ISSN No:-2456-2165 https://doi.org/10.38124/ijisrt/IJISRT24FEB1047

Formulation of Drilling Muds from Methyl Esters Obtained from the Transesterification of *Vitellaria paradoxa* Oil

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Abstract:- ***Vitellaria paradoxa oil was*** **transesterified at temperatures between 55 and 75℃, different stirring speeds and for 40 minutes each. The properties of the methyl ester were studied. Rate is first order with respect to methyl ester. The methyl esters were then used with other additives to prepare the drilling muds. The rheological properties of the muds were within the American Petroleum Institute (API) acceptable limits. The total filter volumes are less than 40 cm^3 which is the highest volume allowed by API. The filter cakes are between 0.8 and 1.3 cm which fall between the lowest and highest values (0.6 and 2 cm respectively). The muds exhibited flat gel properties which all fall in API's limits with values of 10/10 and 10/11 82 % oil muds. The 85 % mud exhibited flat gel property of 15/15.**

Keywords:- *Vitellaria paradoxa, Methyl Ester, Drilling Mud, Transesterification, Bentonite.*

I. INTRODUCTION

The search for greener products for well bore drilling that will achieve a good level of performance particularly with local materials is being intensified (Belluco and De Chiffre 2001). Although, substantial strides have been made in the development of environmentally friendly, sound performance chemistry for corrosion inhibition, emulsifiers etc, much has not been done in the development of such mud systems with local materials. Drilling operations rely heavily on the use of water based muds, (Acevedo, 2007) and (Darly and Gray, 1988). Researches on drilling with vegetable-based cutting fluids (VBCFs) are limited. Therefore, it is necessary to develop and evaluate performance of VBCFs. Most of the studies about VBCFs have been focused on canola oil. The performance of canola-based cutting fluids was tried with various machining operations and compared with other commercial cutting fluids (Belluco and De Chiffre 2004).

Toxicity of drilling muds differs depending on the sources of materials they are prepared from.. The disposal of some of the drilling muds having high toxicity are expensive. When drilling muds are to be disposed, the safety of the environment in which it is to be dispose should be a priority. Now, at the beginning of the 1990's, disposal restrictions are becoming more stringent and restraints are becoming worldwide issues. Mineral oil based muds has been the mud of choice for many environments because of their better qualities (Fadairo 2012). During drilling the environmental suffers a lot thereby disrupting the ecosystem as a result of poor disposal of drilling muds and oil spillages thereby affecting the sources of livelyhood of the host communities. (Fadairo 2012). For this reason, the Environmental Protection Agency (EPA) and other regulatory bodies are imposing increasingly stringent regulations to ensure the use of environmentally friendly muds (Fadairo 2012). Due to increase in environmental legislation the need for safer drilling fluid has become necessary, drilling fluid that will not have effects produced by the oil based drilling fluid even if it is in the environment. Such environmentally friendly drilling mud requires biodegradable materials such as methyl esters which were used in this work. This research is aimed at producing such drilling mud. The researchers obtained such drilling mud through transesterification of *Vitellaria paradoxa* seed oil.

II. MATERIALS AND METHODS

Materials required were locally sourced. The *vitellaria paradoxa* seeds were collected by the researchers and identified at the school herbarium.

- *Sample Collection*

The seeds of ***Vitellaria paradoxa*** were collected from Zin, Marama in Hawul Local government area of Borno state and were partly extracted by solvent extraction and some by maceration.

- *Extraction*

One hundred grammes (100 g) of crushed plant seeds were measured out and packed into the main chamber of the Soxhlet extractor and about 300 cm^3 of n-Hexane was pour into round bottom flash. The heating mantle was turned on and the system heated between 69 ºC to reflux. The solvent vapour travelled up the distillation arm, and flood into the chamber housing the grounded solid material. The condenser

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condensed the solvent vapour, and the vapour dripped back down into the chamber housing the solid material. Then at a certain level, the siphon empties the liquid into the flask. This cycle continued for three hours or until when the sample is observed to contain no oil again. The mixture of oil and hexane was separated via simple distillation.

➢ *Transesterification Process*

Base catalyzed transesterification was carried out using various oil-to-soduim methoxide mole ratios, refluxed with constant stirring at different temperatures 50, 55, 60, 65, 70 and 75 ºC in a 250 cm^3 three flask for forty minutes. For the reaction to occur, therefore, the reactants were stirred continuously by a hotplate stirrer. At the end of the forty minutes the reaction mixtures were transferred to separating funnel and allowed to stand for at least three hours for phase separation. Glycerol, water, unreacted oil and catalyst settled at the bottom while methyl ester at the top. The bottom layer was then removed and the methyl ester was washed with water and dried by passing over magnesium sulphate and the volume measured. The process was repeated three times in each case to insure reproducibility. This process was done in triplicate for all the temperatures mentioned above.

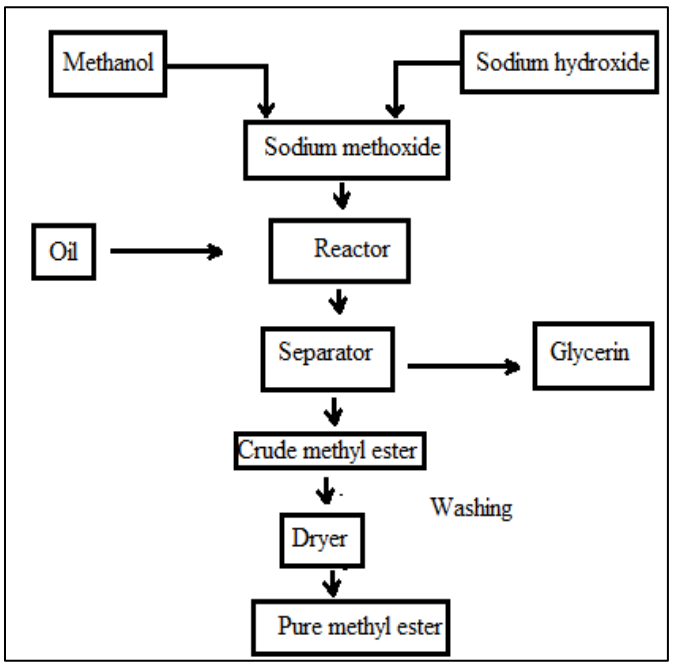

Fig 1: Flow Diagram for methyl ester production

➢ *Determination of Acid Value*

The acid value was determined following the American Oil Chemists Society (AOAC, 1980).

➢ *Specific Gravity Determination*

A clean and dry density bottle was used to determine the specific gravity of the butter after been melted. The empty density bottle was weighed then filled with oil, and water. The value for the weighed density bottle when filled with oil and when filled with water was then compared with that of empty density bottle and the specific gravity was then calculated.

➢ *Determination of Flash Point*

20 cm^3 of the sample was heated in a test cup at a slow and steady rate and continuous stirring. Flame was directed into the cup from time to time. The flask point was taken at the lowest temperature at which the application of the test flame caused the vapor above the sample to ignite momentarily.

➢ *Determination of Cloud Point*

10 cm^3 of oil sample was placed into a 15 cm^3 conical flask and transferred into a refrigerator at 10 °C and observed every 30 minutes until a cloud was observed. The temperature at which the cloud was observed was taken as the cloud point.

Table 1: Properties of the Extracted *Vitellaria paradoxa* oil

Property	Values
Colour	Light yellow
Melting temperature (˚C)	36
Density (kg/m^3)	0.863 (at 35˚C)
Flash point	337
Free Fatty Acid (FFA) value (%)	2.1
Kinematic Viscosity (mm^2/s)	38.7

Some properties of the *Vitellaria paradoxa* oils are presented in table above. It is light yellow in colour. The kinematic viscosity of *Vitellaria paradoxa* oil was found to be 38.7 mm^2/s. Ajala et al., (2017) conducted such studies and obtained 30.68 mm^2/s.

➢ *Free Fatty Acid Values*

The determined value was (2.1 %) similar to 1.84 obtained by Datti *et al.*, (2020) and 2.279 % obtained by Jude and Benjamin (2012). The direct base-catalysed method was used since direct base – catalyzed method is recommended for oils with free fatty acid value less than 5.0 % (Enweremadu and Alamu, 2010).

➢ *Catalyst Loading*

Figures 3 presents the variation of biodiesel yield while varying the amount of sodium hydroxide dissolved in methanol to form the sodium methoxide used as the catalyst in the transesterification of *Vitellaria paradoxa*. In order to determine the amount of sodium hydroxide to be used, the effect of variation of the amount of sodium hydroxide on conversion was studied after Kumar *et al.*, (2010) and Kilonzi *et al.*, (2015). Moles of NaOH were varied in a litre of methanol from 0.01 M to 0.035 M.

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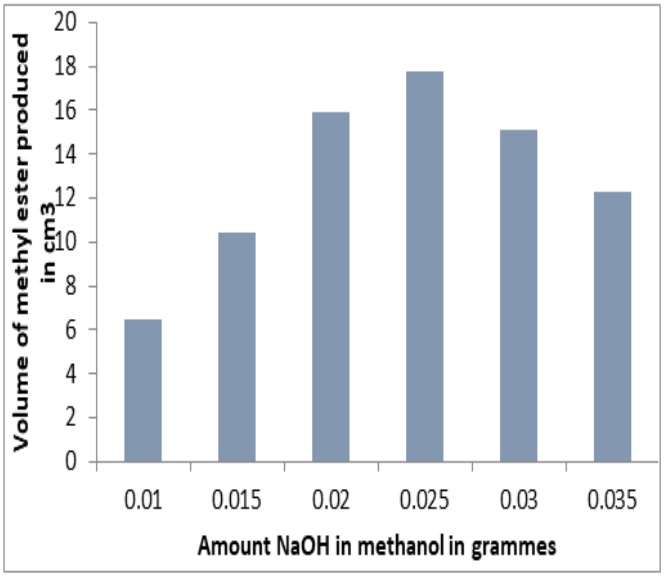

Fig 2: Variation of yield with Catalyst loading for Vitellaria paradoxa oil

The conversion increased at first with increasing moles of sodium hydroxide from 0.01 M to 0.025 M then starts to decrease for from 0.03 M and 0.035 M for due to soap formation which is in line with the work of Enweremadu and Alamu (2010). Since 0.025 M gave the highest biodiesel yield with minimum soap formation it was then used for the transesterification.

➢ *Variation of Methyl Ester Yield with Time*

Figures 3 shows the effect of time of reaction on methyl ester production which shows that methyl ester forms fast between the first minute to the 25th minutes.

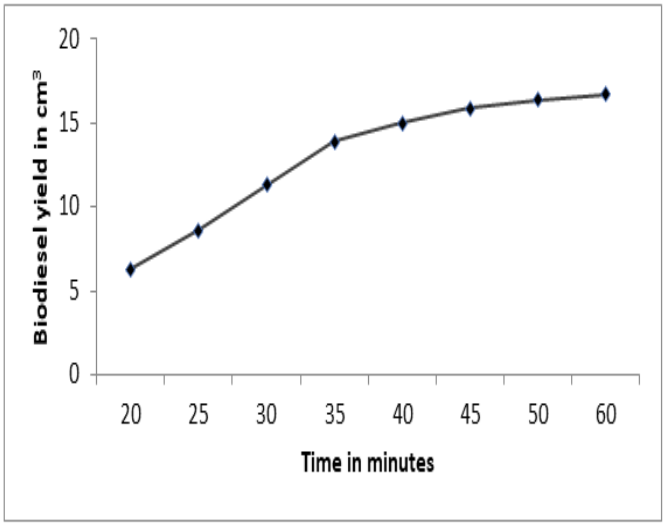

Fig 3: Methyl Ester Yield Against Time

➢ *Volume of Methyl Ester Yield with Increasing Temperatures*

Reactions were carried out at temperatures between 50 ^{o}C and 75 ^{o}C at intervals of 5 ^{o}C as shown in Figure 5 which reveals that methyl ester volume increases for *Vitellaria paradoxa* oil as the volume oil volume is varied as seen in the figure. This agrees with the studies of George *et al.*, (2009) and Gajendra *et al.*, (2010) when producing biodiesel from coconut oil using methanol.

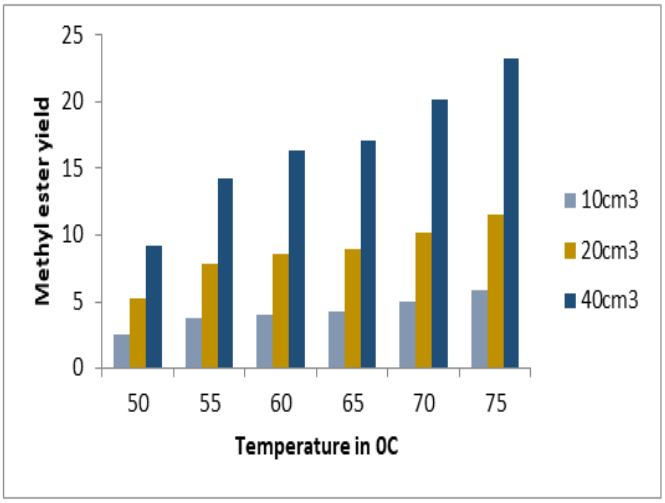

Fig 4: Variation of methyl ester yield with temperature with varying oil volume

➢ *Variation of Methyl Ester Yield with Stirring Speed*

The highest biodiesel yield was obtained when the reaction is stirred at 700 rpm. As the speed increase above 700 rpm the yield decreased. The decrease is as a result of emulsion formation as the speed gets high. This was also observed in the transesterification of coconut oil by Ganjendra et al., (2010) where 800 rpm gave the highest yield of biodiesel and start to decrease with higher stirring speed. This is due to emulsion/soap formation between the reactant instead of reacting.

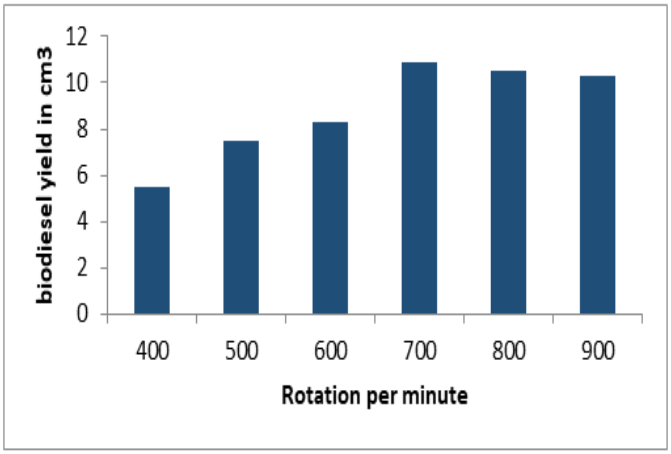

Fig 5: Effect of Stirring Speed on Methyl Ester Yield

ISSN No:-2456-2165 https://doi.org/10.38124/ijisrt/IJISRT24FEB1047

➢ *Properties of Methyl Esters*

Table 2: Properties of the Methyl Ester

S/N	Property		Values	
		Researchers	Eweremadu (2011)	ASTM 6751
1	Density (kg/m^3) at 35°C	6.47	8.77	7.328
2	Specific Gravity	0.87	3	0.88
3	Pour point ($^\circ$C)	5	6	-15 to 10
4	Cloud point ($^\circ$C)	8	4.42	-3 to 12
5	Kinematic Viscosity (mm^2/s)	4.52	171	1.9 - 6.0
6	Flash point ($^\circ$C)	167		130 to 170

The properties of the methyl ester obtained from transesterification as shown in Table 2 compares well with the studies of Eweremadu (2010), Eze and Ejilah (2010) and ASTM 6751 values. The specific gravity is 0.87 which is in line with the work of Datti *et al.*, (2020) though its slightly higher than the ASTM 6751standard value by 0.004 which may be due to residual triglycerides.

➢ *Specific Gravity Determination*

An empty density bottle was weighed then filled with oil and weighed. The same bottle was then filled with water and weighed. The value for the weighed density bottle when filled with oil and when filled with water was then compared with that of empty density bottle and the specific gravity was then calculated.

➢ *Drilling Mud Formulation*

The method of Fadairo *et al.*, (2012) was adopted with slight modification. Exactly 4.5 grammes of texapon was weighed in a beaker and 200 cm^3 of methyl ester was measured and emptied into the beaker containing the texapon and placed on a hot plate stirrer. It was heated at 70˚C with continuous stirring then 5.5 grammes of cetyl alcohol was then added and heated to 100 ˚C with continuous stirring until the cetyl alcohol melted. Then 100 cm^3 of water was added and stirred to a colloidal solution. Then 2 grammes of bentonite, 4.5 grammes of barite, 3 grammes of calcium hydroxide and 5 grammes of calcium chloride were added subsequently in that order with continuous stirring with each addition. Other formulations were carried out following the same method.

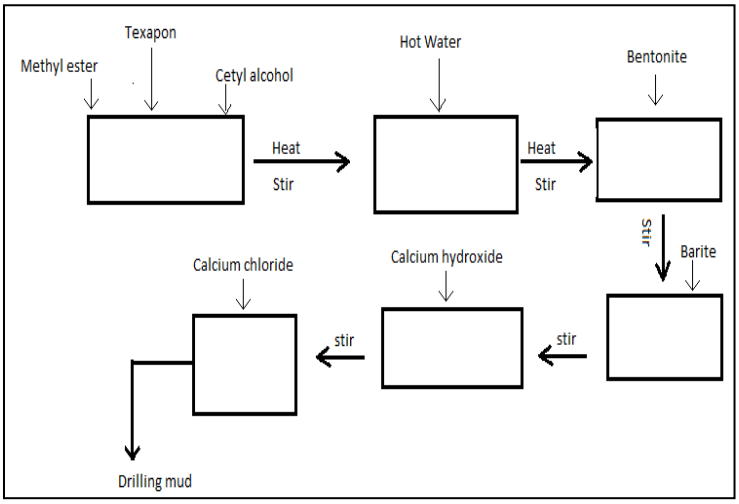

Fig 6: Flow Diagram for Drilling Mud Formulation

Table 3: Quantities of the Ingredients used in Formulation

S/N	Ester(cm^3)	Water (cm^3)	Cetyl alc. in grams	Texapon in grams	Bent in grams	Barite in grams	Ca(OH)$_2$ in grams	Fe$_2$0$_3$ in grams	CaCl$_2$in grams	Ratio of oil to water
1	200	45	10	8.0	11	14.5	4	6	5	82/18
2	350	60	7.1	9.3	12	18.5	5	6	6	85/15

➢ *Determination of Flash Point*

10 cm^3 of oil sample was placed into a 15 cm^3 conical flask and transferred into a refrigerator at 10 °C and observed every 30 minutes until a cloud was observed. The temperature at which the cloud was observed was taken as the cloud point.

➢ *Determination of Cloud Point*

10 cm^3 of oil sample was placed into a 15 cm^3 conical flask and transferred into a refrigerator at 10 °C and observed every 30 minutes until a cloud was observed in the temperature at which the cloud was observed was taken as the cloud point.

➢ *Determination of Mud Weight*

The procedure of Okorie *et al.*, (2015) was employed. A Fann mud balance model 140 was calibrated using water. Then the mud balance cup was filled to the top with the mud sample. The lid was placed on the cup and was turned to ensure that it was firmly put in place. Excess mud spilled through the vent was wiped off from the lid. The balance was placed on a knife edge and the rider was moved along the graduated arm until the cup and the arm were balanced as indicated by the bubble. The mud weight in pounds per gallon (lb/gal) was read at the edge of the rider towards the mud cup as indicated by the arrow on the rider and recorded. The procedure was repeated with the next sample.

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➢ *Measurement of Viscosity, Gel Strength, Plastic Viscosity and Yield Point*

Viscosity, gel strength, yield point and plastic viscosity are measured at room temperature and atmospheric pressure with a FANN 35A viscometer.

➢ *Filtration Loss Determination*

The rate at which fluid is forced out from a filter press under specific conditions of time, temperature and pressure was monitored and the after which the thickness of the filter cake deposited on a filter paper was measured (Kamal 2015).

➢ *Rheological Properties.*

The plastic viscosities (PV), the yield points (YP) and apparent viscosities (AV) all fall within the API accepted values for a good drilling mud.

Table 4: Plastic and Apparent Viscosities and Yield Point for *Helianthus annuus* methyl ester drilling mud

Properties	**Percentage of methyl ester in the drilling fluids**		
	82	**85**	**API values**
PV	37	37	25-50
YP	11	18	10-19
AV	42.5	44	30-50

➢ *Yield Point*

The Yield points are presented in Figure 3 in which all met the required API standard which implies that the formulation of 82/18 and 85/15 oil/water ratios are good to serve as drilling fluids though Onojake and chikwe (2019) are of the opinion that low yield lead to better drilling performance.

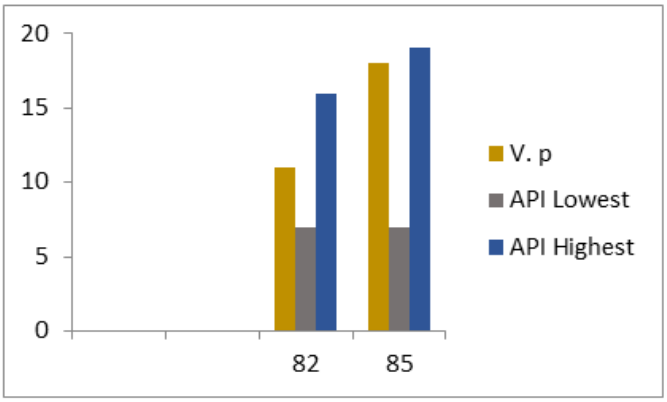

Fig 6: Yield Point of the Formulated Fluids

➢ *Gel Strength*

From Figure 7 The gel strength of muds is almost attending flat gel with gel_0 and gel_{10} almost the same implying that they can be used efficiently in drilling. If the gel strength of a drilling mud at 10-minute is higher than the 10-second gel value, indicating that the mud exhibits a progressive gel structure which is an indication that the gelation of the mud is rapidly gaining strength with time, which is an undesirable feature of a drilling mud, therefore the muds the formulated muds can be used in drilling. The properties are desirable during drilling operation as the gel can be broken easily with lower pump pressure to make circulation (Okorie *et al*., 2015).

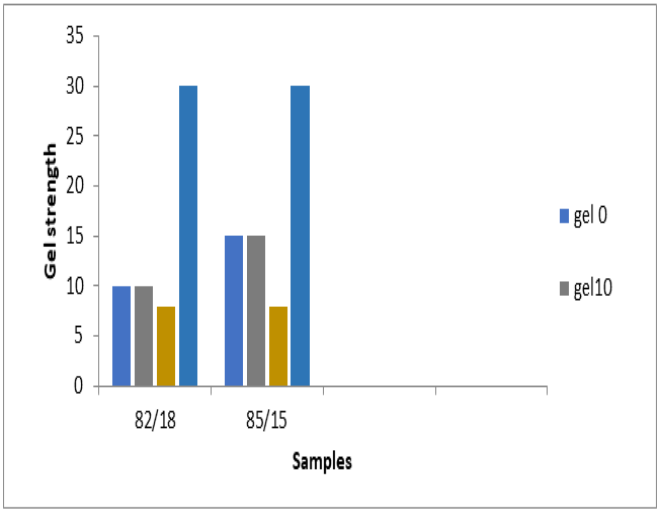

Fig 7: Gel Strengths of the Formulated Drilling Fluids

III. CONCLUSION

From the data collected and analyzed it is clear that the formulated drilling muds are fit for drilling both in terms of safety (since methyl esters are not harmful) and effective as the rheological properties are in line with API standards.

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