ºF at 20 ºF increments.

RESULT AND DISCUSSION

FTIR Spectroscopic Studies: Infrared spectroscopy is one of the analytical tools used in the qualitative and quantitative analyses of organic compounds. The infrared spectra of organic compounds are interpreted by using known functional group frequencies for detection of the functional groups present in the compound. The two laurate ester compounds synthesized were subjected to FTIR spectroscopic analysis to confirm the presence of the ester linkage. The absorption peaks in the IR spectra of the laurate esters were compared with those of standards in order to appropriately assign the respective functional groups. Table 2 gives a summary of the major peaks observed in the spectra, their absorption frequencies, and the functional groups assigned to them. The characteristic absorption peaks of the ester functional group were observed at approximately 1111 and 1180 cm^{-1} (C-O), 1720 cm^{-1} (C=O), and 2924 cm^{-1} (CH). The spectrum of butyl and ethyl laurate were also compared with the spectra of lauric acid, butanol and ethanol. The absence of the absorption peak of the OH functional group of the carboxylic acid and alcohol, and the movement of the absorbance for lauric acid carbonyl toward shorter wavelengths indicate that the synthesis of butyl and ethyl laurate (with spectra shown in Figure 2) was successful.

Table 2: Major Peaks in the FTIR Spectra of the Esters

Functional Group	FTIR Bands (cm^{-1})	
	BL	EL
υ O-C-O (ester) stretching	1450.52 1111.03 1180.47	1450.52 1111.03 1180.47
υ C=O (ester) stretching	1720.56	1728.28
υ C-H sym and C-H asym stretching	2924.18 2862.46	2924.18 2862.46

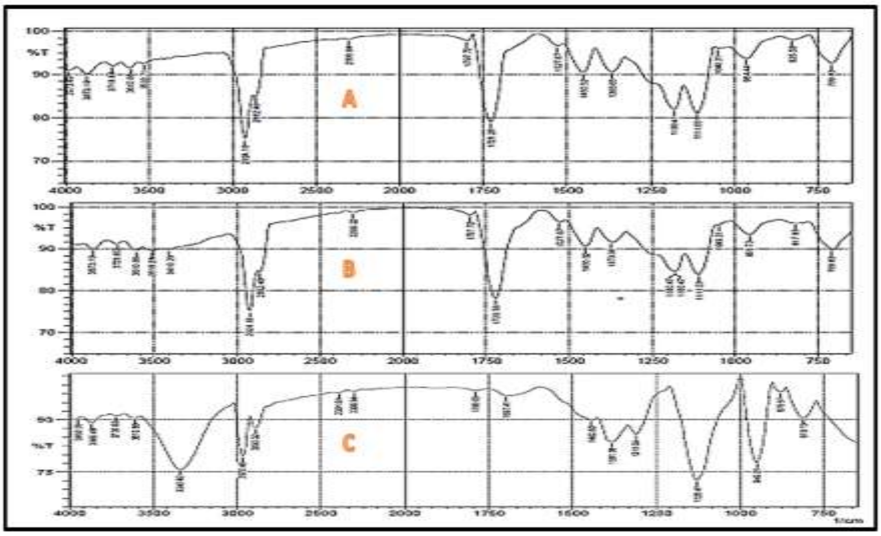

Fig 2: FTIR Spectra of A) BL B) EL C) Butanol

Benchmarking of the Esters through their Physical Properties: Base fluids are the basis for controlling various properties of a mud because they serve as a carrier for all other additives that make up the drilling mud, and ensure efficient and safe drilling operations, as well as addressing environmental concerns. The choice of base fluid for mud formulation therefore, is critical to the technical performance and properties of the mud (Bhola, and Vikas, 2020). Consequently, the properties of the esters synthesized in this work were investigated and benchmarked with that of a reference fluid and the results are presented in Table 3. Ideally, a base oil should have a neutral pH, low viscosity and pour point, with high flash point, thermal and hydrolytic stability (Razali *et al.,* 2018). The pH results showed that the two ester samples synthesized in this work have slightly lower values than that of the reference sample, but BL showed the closest value to that of the reference. This however, could be easily remedied by subjecting the esters to a more vigorous work up procedure using a mild base. The densities at 30 ºC, SG at 60 ºC and viscosity of all the samples formulated in this work were all slightly higher than that of the reference. The trend shows that the values are increasing as the number of carbon atoms in the alcohol component of the ester increases. Thus, EL with only two carbon atoms in the ethanol has the lowest values of all, except for density, where it exhibited the highest. Research has shown that a base fluid for drilling mud formulation should possess a kinematic viscosity between 1 to 10 cSt, preferably between 1 – 6 cSt at 40°C. Though, the viscosity of all the samples at 40 ºC including the reference fell within this range, that of the samples synthesized in this work are all slightly higher than that of the reference, with the BL sample exhibiting the highest viscosity of 4.16 against 3 cSt for the reference fluid. The result seems to indicate that the increase in the chain length of the oil may have affected the viscosity of the samples. All the synthesized base fluids have slightly higher specific gravity compared to the reference fluid; thus, they would be better substitutes in formulating drilling muds because less quantity of weighting agent would be required to achieve the desired mud weight (Ekeinde, *et al.,* 2024). While the BL sample has a higher flash point relative to the reference, the EL sample has a lower flash point. However, the difference is not much, just about 11% difference between them. This same trend for all the samples was observed in the fire point as well. The flash point of any fluid gives a lot of information regarding its volatility. This has significant health and safety implications as the lower flash point base fluids will generate more fumes, thereby posing greater health and safety risks. A significantly higher flash point has the advantage of minimizing the risk of fire during handling and offers safer storage management. The results of the pour point analysis showed that the ester base fluid samples exhibited comparable cloud point with the reference fluid, with two of the fluids exhibiting slightly lower pour points of -6 ºC, when compared to the value for the reference, which is -4 ºC. Pour point affects the rheology of drilling fluids at low temperatures, mostly if the mud is formulated for applications in offshore or cold environments (Carpenter and Toye, 2001). Low pour point base fluids are desirable because they will make allowance for more flexibility in the rheological behaviour of the mud at lower temperatures. Generally, the result of the benchmarking of the esters with the reference fluid indicates that the fluids synthesized in this study have suitable properties that would warrant their application in the formulation of drilling muds for oil well drilling. The results also show that the identity of the alcohol moiety in terms of chain length will significantly affect the properties of the synthesized ester as documented by Razali *et al.,* (2018).

Table 3: Physical properties of the ester base fluids

Property	BL	EL	RBF
pH	6.95	6.43	7.02
Density (SG) at 30 ºC	0.839	0.842	0.808
SG at 60 ºF	0.860	0.844	0.813
Viscosity at 40 ºC (cSt)	4.16	3.2	3.0
Flashpoint ºF	300	195	219
Fire point ºF	350	223	260
Cloud point ºC	20	17	17
Pour point ºC	-2	<-6	<-4

BL= Butyl Laurate EL= Ethyl Laurate RBF= Reference Base Fluid

Table 4: Electrical Stabilities and Rheological properties of the muds at 80 ºF

S/N	Property	EL	BL	RBF
1	PV (cP)	12	22	27
2	YP (lb/100ft^2)	6	9	10
3	10 Sec Gel Strength (lb/100ft^2)	2	5	7
4	10 Min Gel Strength (lb/100ft^2)	4	6	3
5	Electrical stability (Volts)	1218	1416	978

BL= Butyl Laurate EL= Ethyl Laurate RBF= Reference Base Fluid

Rheological Properties: The results of the mud properties at 80 ºF presented on Table 4 show that the mud samples formulated with the ester base fluids synthesized in this work have lower plastic viscosity values than the reference mud at 80 ºF, but the reverse is the case with the electrical stability (ES) values. Electrical stability is one of the most important parameters of an oil-based mud. It is a measure of the strength of the emulsion formed from the base fluid. A high electrical stability value indicates that a stable emulsion has been formed and that the mud can handle some degree of contamination during drilling operations. The higher ES of the muds formulated with the ester base fluids means that the BL and EL based drilling muds are more stable than the reference.

Effect of Temperature on Mud Rheology: The yield point (YP) is the initial resistance to flow or the minimum shear stress required to initiate flow caused by the electrochemical forces of attraction between the solid particles and liquid phase (Aftab, *et. al.*, 2022). It involves a dynamic process that determines the ability of the drilling mud to suspend and lift drill cuttings to the surface (Idress and Hasan 2020). A higher YP indicates better suspension properties, allowing the drilling mud to effectively transport the cuttings out of the wellbore (Akpan *et al.*, 2020). This might also require a high pumping power to initialize flow. The YP for the BL mud sample decreases as the temperature increases, with its highest value of 37 lb/100ft^2 at 80 ^{o}F and its lowest YP value of 6 lb/100ft^2 at 200 ^{o}F. The YP values of EL mud samples were found to be lower than that of BL except at 200 ^{o}F where the YP value of EL was slightly higher than that of BL. The RBF mud sample has the least YP values across all temperatures. This implies that the ability of the BL sample to suspend and carry cutting is greater than that of EL and RBF respectively for the temperature range of 80 - 160 ^{o}F. At 180 ^{o}F, BL and EL have the same YP while EL has a higher YP value than BL and RBF respectively at 200 ^{o}F. However, the YP values of the three mud samples meet the minimum API of 5 lb/100ft^2 for YP (Akpan *et al.*, 2020) except the YP of RBF at 200 ^{o}F which has a value of 3 lb/100ft^2. The results obtained from this research as shown in Figure 3, indicates that the PV of the RBF mud sample decreased as the temperature increased from 27 cP at 80 ^{o}F to 11.5 cP at 160 and remained unchanged even at 200 ^{o}F. The PV values of BL and EL mud samples decreased and increased in no particular trend or order and were lower in value than the PV of the RBF mud sample except the PV value of BL at 200 ^{o}F which was slightly higher than the PV of RBF. The PV value for BL was lowest and preferred at a low temperature range of 80 - 140 ^{o}F, while the PV value of EL was the lowest and preferred at a higher temperature range of 160 to 200 ^{o}F. Higher PV values imply higher viscosity of the mud. Since the PV is the resistance to flow due to the mechanical friction acting between the dispersed solids and the viscosity of the liquid phase of the mud (Onojake and Chikwe, 2019), which is dependent on the properties and concentration of the solids in the mud, It can significantly reduce the performance of the mud in carrying cuttings to the surface due to high resistance to flow (Onuh, *et al.*, 2019). Low PV value is desired for efficient hole cleaning (Allawi et. Al., 2019; Okon and Agwu, 2015), provided it's carrying capacity and other essential properties are not compromised. The PV values of the three mud samples fall within the API accepted range for plastic viscosity of 8 – 35 cP except the PV values of 5 cP at 100 ^{o}F and 6 cP at 140 ^{o}F of the BL mud sample (Orji *et al.*, 2018; Biwott *et. al.*, 2019).

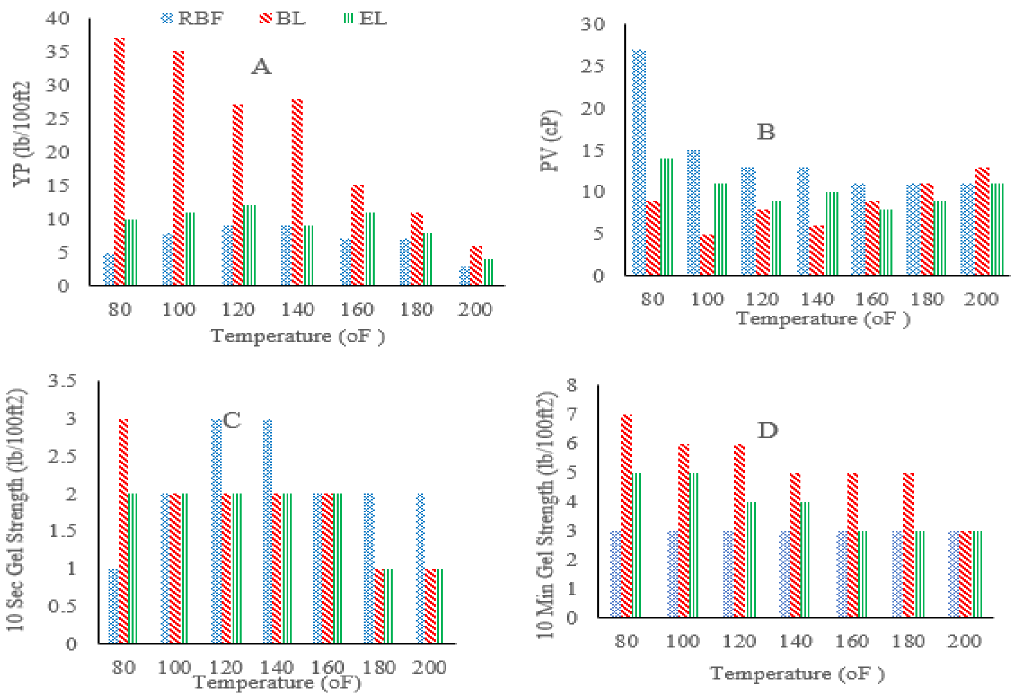

Fig 3: Effect of temperature on A) Yield Point B) Plastic Viscosity C) I0 Sec Gel Strength D) 10 Min Gel Strength

The ability of the drilling mud to suspend drilling cuttings and other solid additives when there is no applied shear stress is known as the gel strength (Salem *et al.*, 2023). It is a measure of the shear stress required to initiate flow after a static period or a period of inactivity (Onojake and Chikwe, 2019). A higher gel strength value is required to prevent mud sagging and fluid loss, and also to maintain wellbore stability when drilling operations are paused provided it falls within the API range of 2 - 35 lb/100ft^2 for 10 minutes gel strength (Biwott *et al.*, 2019; Khan *et al.*, 2023). The variation of the 10-Minutes and 10 - seconds gel strength with temperature is also presented in Figure 3. The graphs show that the 10 Minutes gel strength of BL and EL decreases with increase in temperature. For the BL mud, there's a decrease from 7 lb/100ft^2 at 80 ºF to 3 lb/100ft^2 at 200 ºF, while that of EL decreases from 5 lb/100ft^2 at 80 ºF to 3 lb/100ft^2 at 200 ºF. Conversely, for the RBF mud sample, the 10 Minutes gel strength values remain constant at 3 lb/100ft^2 with increase in temperature. This suggests that the BL mud sample with a higher 10-minute gel strength value has a better carrying capacity (Sami, 2015) relative to the EL and RBF mud samples.

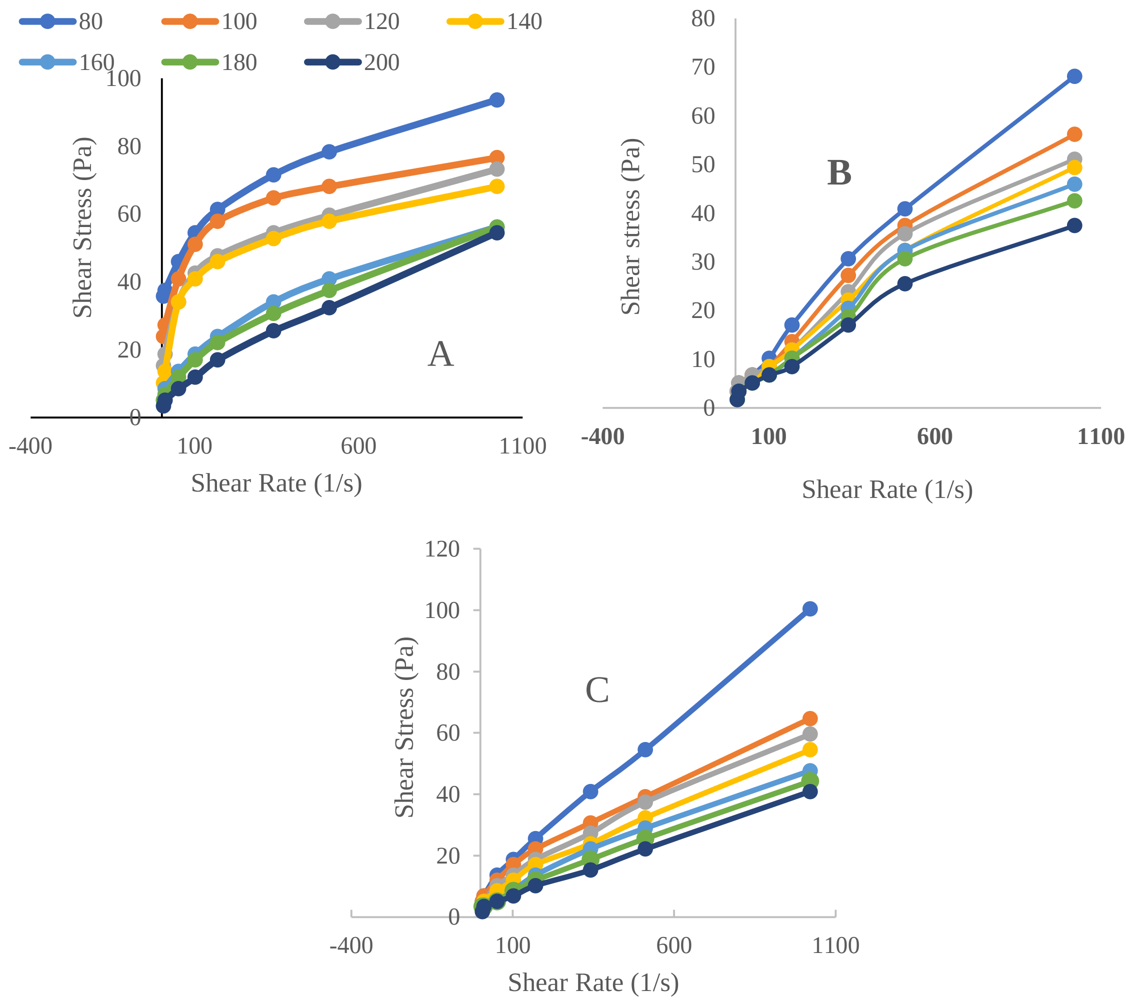

Fig 4: Shear Rate and Shear Stress Relationship for A) BL B) EL C) RBF mud sample at different temperatures

Shear Rate and Shear Stress Relationship: A plot of shear stress against shear rate for non-Newtonian fluids gives plastic viscosity (PV) as the slope of the graph and yield point (YP) as the intercept (Onojake and Chikwe, 2019), especially fluids whose pattern aligns with Bingham plastic and Herschel Bulkley rheology models. The shear stress–shear rate relationship for all the muds investigated in this research, including that of the reference fluid were found to be nonlinear; implying that the flow curves of the muds follow a non-Newtonian flow behaviour over the range of shear rate investigated. Thus, for all the mud samples, the shear stress increases with increase in shear rate and decreases with an increase in temperature, patterning in line with the Herschel Bulkley's fluid model (Aftab, *et al.*, 2022). The decrease in shear stress as the temperature increases signify that the properties of the RBF mud sample deteriorate as the temperature increases model (Onuh *et al.*, 2019). Also evident is that the rate of change of shear stress to shear rate decreases with increasing shear rate due to the shear thinning effect of the mud samples. As the temperature increases, the rate of change of shear stress to shear rate decreases and tends to linearize as the shear rate increases above 200 S^{-1} (Onuh *et al.*, 2019). The RBF mud has very low yield value, as well as the EL mud, but the BL sample showed better thermal stability than the RBF mud sample (Figure 4), and the EL mud sample has the best thermal stability of all. Thus, the thermal stability of the muds follows the trend EL ＞BL ＞RBF.

Shear Rate and Viscosity Relationship: The relationship between the viscosity of the muds and shear rate was investigated, and the result presented in Figure 5

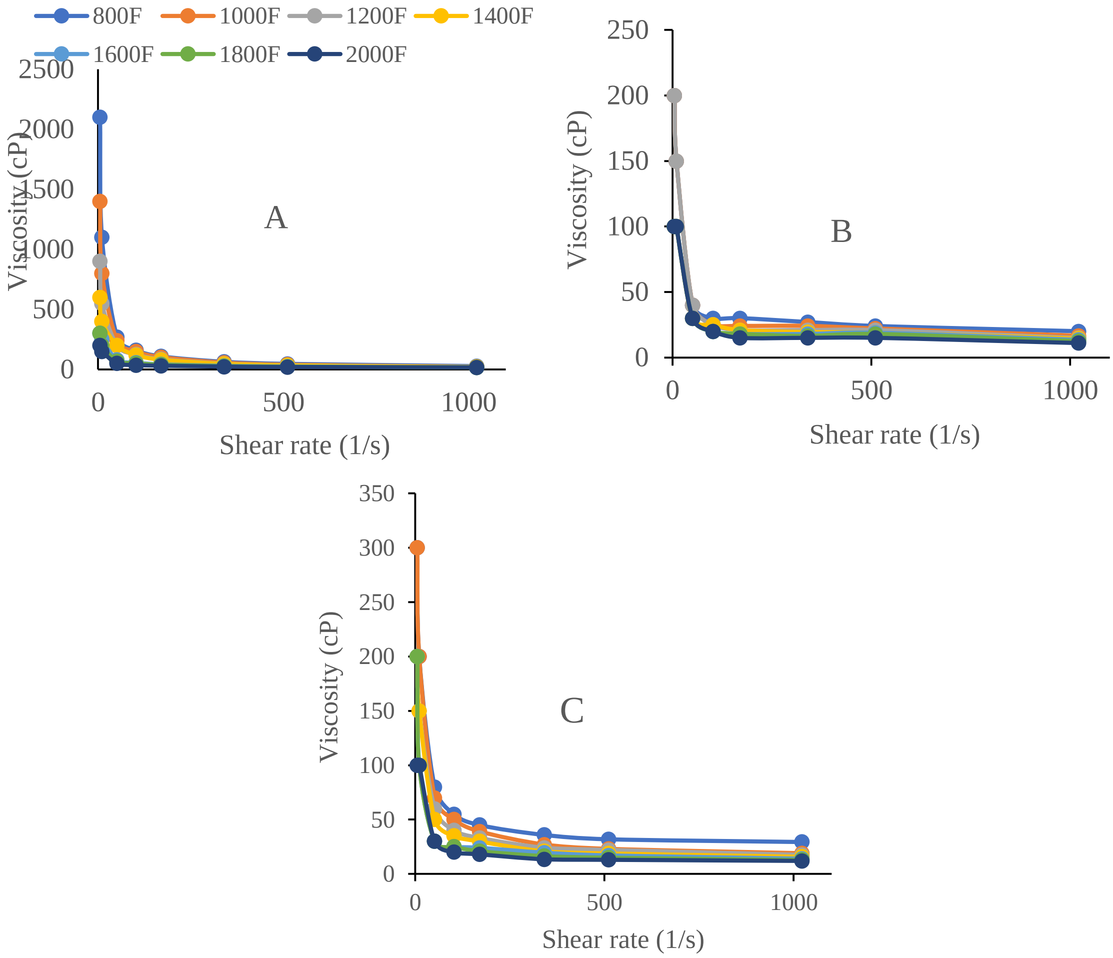

Fig 5: Shear Rate and Viscosity Relationship for A) BL B)EL C)RBF mud sample at different temperatures

The results obtained show that the viscosity of the three mud samples decreases with a thinning effect (Akpan, *et al.*, 2020) as indicated by the shear rate vs shear stress relationship analyzed above. At a lower shear rate, the BL mud sample has a higher viscosity than EL and RBF respectively. This is also reflected in their yield values across the various range of temperatures. This implies that the viscosity of the three mud samples will remain relatively constant as the shear rate increases. Thus, maintaining the pumping efficiency in the circulation of drilling mud as shear rate increases.

Conclusion: In this research, two synthetic ester base fluids were produced, FTIR spectral analysis was employed to identify the functional groups in the esters. Investigation of the physicochemical properties of the esters showed that they have comparable properties relative to a commercial base fluid reference in terms of pH, SG and viscosity at 40 ºF. The flash points and electrical stabilities of the two esters were higher than that of the reference sample. The mud formulations using BL showed better rheological properties than the reference mud at the temperatures under investigation, while EL mud showed promise as a potential ester base fluid since it has better temperature stability than the reference, and its gel strength at 10min. and 10sec. were within the recommended values. The esters synthesized in this work have the capacity to replace commercially available base fluids already employed for the formulation of drilling muds.

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Conflict of Interests: The authors declare that there is no conflict of interest among them in the course of preparing this work.

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