Low Oil-Water Ratio Invert Emulsion Mud for Unconventional Shale Reservoirs

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Acknowledgement

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Declaration

"**I, PEACE CHIKAODI EGEJURU,** declare that this thesis is submitted in fulfilment of the requirements for the degree of PhD at the University of Salford. The work described in this thesis is my own work. Any section, part, or phrasing of more than consecutive words that is copied from any other work or publication has been clearly referenced at the point of use and fully described in the reference section of this thesis"

Peace Chikaodi Egejuru

Supervisors (Joint)

Dr M. Burby **Prof. G.G Nasr**

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Nomenclature

AP	Apparent viscosity
API	American Petroleum Institute
ASME	American Society of Mechanical Engineers
Bbl	Barrel
$CaCO_3$	Calcium carbonate
$CaCl_2$	Calcium chloride
CaO	Calcium oxide
CO_2	Carbon dioxide
cP	Centipoise
DOBM	Diesel oil based mud
ES	Electrical stability
EY	Egg yolk
H_2S	Hydrogen sulfide
H_2SO_4	Sulfuric acid
HCO_3^-	Bicarbonate ion
HTHP	High temperature high pressure
JOBM	Jatropha oil based-mud
LCD	Liquid crystals display
OBM	Oil based mud
OH^-	Hydroxide ion
OWR	Oil-water ratio
ppg	Pounds per gallon
PV	Plastic viscosity
ROP	Rate ofpPenetration

RPM	Revolution per minute
SBM	Synthetic based mud
SG	Specific gravity
Tcf	Trillion cubic feet
T	Temperature
V	Voltage
WBM	Water based mud
$\acute{y}$	Shear rate
YP	Yield point
τ	Shear stress
μ	Viscosity

Conversion Table

In the oil and gas industries, it is a normal practice that measurements are made in imperial units. This work adopted the SI units of measurements, however, where it was absolutely necessary, the imperial units were used. The conversion units used in this work are listed below.

Parameters	SI Units	Other Conversion Factors
Length	1 m	1000 mm 3.2808333 ft. 39.37 in 1.09361yd
Area	1m^2	10.76387 ft^2 1550 in^2
Volume	1m^3	1000 litres 6.28983 bbl
Viscosity	1 Ns/m^2	1000 Cp
Pressure	101.325 KPa	1 atm 0.101325 Mpa 14.5 psi 14.7 psi
Temperature	0˚C	32˚ F 273.15
Mass	1 Kg	1000 g
Time	1 day	24 hr 86400 s

Publications and Conferences

1 **Ihenacho, P.C,** Burby,M and Nasr,G.G, (2015) Mitigating The Environmental Implications of Drilling Operations in Unconventional Shale Reservoirs. *Salford Postgraduate Annual Research Conference (SPARC 2015)*

2 **Ihenacho, P.C.**, Burby,M., Nasr, G.G and Enyi,G.C., (2016) Utilisation of Egg Yolk as a Non-Toxic Emulsifier in Environmentally Friendly Invert Emulsion Drilling Mud. *Journal of Petroleum Engineering & Technology 2016:6 (1): 41-49p*

3 **Ihenacho, P.C.,** Burby, M., Nasr, G.G and Enyi,G.C. (2016) 50/50 Oil-Water Ratio Invert Emulsion Drilling Mud Using Vegetable Oil as Continuous Phase. *International Journal of Chemical, Molecular, Nuclear and Metallurgical Engineering. Vol.10, No3, 2016*

4 **Ihenacho, P.C.,** Burby, M., Nasr, G.G and Enyi,G.C. (2016) 50/50 Oil-Water Ratio Invert Emulsion Drilling Mud Using Vegetable Oil as Continuous Phase. *18th International Conference on Oil, Gas and Petrochemical Engineering. Rome, Italy March 21-22,2016*

5 **Ihenacho, P.C.,** Burby,M., Nasr, G.G. and Enyi,G.C. (2017) Economic Evaluation of Environmentally Friendly Vegetable Oil-Based Invert Emulsion Drilling Mud**.** *Journal of Engineering Technology. Journal of Engineering Technology (ISSN:0747-9964) Vol 6, issue 2, July,2017, pp776-783*

6 **Ihenacho, P.C**., Burby,M. and Nasr,G.G Shale-Fluid Interaction in Environmentally Friendly Invert Emulsion Using an X-Ray Computed Tomography (CT) Scan. *Petroleum Science PETROSCI-2016-0355* (Under Review)

Abstract

Due to stringent environmental regulations on the disposal and management of the traditional diesel oil based mud used for drilling difficult formations such as shale, there is the necessity to develop an environmentally friendly drilling mud. Vegetable oils such as Jatropha have proven to be a comparable alternative to diesel oil. However, there have been concerns of compatibility of the vegetable oils with the chemical additives and the ability to achieve a low oil-water ratio mud, which is beneficial to low fluid loss for enhanced wellbore stability.

The focus of this study is to achieve a novel low oil-water ratio invert emulsion using Jatropha oil and egg yolk as an emulsifier. Shale-fluid interaction and the economic viability of the mud were also evaluated. The findings from this study show that the low oil-water ratio invert emulsion is beneficial to reducing fluid loss for enhanced wellbore stability and the reduction of oil retention on cuttings, thereby reducing cost of disposal and environmental impact. The mud was formulated without a fluid loss additive, wetting agent, secondary emulsifier and the need for high water content, thereby savings could also be made in material costs.

The results from this experimental study demonstrated that the electrical stability of the mud emulsified by egg yolk at a test temperature of 48.9 and 120 ˚ C for any variation, were 398 and 289V respectively. Comparatively, the mud emulsified with a standard emulsifier *versacleanVB* gave stability values of 201 and 188V thus indicating higher stability with the egg yolk. Moreover, the 50/50 oil-water ratio mud gave stability values of 353 and 258V hence, giving plastic viscosity of 36 cP, at the yield point of 17 Ib/100 ft^2 and 30 minutes fluid loss of 6ml with filter cake of 1.7 mm. This resulted to 50% reduction in fluid loss over the conventional 70/30 oil-water ratio mud, which was emulsified with the standard emulsifier with stability values of 480 and 393V, plastic viscosity of 31cP, yield point of 17 Ib/100 ft^2 and fluid loss of 12 ml with filter cake of 3.2 mm.

Using the X-Ray Computed Tomography (CT) Scanner, the shale-fluid interaction also showed a volume increase by 11 and 23% of the core sample when immersed into water based mud from one to 7 days respectively. The result indicates that a high interaction with the fluid are possible, thus leading to a non-stable condition compared to 0.88 and 2.53% obtained from diesel and jatropha oil based muds. There was negligible variation in the structure of the samples exposed to diesel and jatropha oil based muds, which further suggests the suitability of jatropha as diesel substitute.

The economic analysis of 50/50 oil-water ratio invert emulsion equally showed a potential saving of 57.91% of the $65.31 of the cost of formulation and 47.5% of $60 of the cost of disposal of the conventional diesel oil-based mud. This has the potential to equate to saving of $37.82 per barrel of invert emulsion formulated and $28.50 per barrel disposed.

Chapter 1

Introduction

The rapidly growing energy demand worldwide and the depletion of existing reserves have resulted in a major gap between supply and demand. This situation has necessitated the exploration and development of unconventional reservoirs in order to meet the need for affordable energy. However, the development of these unconventional reserves involves a number of environmentally challenging considerations. These involve drilling vertically or horizontally into the shale formation using oil-based mud, and hydraulically fracturing the formation using a combination of water, sand and chemical additives. The challenges also include the recycling or disposal of the oil based mud, and flow back water or formation water produced during the fracturing operation. Therefore, to sustain a continuous energy supply to the global community by developing these unconventional resources, the environmental issues must be timely addressed. One of the promising approaches is the development of an environmentally friendly low oil-water ratio invert emulsion mud for the drilling operation, which is the main focus of this research investigation.

Generally, there is no formal standard definition for unconventional resources, they have been defined or classified from different perspective by different groups based on the mode of development, interpretation of geology or petroleum system. Unconventional reservoirs according to Etherington and McDonald (2004) are the ones that cannot produce at economic flow rates without the assistance from massive stimulation treatments or special recovery processes. These include tight gas sands, low permeability oil system, heavy oil, gas shales coal bed methane, oil shale and gas hydrates. Chan et al. (2012) classified unconventional and conventional resources based on the effect of hydrodynamic influences. Unconventional resources exist in hydrocarbon accumulations that are pervasive throughout a large area and are generally not significantly affected by hydrodynamic influences (also called continuous – type deposits). The recovery of the petroleum requires specialised extraction technology and in the case of oil, the raw production may require significant processing before sale.

While conventional resources are those that exist in discrete petroleum accumulation related to localised geological structural feature and or stratigraphic condition, typically with each accumulation bounded by a down-dip contact with an aquifer, and that is significantly affected by hydrodynamic influences based on buoyancy of petroleum in water. The petroleum is recovered through wellbores and typically requires minimal processing before sale. Unconventional reservoirs may be divided into three types: tight gas, Coal-Bed Methane and Shale gas or oil (Soliman and Kabir, 2012), Shale gas is naturally fractured and usually contains both the free and adsorbed gas.

With many shale reserves being in environmentally sensitive and or densely populated areas, the traditional drilling fluids design and the use of strong chemical inhibition or conventional invert emulsions may not meet future environmental requirements, (Young and Friedheim, 2013). The development of these unconventional reserves involve a number of environmentally challenging considerations involving: drilling vertically or horizontally into the shale formation using oil based mud; hydraulically fracturing the formation using combination of water, sand and chemical additives; recycling or disposal of the oil based mud used in the drilling operation and flow back water or formation water produced during the fracturing operation. Environmental concerns and wellbore instability are the most significant problems associated with development of unconventional shale. Major wellbore construction related issues in unconventional shale reservoirs are borehole instability, hole cleaning and rate of penetration. Shale instability is a major cause of wellbore problems in drilling operations.

1.1 Problem Statement

Despite the continuous efforts put in place to develop unconventional shale reservoirs in a cost-effective environmentally friendly manner, the development has been hampered by persistent environmental implications associated with the drilling operations. Borehole instability, hole cleaning and rate of penetration which are the major wellbore construction related issues in these unconventional shale reservoirs have also persisted as a major global concern in the petroleum industry. Drilling and fracturing produce large amounts of waste water, which may contain dissolved chemicals and other contaminants that require treatment before disposal or reuse. The use of potentially hazardous drilling fluids and chemicals means that any release of these fluids can result in the contamination of surrounding areas, including sources of drinking water, and can negatively impact natural habitats.

Developing a high performance environmentally friendly invert emulsion mud is one of the promising ways to mitigate these issues. Oil-based mud is the preferred drilling mud especially in highly technical and challenging formations such as shale. This is due to its sterling properties including excellent borehole control, thermal stability, high lubricity, penetration rates, and greater cleaning abilities with less viscosity and enhanced shale inhibition which is critical in shale formations for multi-stage fracking. These properties may help to reduce operational costs; however, there are several environmental issues associated with its disposal and hazards to personnel. This has continued to pose challenging issues due to stringent environmental legislation over its disposal. It has become imperative for the drilling fluids industry to provide oil-based drilling fluids using alternative base oils to diesel and mineral oils that will be environmentally friendly while maintaining high technical competence. Over the years several plant oils such as rapeseed oil, mahua oil, cottonseed oil, Sesame oil, Soya bean oil, palm oil, canola oil, moringa seed oil, soapnut and Jatropha have become popular as diesel substitute because of their low toxicity. However, there have been conflicting issues with maintaining the mud technical properties such as good rheology, high stability, low fluid loss and compatibility of the chemical additives with the vegetable oil continuous phase. Apaleke et al. (2012) stated that toxic additives are the high performers but need to be replaced. Among the most important research questions which remain unanswered are:

- Considering toxicity, compatibility and sustainability what is the possibility of replacing the high performers' toxic additives with non-toxic ones of technical competence?
- Which drilling fluid property is a major cause of wellbore instability?
- A low oil-water ratio of 50/50 is beneficial in achieving low fluid loss which is good for wellbore stability and also economical, with vegetable oil continuous phase, what is the possibility of achieving an oil-water ratio below 70/30 while maintaining good rheology and stable emulsion?

1.2 Contribution to Knowledge

The realisation that a low oil-water ratio mud can be developed using vegetable oil and replacement of conventional chemical additives such as emulsifier with an organic supplement could be an innovative concept. The overarching contribution to knowledge of this investigation is to provide a low oil-water ratio invert emulsion mud, with underpinned rheological, filtration and consistent properties for enhanced wellbore stability and effective cuttings removal. This can also lead to reduction in mud disposal costs and environmental impact.

1.3 Aims

The aims of this study are as follows:

1) The development of a low oil-water ratio (50/50) invert emulsion with conventional emulsifier substituted with egg yolk.
2) The reduction of cost of drilling operations by reducing the cost of formulation and disposal of the conventional diesel oil-based mud

1.4 Objectives

The objective of this experimental study is as follows:

i) To analyse the invert emulsion using vegetable oil and egg yolk as a non-toxic emulsifier
ii) Comparison of mud emulsified by a standard emulsifier and mud emulsified using egg yolk.
iii) Comparison of 70/30 and 50/50 oil-water ratio mud
iv) Shale-fluid interaction by immersion testing and characterisation using CT scanner and Volume Graphics (VG) Studio Max 2.2 software.
v) Economic consideration of the invert emulsion mud in relation to cost of formulation and disposal.

1.5 Thesis Outline

This thesis comprises of the following chapters:

Chapter 2: is the literature review providing an overview of the development of unconventional shale reservoirs, drilling fluids classification, properties, functions, additives, including choice of vegetable oil for invert emulsion. Rheological models and wellbore instability in shale are also discussed

Chapter 3: describes mud formulation and full experimental analysis of the properties of invert emulsion mud using vegetable oil and egg yolk as a non-toxic emulsifier, while achieving 50/50 OWR mud. Shale-fluid interaction by immersion test was also investigated

Chapter 4: discusses the results of the experimental findings of the research comparing properties of diesel and jatropha oil-based muds, emulsifying ability of egg yolk compared to a standard emulsifier-VersacleanVB. The properties of a novel 50/50 OWR invert emulsion was compared with 70/30 OWR mud and shale-fluid interaction in water based mud, diesel and jatropha oil-based muds are compared.

The economic viability of environmentally friendly invert emulsion mud considering the cost of formulation and cost of disposal of one barrel of diesel and of jatropha oil-based muds was also discussed.

Chapter 5: Summarises this research work relating the experimental results to the aims and objectives and highlighting the major contributions of the study. Recommendations include: characterisation of invert emulsion by emulsion droplet size, effect of fluid chemistry and exposure time on shale strength and comparison of rate of penetration using different nozzle diameter.

Chapter 2

Literature Review

2.1 Introduction

This Chapter presents an overview of drilling fluid in relation to drilling operations in unconventional shale and a review of previous studies in the trend of vegetable oil-based mud in addressing the knowledge gap. The idea of vegetable oil-based mud is to address the environmental issues associated with the conventional diesel oil-based mud. However, the need for non-toxic and compatible chemical additives have not been addressed in previous studies. Achieving a low oil-water ratio mud with the vegetable oil continuous phase is also a major gap in this trend of vegetable oil-based mud. The present study is geared towards addressing this issues.

2.2 Drilling fluid

Drilling fluid, often referred to as drilling mud is a common term used for the different types of fluids used in the drilling of oil and gas wells. In this thesis drilling fluid and drilling mud will be used interchangeably. Growock and Harvey (2005) defined drilling fluid as any fluid that is used in a drilling operation in which the fluid is circulated or pumped from the surface down the hole, drill string, through the bit and back to the surface via the annulus with the capacity of performing some functions. These functions include: removal of drilled cuttings from the wellbore, suspension and release of cuttings and maintenance of wellbore stability. Drilling fluid composition varies based on wellbore demands, rig capabilities, cost and environmental concerns. They are classified according to the type of the base fluid as water, gas or oil-based mud.

Borehole stability remains a major problem in drilling operation and the selection of the right type of drilling fluid and composition is a bedrock for successful drilling operation. (Khodja, 2010). Wellbore instability is major problem in drilling unconventional shale, in the present a low oil-water ratio invert emulsion is aimed at reducing fluid loss which is a major cause wellbore instability in Shale. Proper circulation of the mud is essential for it to effectively perform the desired function. The schematic of the mud circulation system is shown in Figure 2.1.

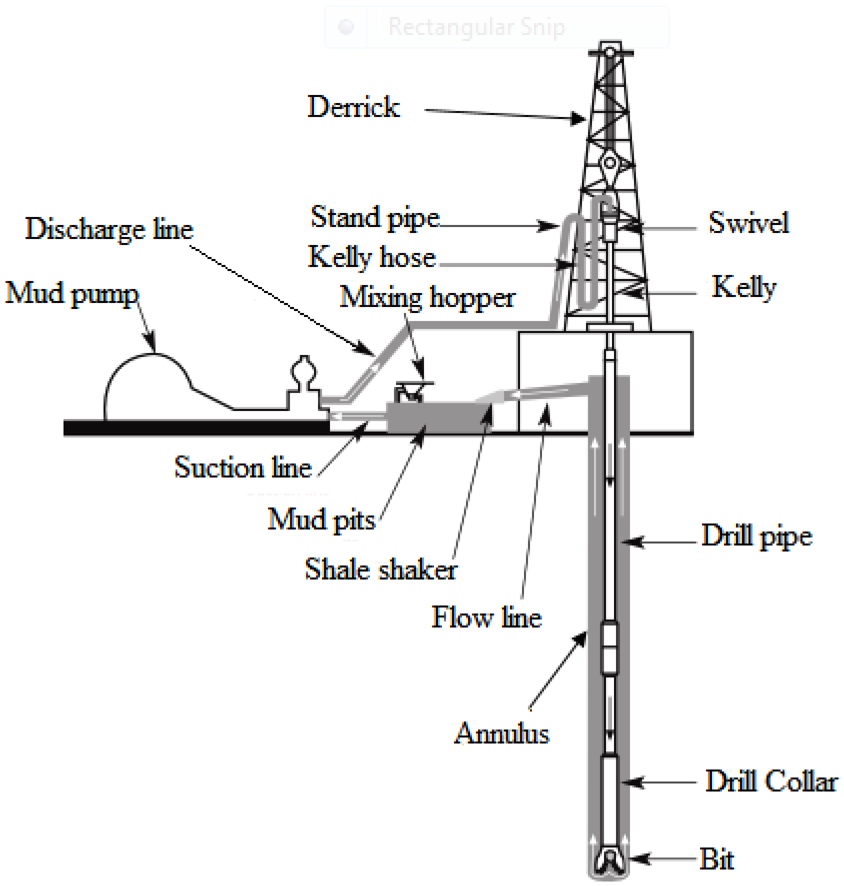

Figure 2.1: Mud circulation system (M-I SWACO, Drilling Fluids Engineering Manual, Houston, TX, 2006)

The mud pump through the *suction line* takes in mud from the mud pits and sends it out in a discharge line to a *standpipe*. The mud is pumped up the standpipe into a flexible reinforced rubber hose called the *rotary hose* or *Kelly hose.* The mud flows through *swivel* down the *kelly, drill pipe* and *drill collars* and exits at the *drill bit*. The mud flows back up the hole in the annulus and finally leaves the hole through a steel pipe called the *flow line* and flows over the shale shaker and back to the mud pit

2.2.1 Emulsion

Emulsion is a colloidal system in which fine droplets of one liquid are dispersed in another liquid where the two liquids are immiscible. The phase that is present in the form of droplets is the dispersed or internal phase, while the phase in which the droplets are suspended is called the continuous or external phase. A stable emulsion is achieved with the use of an emulsifier, which reduces the interfacial tension between the liquids. Two types of emulsion are used as muds: oil-in-water or direct emulsion, which is known as *emulsion mud* and the water-in-oil or invert, which is known as *invert emulsion mud*. An invert emulsion was considered in the present investigation.

2.2.1.1 Invert emulsion mud

Invert emulsion mud is the oil- based mud to which water has been added. In this case oil is the continuous phase and the dispersed phase is water. It can be run with 5 to 50% of water, although some systems are run with 100% oil. According to Wagle et al.(2012) the primary reasons for worldwide use of invert emulsion drilling fluid are the improved wellbore stability and increased rate of penetration while drilling shale formations. Again, while drilling through shale, the water concentration of the invert emulsion fluid is maintained at a lower level than the water concentration of the shale thereby creating an osmotic pressure, which drives the flow of water from the shale to the invert emulsion fluid thereby preventing shale hydration and reducing fluid loss.

A low oil-water ratio is beneficial to low fluid loss and shale stability, for these reasons **i**n this trend of vegetable oil-based mud Apaleke et al.(2012a) noted that a low oil-water ratio mud is a major gap as excessive viscosity may result., which is undesirable for good penetration. In this present study, a 50/50 OWR mud is aimed at reducing fluid loss for improved wellbore stability in shale while maintaining good rheology and stable emulsion.

2.2.2 Properties of drilling fluid

Drilling fluids have several properties to be controlled and maintained to function effectively in any drilling operation and to avoid technical and economic losses. The main fundamental properties are density, rheology and filtration. Others include pH control, lubricity, toxicity and electrical stability as in the case of invert emulsion. The present study will investigate these fundamental properties including the electrical stability which is essential for an invert emulsion.

- Density

  In the present study, mud density and weight will be used interchangeably. The density of drilling fluid is a very important property of drilling fluid as it determines the hydrostatic pressure imposed in the wellbore and the basis for controlling formation pressure during drilling operations. If the mud weight is too high, it can lead to formation fracturing and lost circulation, while a very low mud weight can result in influx of formation fluids which if not controlled can lead to blow out. Density is expressed in pounds per gallon or kilogram per cubic meter (kg/m^3) and is sometimes compared to the weight of an equal volume of water which is known as specific gravity (SG) and in terms of pounds per square inch per foot

- Rheology

Rheology is defined as the study of the deformation and flow of matter. In drilling operations, rheological properties are used to design and evaluate the hydraulics and to assess the functionality of the mud system. The main rheological properties of drilling mud are plastic viscosity, yield point and gel strength. These properties will be investigated in the present study

Plastic viscosity is the part of the flow resistance of the fluid caused by mechanical friction within the fluid. The rate of penetration is directly affected by the plastic viscosity. A low plastic viscosity indicates that the mud is capable of drilling rapidly because of the low viscosity of mud exiting at the bit. Plastic viscosity is calculated from the difference between the 600-rpm and 300-rpm from viscometer dial readings and measured in centipoise (cP) as shown in equation 2.1

$$\eta_{PV} = R_{600} - R_{300} \qquad (2.1)$$

Where

η_{PV} is the plastic viscosity expressed in centipoise

Yield point is the resistance of initial flow of fluid or the stress required to start the movement of the fluid. The yield point is primarily associated with two mud functions: the hole-cleaning capability and the pressure control characteristic of the mud. It is used to evaluate the ability of mud to lift cuttings out of the annulus. A higher yield point implies that drilling fluid has an increased ability to carry cuttings than a fluid of similar density but lower yield point. YP is calculated from the difference between 300-rpm dial reading and the value of plastic viscosity as shown in equation 2.2

$$Y_{PB} = R_{300} - \boldsymbol{\eta_{PV}} \qquad (2.2)$$

Where

Y_{PB} is the yield point expressed in pounds per one hundred square feet (lb/100 ft^2)

- **Gel Strength**

The gel strength demonstrates the ability of the drilling mud to suspend drilled solids and weighting material when circulation is ceased. This is a measure of the thixotropic nature of most drilling fluids, which is the ability to have a gel-like consistency under static condition. The gel strength is usually evaluated at two intervals on the 8-speed viscometer, firstly at 10 seconds and then at 10 minutes, called initial gel strength and final gel strength respectively. Gel strength is expressed in lb/100 ft^2.

Initial Gel Strength is the shear stress measured at low shear rate after a mud has set quiescently for 10 seconds. The 3-rpm reading is recorded after stirring the mud at 600 rpm from a viscometer and the mud is left in a static condition for 10 seconds.

Final Gel Strength is the shear stress measured at low shear rate after a mud has set quiescently for 10 minutes. The 3-rpm reading is recorded after stirring the drilling fluid at 600 rpm from a viscometer and the mud is left in a static condition for 10 minutes.

Gel strength could be described as progressive, high and low-flat. Figure 2.2 depicts that a low-flat gel is most desirable while progressive and high-flat are both undesirable (Hossain and Al-Mejed, 2015). A flat gel describes a situation where the values of the 10-second and 10-minute gels are similar. Flat gels indicate that the mud will remain pump-able with time in a static situation in the hole while progressive situation demands excessive pump pressure to break to circulation. High –flat gel is undesirable because, the situation also requires high pump pressure to return to circulation. The gel strength of the 50/50 OWR mud will be determined to ascertain its behaviour in a static condition as this is essential for wellbore stability.

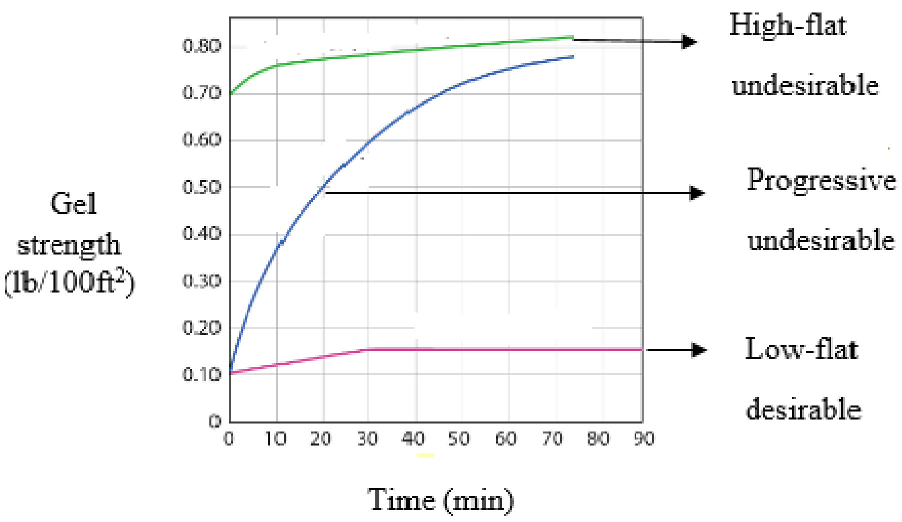

Figure 2.2: Variation of gel strengths with time for drilling muds (Hossain and Al-Mejed 2015)

Fluids are characterised by their rheological behaviour by establishing relationships between applied shear stress and shear rate of the fluid. On the basis of this relationship, two basic types of fluids are identified: those whose viscosity remains constant with changing shear rate and those whose viscosity varies with changing shear rate. Fluids are therefore characterised as Newtonian and non-Newtonian, accordingly.

- ***Newtonian Fluids***

Newtonian fluids are fluids in which the viscosity is constant for different shear rates and does not vary with time. These fluids exhibit a linear relationship between the shear stress and shear rate passing through the origin with viscosity being the constant of proportionality as shown in in Figure 2.3

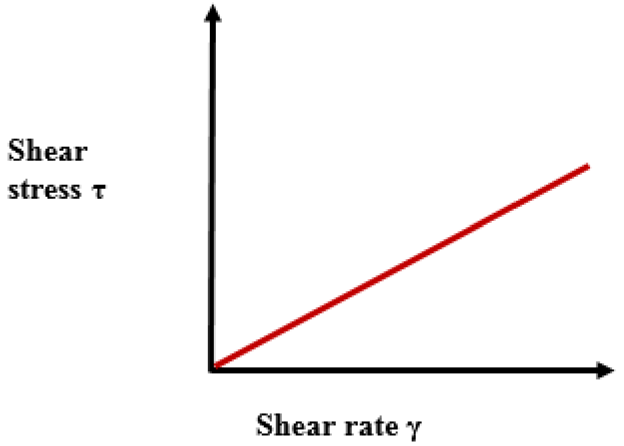

Figure 2.3 Newtonian Fluid Model (Schlumberger oilfield glossary)

The relationship characterising Newtonian fluid is expressed mathematically as shown in equation 2.3

$$\tau = \mu \acute{y} \qquad (2.3)$$

where,

τ = shear stress

$\acute{y}$ = shear rate

μ = viscosity

Examples of Newtonian fluids are air, water, glycerine and kerosene.

- ***Non-Newtonian Fluids***.

Non-Newtonian fluid is fluid whose flow properties differ in any way from those of Newtonian fluids. These are fluids whose viscosity varies with time and shear history. There is no linear relationship between the shear stress and shear rate. Examples of non-Newtonian fluid are blood, polymer solutions, and drilling fluids. The fluid models of this group include Bingham plastic, Power- Law and Herschel Bulkley models, however the model commonly used in the drilling fluids industry is the Bingham plastic model.

- ***Bingham Plastic Model***

The Bingham plastic model is a two-parameter model widely used in the drilling fluids industry to describe flow characteristics of many types of muds. It can be described mathematically as fluids that exhibit a linear shear-stress/shear-rate behaviour after an initial shear stress threshold has been reached. Plastic viscosity (PV) is the gradient of the line and the yield point (YP) is the threshold stress as shown in Figure 2.4

$$\tau = YP + PV\,(\gamma) \qquad (2.4)$$

where,

τ = measured shear stress in lb/100 ft^2

γ = shear rate in sec-^{1}

YP = Yield point

PV = Plastic viscosity

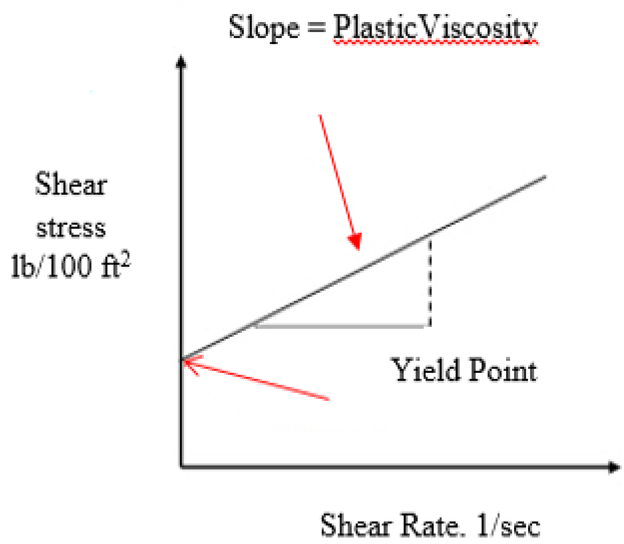

Figure 2.4: Bingham Plastic Model (Schlumberger oilfield glossary)

- **Filtration**

The filtration property of a drilling fluid is the ability of the solid components of the mud to form a filter cake and the magnitude of cake permeability. The lower the permeability, the thinner the filter cake which then lowers the volume of filtrate from the mud. A thick filter cake is undesirable in drilling operations as it constricts the walls of the borehole and allows excessive amount of filtrate to move into the formation. This could result in further problems such as *caving, tight pulls, held ups* and *stuck ups*. In a caving situation, there is excessive cuttings in the hole, which leads to jamming of the bit and consequent collapse of the hole in an attempt to insert the casing. Stuck ups is used to describe the situation when part of the drill pipe or collars are stuck in the hole. In this situation, the pipe cannot be conveniently rotated, or pulled out of the hole without damaging the pipe and exceeding the maximum allowed hook load of the drilling rig. These situations are described as tight pulls and held ups. Therefore, a satisfactory fluid loss value and deposition of a thin, impermeable filter cake are often among the essential factors for successful performance of a drilling fluid. Excessive fluid loss is major cause of wellbore instability in shale. The present study aims to address this issue with a low OWR mud of 50/50. Amanullah, (2005) noted that Duratone used as fluid-loss control additive in mineral oil based muds is not suitable for vegetable oil-based mud, however, a 50/50 OWR mud formulated without a fluid loss additive bridges the gap of incompatible fluid loss additive.

- **Electrical stability.**

The electrical stability represents the mud emulsion stability and wetting ability. It shows the voltage of the current flow in the mud. Oil base fluid is not a conductive material, therefore will not transfer any current. Only the water phase in the mud conducts electricity. A mud with a good emulsion will have a high value of electrical stability. A high stability is important to enable the mud incorporate additional water volume if down hole water flow is encountered. The stability of an emulsion can be enhanced by the addition of surfactant or emulsifier to decrease the surface tension making the droplets more stable. In the present study, egg yolk was used as emulsifier for compatibility with the vegetable oil base fluid.

- **pH control**

pH is potential of hydrogen, which is measure of the degree of the acidity or alkalinity of a solution equal to the common logarithm of the reciprocal of the concentration of hydrogen ions in moles per cubic decimetre of solution. Solutions with a pH above 7 are considered as alkaline while those with pH below 7 are defined as acidic. The desired operating pH for drilling mud is generally 8.5 to 9.5 in order to reduce the corrosion of drill pipe and casing

It is very essential that these properties are maintained throughout the drilling operation, to reduce drilling fluids related problems while drilling. Estimated requirements for oil mud properties according to Lyons and Plisga (2011) is shown in Table 2.1

Table 2.1: Estimated requirements for oil mud properties (Lyons and Plisga 2011)

Mud weight(ppg)	**Plastic Viscosity(cP)**	**Yield Point (lbs/sq ft^2)**	**Oil-Water Ratio**	**Electrical Stability**
8-10	15-30	5-10	65/35-75/25	200-300
10-12	20-40	6-14	75/25-80/20	300-400
12-14	25-50	7-16	80/20-85/15	400-500
14-16	30-60	10-19	85/15-88/12	500-600
16-18	40-80	12-22	88/12-92/8	Above 600

2.3 Drilling of unconventional shale reservoirs

Drilling the wellbore is the first and the most expensive step in the oil and gas industry. Expenditures for drilling represent 25% of the total oilfield exploitation cost and are concentrated mostly in exploration and development of well drilling. In the 90s, drilling operations represented about $10.9 billion, compared with $45.2 billion of the total cost of US petroleum industry exploration and production. (Khodja et.al., 2010) Different methods of drilling could be employed in oil and gas well drilling depending on the hydrocarbon target zone. The major factors affecting the choice of drilling method include reservoir rock characteristics, cost, completion and production methods. Drilling could be done vertically or horizontally as long as the target zone is reached safely and economically. Khodja et al. (2010) also noted that the paramount drilling objectives are to reach the target zone safely in the shortest possible time and lowest possible cost with required additional sampling and evaluation constraints dictated by the particular application

As mentioned in the previous chapter, unconventional shale reservoirs demand the use of special techniques for the production of the reserves to be economically viable. Horizontal drilling, multiple wells from a pad and hydraulic fracturing are the three major methods and are often used together to develop unconventional resources. (Alberta Energy, 2016). ***Horizontal drilling*** is the process of drilling a well from the surface to a subsurface location just above the target oil or gas reservoir called the "kick-off point", then deviating the well bore from the vertical plane around a curve to intersect the reservoir at the "entry point" with a near-horizontal inclination, and remaining within the reservoir until the desired bottom hole location is reached (Helms, 2008 and Curtis, 2011) Figure 2.5 represents horizontal and vertical wells showing greater length of producing formation exposed to the wellbore in the horizontal well than in the vertical well.

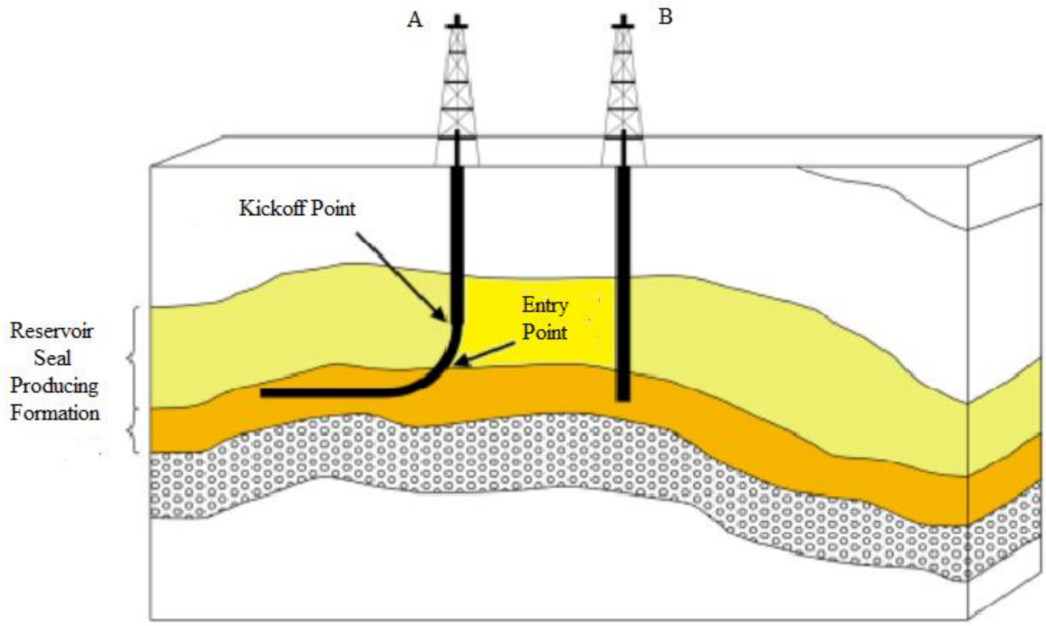

Figure 2.5: Greater Length of Producing Formation exposed to the wellbore in Horizontal well (A) than in a Vertical well (B) (Source: Helms, 2008)

Hydraulic fracturing or "fracking" or "hydrofracking" as it is commonly known has been described as a technique in which water, chemicals, and sand are pumped into the well at a high pressure to unlock the hydrocarbons trapped in shale formations. This is achieved by creating fissures, fractures or small cracks in the underground formations to allow natural gas to flow into the wellbore and upto the surface, where gas is collected and prepared for sale. When used in conjunction with horizontal drilling, hydraulic fracturing enables gas producers to extract shale gas at a reasonable cost. Figure 2.6, represents **a** natural gas well that has been constructed with horizontal drilling through the Marcellus Shale and hydraulic fracturing on the horizontal portion of the well.

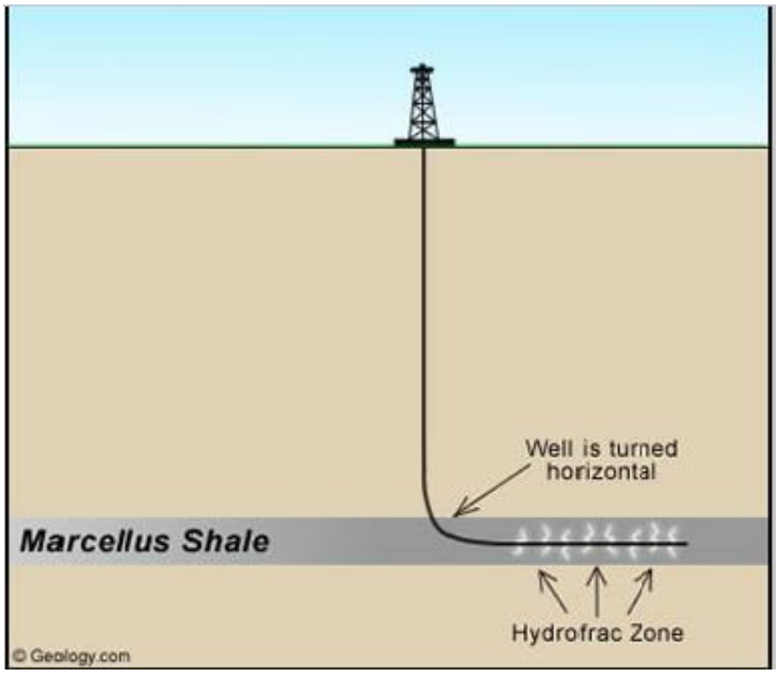

Figure 2.6: Simplified diagram of a natural gas well that has been constructed with horizontal drilling through the Marcellus Shale and hydraulic fracturing on the horizontal portion of the well (http://geology.com/articles/hydraulic-fracturing/ accessed on 02/12/2016)

For sustained utilisation of shale gas to meet the world's energy demands, there is the need for a long-term cost effective means of managing the issues raised in horizontal drilling and hydraulic fracturing especially wellbore instability and environmental concerns. The focus of this study is the emulsifying ability of egg yolk in a low oil-water ratio invert emulsion. This is essential for enhanced wellbore stability, high penetration rate and lubricity required for horizontal drilling in unconventional shale.

2.4. Choice of vegetable oil for invert emulsion mud.

Drilling fluids have several properties to be controlled and maintained for it to function effectively in any drilling operation. The main fundamental properties are density, rheology and filtration. Drilling of unconventional shale reservoirs demand the use of drilling mud such as oil-based mud with sterling properties including excellent borehole control, thermal stability, high lubricity, penetration rates. The greater cleaning abilities with less viscosity and enhanced shale inhibition is critical in shale formations for multi-stage fracking. These properties may help to reduce operational costs; however, there are several environmental issues associated with its disposal and hazards to personnel. This has continued to pose challenging issues due to stringent environmental legislation over its disposal. It has become imperative for the drilling fluids industry to provide oil-based drilling fluids using alternative base oils to diesel and mineral oils that will be environmentally friendly while maintaining high technical competence. Over the years several plant oils such as rapeseed oil, Mahua oil, Cottonseed oil, Sesame oil, Soya bean oil, palm oil, Canola oil, Moringa seed oil, Soapnut and Jatropha have become popular as diesel substitute because of their low toxicity (Fadairo et al. 2012)

However, there have been conflicting issues with maintaining the mud technical properties such as good rheology, high stability, low fluid loss and compatibility of the chemical additives with the vegetable oil continuous phase. Hall (2005) noted that environmental and technical performances are very important characteristics of drilling fluid additives. However, in some cases the technical performance and environmental characteristics of material used to make such additives seem to be at odds with each other. For instance, it has been observed that materials that exhibit high stability in response to temperature-related effects are frequently poor biodegraders and those that are chemically active have shown high toxicities. Within the industry it has been acknowledged that toxic additives are the high performers and need to be replaced with environmentally friendly mud additives. The hazardous effect on marine and human life have been reported due to the usage of additives such as defoamers, descalers, thinners, viscosifiers, lubricants, stabilisers, surfactants and corrosion inhibitors. Ferro-Chrome lignosulfonate (a thinner and deflocculant) affected the survival and physiological responses of fish eggs and fry. Filtration control additive carboxymethyl cellulose (CMC) causes the death of fish fry at high concentrations (1000-2000mg/ml) as physiological changes starts at the level of 12-50 mg/ml. As was published by Greenpeace in 1995, about 896 tonnes of drilling mud containing soltex, a fluid loss additive was dumped along the coast of Great Britain thus contaminated the coast. This is because soltex contains potentially toxic heavy metals like antimony, arsenic, cadmium, nickel, lead, mercury, vanadium, zinc barium, fluoride and copper.

Apaleke et al.(2012a) noted that a low oil-water ratio is a major gap in the trend for developing oil-based mud using vegetable oil stating that the industry has not gone beyond OWR of 85/15. Apaleke (2012a) also emphasised the need for careful selection of additives for vegetable oil-based mud stating that additives that will function in base-oil from hydrocarbon or synthetic source may not function well in a vegetable grade oil-base medium. Amanullah, (2005), has already noted the issue of incompatibility of additives with vegetable oil-base medium in regards to duratone used as fluid-loss control additive in mineral oil based muds, which is not suitable for vegetable oil-based mud.

2.4.1 Choice of jatropha oil as base fluid

The consideration of vegetable oils as alternative to diesel oil in the formulation of oil-based mud is from a chemical point of view that diesel is toxic and non-biodegradable due to its aromatic content (Agwu et.al. 2015). Diesel comprises of about C_8-C_{21} aliphatic hydrocarbons with up to 25% aromatic compounds. (Chilcott, 2006). According to Amit and Amit (2012) vegetable oils are triglycerides of fatty acids. In this study, jatropha oil was used as a base fluid alternative to diesel oil in the formulation of an environmentally friendly invert emulsion. Jatropha oil, is a non-edible oil and has been identified as a potential base oil source. Amit K. J and Amit, S (2012) noted that India is the highest importer of edible oils in the world with approximately 16.6 million tonnes of edible oils consumed each year in India. This means that edible oils are already in high demand to meet the domestic requirements; therefore, inedible oils are preferred as a base oil in oil base mud. In comparison with other sources, jatropha oil in addition to high flash and pour points, has the added advantages of rapid seed growth, higher productivity and suitable for tropical and subtropical regions of the world.. Fadairo et al.,(2012a) compared toxicity level of diesel, canola and jatropha oil based muds by exposing planted bean seed to 100ml of each of the mud for one week. The bean seed exposed to Jatropha OBM survived for 18 days and the soil sampling indicated the presence of living organisms such as earthworm while the seed exposed to diesel OBM withered after 6 days and resulted in no trace of living organisms in the soil. This indicates that jatropha OBM has lower toxicity level. The economic consideration of Jatropha oil based mud is discussed in Chapter 4 of this work.

2.4.2 Drilling fluid additives

Drilling fluid properties are usually modified by additives to enhance the performance of the fluid. There is no formal definition for drilling fluid additive, but any substance or material both reactive and inert added to a drilling fluid to enhance its performance is known as drilling fluid additive. They are very essential constituent of drilling fluids to achieve a safe drilling operation ranging from weight control, viscosity, fluid loss, stability, pH control and others.

- **Weight control materials**: **(Densifiers)**

Weighting materials are finely divided solid materials with high specific gravity used to increase the density of drilling mud in order to control formation pressure. These include barite, hematite and calcium carbonate. In the present study, Barite was used as the weighting material. *Barite* is a naturally occurring barium sulfate ($BaSO_4$) which is the most common weighting material used in drilling operations. It has a specific gravity of 4.2 which enables it to increase the mud density to 21 ppg (2.4 SG).

- **Viscosity control materials (viscosifiers)**

Drilling fluids have transport abilities, suspension capabilities and gelatin properties. Viscosifiers are the products that enable drilling fluids to maintain these basic rheological properties. Clays and natural or synthetic polymers such as bentonite and attapulgite clays are the common materials used as viscosifies. For this study, *organophilic bentonite* which is oil dispersible was used as the viscosifier.

- **Fluid loss control**

Control of fluid loss helps to maintain wellbore stability. Filtrate-control additives are used to reduce the amount of water lost to the formation. Duratone, carboxy methylcellulose (CMC), polyanionic cellulose (PAC) and lignites are common fluid loss additives. Aston et al. (2002) noted that a low oil-water ratio is beneficial in providing low fluid loss. In the present study, the low oil-water ratio of 50/50 invert emulsion with a vegetable oil continuous phase was prepared without a fluid loss additive.

- **pH control**

These are additives designed to control the acidity or alkalinity of drilling fluids. The common pH control additives are lime (calcium oxide,) sodium carbonate, sodium hydroxide, sodium bicarbonate, sulphuric and hydraulic acids. Lime, sodium carbonate, sodium bicarbonate and sodium hydroxide increases the pH of drilling fluids, while sulphuric and hydrochloric acids reduce it.

- **Emulsifier**

Emulsifiers lower the interfacial tension between oil and water, which allows stable emulsions with small drops to be formed. They are usually fatty acids. The stability of an emulsion can be enhanced by the addition of surfactant or emulsifier to decrease the surface tension making the droplets more stable. Emulsifiers are molecules that have hydrophilic and hydrophobic ends. While the hydrophilic end forms chemical bonds with water, the hydrophobic end forms chemical bonds with oil. The need for compatible and friendly has been earlier mentioned in Section 2.4. In the present study, egg yolk was used for compatibility and non –toxic emulsifier in comparison with a conventional emulsifier, versa clean VB.

Versaclean VB contains fatty acids, tall –oil and reaction products with amines such as diethylenetriamine, and maleic anhydride. Though it has not been classified as environmentally hazardous, large and frequent spill can cause harmful effect on the environment. The inhalation of versaclean VB may cause irritation of respiratory tract while contact with the skin and eyes cause irritation (Mi-swaco, 2015).

- **Choice of egg yolk as non-toxic emulsifier**

In toxicity assessment of individual ingredients of synthetic based drilling muds, the emulsifier caused the strongest biomarker responses. The primary emulsifier (Emul S50) followed by the fluid loss agent (LSL 50) caused the strongest biochemical responses in fish (Bakhtyar and Gagnon 2012). In line with the replacement of toxic additives with non-toxic ones for environmental concerns and for compatibility with the vegetable oil continuous phase, hen egg yolk was used as non –toxic emulsifier in the present study. Egg yolk is the yellow internal part of the egg rich in protein and fat. It contains amino acids with a hydrophobic end, that is attracted to the oil molecules and a hydrophilic end that is attracted to the water molecule. Lecithin which is a common phospholipid found in egg yolk is an effective emulsifier because of its polar and non-polar properties. Egg yolk is a pseudo-plastic non-Newtonian fluid, with a viscosity, which depends on the shear forces applied. Its surface tension is 0.044Nm^{-1}(25 ° C), whilst its pH is 6.0 (Belitz et al., 2009). The percentage composition of egg yolk and white is shown in Table 2.2

Table 2.2: Percentage composition of egg yolk and white (indianchickens.com/consultancy. chemical composition of egg accessed on 15/06/2015)

Nutrients	egg yolk	egg white
Water	48.0	88.0
Protein	17.5	11.0
Fat	32.5	0.2
Minerals	2.0	0.8

The minerals that made up the two percentage mineral composition of the egg yolk as was highlighted in Table 2.2 is shown in Table 2.3

Table 2.3: Mineral composition of egg (Ahmed 1993)

Minerals	Component (ppm)	
	Egg White	**Egg yolk**
Phosphorus	1000	5900
Sulphur	1960	1570
Potassium	1600	1100
Sodium	1600	700
Chlorine	1500	1000
Calcium	100	1300
Magnesium	100	1300
Iron	1	86
Copper	0.85	4.15
Manganese	-	1.13

Egg yolk provides appreciated organoleptic and functional characteristics such as emulsifying, coagulating and gelling. It is mainly composed of two fractions: plasma and granules which are natural and micro-assemblies consisting of different composition, structures and functionalities and exhibit specific behaviour under treatments such as high pressure and temperature. The percentage composition of egg yolk on dry weight basis showing the fractions; plasma and granules is shown in Table 2.4.

Table 2.4: Percentage composition of egg yolk on dry weight basis (indianchickens.com/consultancy. chemical composition of egg accessed on 15/06/2015)

Nutrients	Granules	Plasma
Lipid	34	78-81
Protein	60	18
Ash	66	2

The plasma exhibits better emulsifying activity than the granules because it consists of mostly lipoproteins, which are soluble. Granules are not soluble but can stabilise the formed emulsion bridging the droplets. It was demonstrated that plasma is more sensitive to heat treatments (55–76 ˚ C) than granules, and can be subjected to greater treatment without lowering their emulsifying properties. (Anton, 2013). The coagulating property of egg yolk makes it a good binding agent (Belitz et al., 2009). This is important in its emulsifying function as a more stable emulsion could be achieved. Hen egg yolk is cheap and readily available. Most of the eggs used were collected from local shops where they have past their sell by date.

2.5 Wellbore instability in shale

Wellbore instability and borehole instability will be used interchangeably. Shale stability has greatly suffered from a lack of understanding of shale – drilling fluid interaction which may induce wellbore failure by changing the stress state and or the shale strength (Van Oort et al., 1996). Wellbore instability is often seen as sloughing and caving shale, resulting in hole enlargement, bridges and fill. The most common consequences are stuck pipe, sidetracks, with difficulties in logging and interpretation, sidewall recovery, running of casing, poor cement jobs and lost circulation. All these contribute to increased cost of drilling and the possibility of losing part of the hole or entire well or reduced production. Azar and Samuel (2007) defined borehole instability as an undesirable condition of an open hole interval that does not maintain its gauge size and shape and / or its structural integrity. Improper selection of either the weight or selection of the drilling fluid can cause wellbore instability, stuck pipe, low rate of penetration, difficulties in casing, logging and formation damage (Patil et.al., 2016).

According to Likrama and Diaz (2015) causes of wellbore instability in shale may be separated into mechanical and chemical categories stating that mechanical instability results from mechanical failure of weak rock formations owing to the stresses in the vicinity of the wellbore. Figure 2.7 represents mechanical pipe sticking due to borehole instability

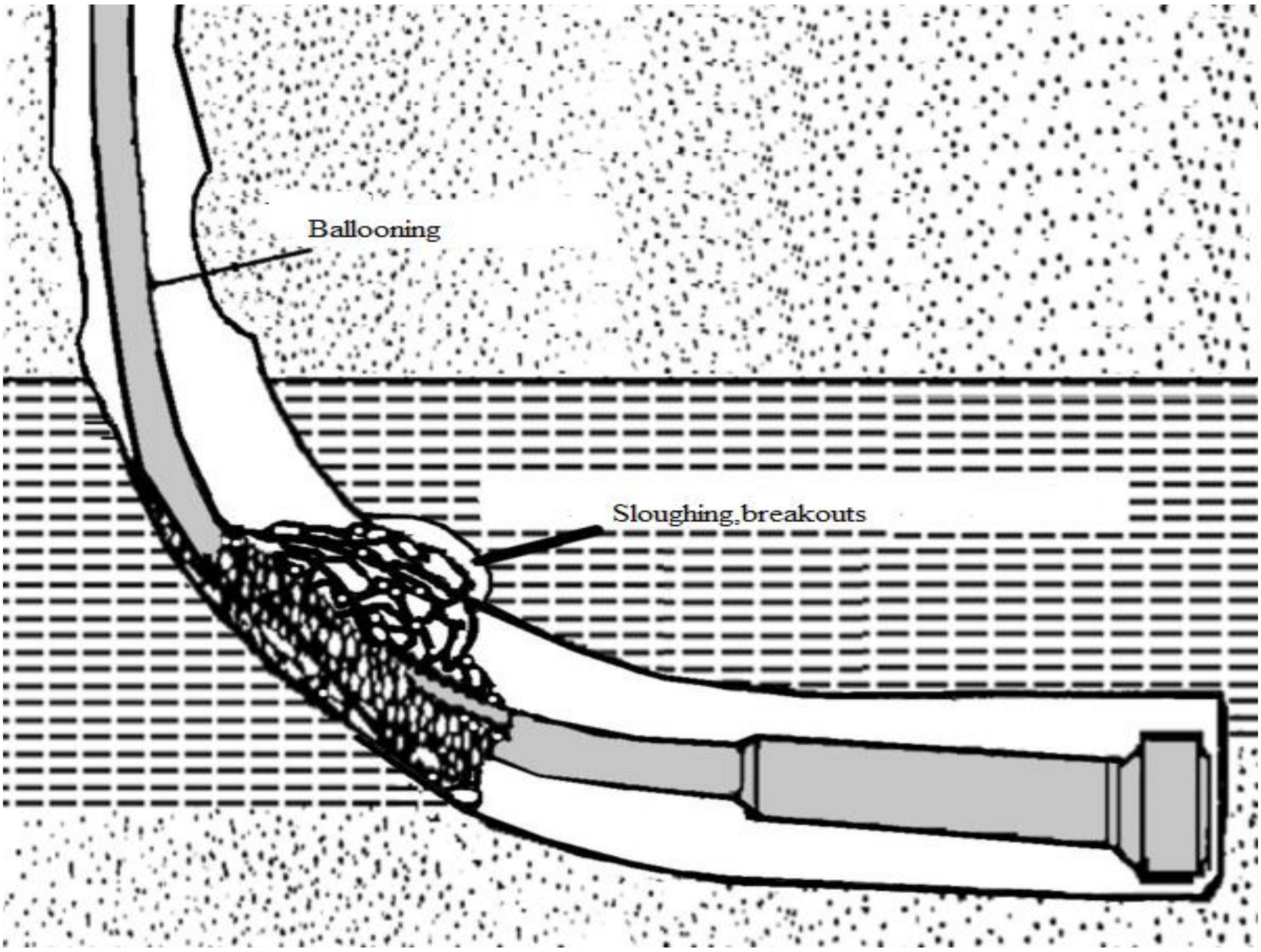

Figure 2.7: Mechanical Pipe sticking due to wellbore instability
(http://petrowiki.org/Mechanical_pipe_sticking accessed on 30/11/2016)

On the other hand, chemical instability results from the chemical reaction between water-based drilling fluids and clays present in the shale rocks, which in turn cause swelling, weakening and destabilisation of the wellbore. Physical interactions can also lead to wellbore instability. These include erosion, wetting along pre-existing fractures and fluid invasion resulting in pressure transmission. The amount of erosion that will occur depends on the strength of the rock. Erosion in shales and sandstones has been shown to be greater if the bit nozzle shear rate is greater than 100,000 sec^{-1}. It is important to prevent fluid invasion and consequent pressure transmission in shale. Emulsion drilling fluids are the ultimate membrane generating systems with the ability of preventing fluid invasion and reducing pressure transmission in shale. (Hughes, 2006)

The physical properties and behaviour of shale exposed to drilling fluid depend on the type and amount of clay in the shale. (Khodja et al., 2010). The traditional laboratory methods such as dispersion test, bulk hardness test and swelling test cannot fully reflect the impacts of rock structure on fracture development and rock failing. Immersion test is one of the most efficient methods of evaluating fluid-rock interaction and fracture development (Gomez and He 2012). In the presnt study, shale fluid interaction was investigated by immersion test using an X-ray computed tomography (CT) Scanner for imaging and analysis of the shale sample.

2.6 Shale characterisation using X-ray Computerised Tomography (CT) Scanner

Shale characterisation provides knowledge on the composition and properties of the shale. Characterisation also provides information on minerals and their relative abundance in the shale and shale reactivity. In this study, characterisation was carried out to reveal original fractures, enlargement of existing fracture or the development of a new fracture before and after the immersion test. The CT Scan is a non-destructive technique for visualising interior features within solid objects, and for obtaining digital information on their 3-D geometries and properties. It is useful for petrophysical , fluid and gas flow analysis with few restrictions on the type of sample or design of experiment (Jacobs, et al., 1995) A CT image is typically called a *slice*, as it corresponds to what the object being scanned would look like if it were sliced open along a plane. A CT slice corresponds to a certain thickness of the object being scanned, while a typical digital image is composed of pixels (picture elements).

The images captured on the CT scan were analysed using Volume Graphics (VG) Studio Max 2.2 software. VG Studio Max is a software package for the visualisation and analysis of voxel data. It is used in a variety of application areas such as industrial CT, medical research, life sciences, animation and many others. VG Studio Max 2.2 has many functions including volume analysis, wall thickness analysis, porosity/inclusion analysis, coordinate measurement and CT reconstruction. The present study investigated the volume analysis.

2.6.1 Choice of shale sample

The selection of a test sample depends on the availability and purpose of the tests. It is expected that under ideal conditions, correctly preserved shale core samples would give accurate results though such samples may not be readily available due to high cost of acquiring them. Shale cuttings taken from drilled formations can also be used for some tests. (Friedheim et al 2011). The core samples used in this study are from the Marcellus shale of the Middle Devonian-age, found in Pennsylvania, New York, Ohio and West of Virginia. It is a black, low density carbonaceous (organic) rich shale. The Marcellus formation extends more than 34,000,000 acres of real estate with at least 50 feet formation thickness which could contain 500 Tcf gas in place with estimates of recoverable gas at 50Tcf. (Belvalkar and Oyewole 2010). Like any other shale, the Marcellus has low permeability. Agbaji et al. (2009) pointed out that Marcellus has favourable mineralogy, because it is a lower- density rock with more porosity, which has the potential of been filled with more free gas. Agbaji, further noted stated that Marcellus formation is variable in depth, while some outcrops, appears at the surface in some areas of New York, majority is more than a mile deep and in some areas extends 9,000 feet below the surface.

2.7 Management and disposal of used mud and drilled cuttings

Throughout the drilling process, drilling mud is recirculated, which helps to decrease waste by reusing as much mud as possible however when the drilling process is completed, the drilling waste must be disposed of in one way or the other. Method of disposal of used mud and drilled cuttings vary depending on the choice of the operator. The choice largely depends on the type of generated cuttings and cost of treatment and disposal. Disposal method could be onsite, offsite, using farmlands, landfills, and thermal technologies. Salt water muds and oily cuttings are not suitable for onsite management. In some cases, environmental sensitivity precludes onsite waste management. Cost effectiveness is also another reason for commercial waste management facilities because rather than constructing, operating and closing an onsite facility for a relatively small volume of waste (Puder and Veil 2007). The management and disposal cost of the with vegetable oil invert emulsion mud will be evaluated, and compared with that of diesel oil based mud.

2.8 Chapter summary

- The overview of drilling fluid, properties and additives, drilling and fracturing in unconventional shale reservoirs were presented.
- Development of drilling fluid using vegetable oils, the progress so far and the challenges including achieving a low oil-water ratio and replacement of toxic additives with non-toxic supplements has been discussed.
- Shale –fluid interaction by X-ray computerised tomography scanner was highlighted.
- Choice of disposal methods of used mud and drilled cuttings were mentioned. The next Chapter presents the experimental work carried out in this study.

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Chapter 3

Apparatus, Materials and Procedures

3.1 Introduction

This Chapter discusses the detailed experimental apparatus and procedures used in carrying out this research work. As shown in Figure 3.1, the procedure is divided into two distinct phases*: phase –I*, mud preparation and testing, and *phase-II* topology (*shale-fluid interaction*)

- **Phase-1:** The equipment and mode of operations used for mud formulation and testing of the various mud properties are described in Section 3.2.1. Preparation of experimental materials involved the testing of the properties of experimental materials, *diesel oil, jatropha oil and bentonite* as described in Section3.2.2.2, as well as the separation of the egg yolk as detailed in Section 3.2.2.3. The terms mud preparation and mud formulation will be used interchangeably throughout the present investigation.
- **Phase-II:** Shale-fluid by immersion of Marcellus shale samples into water based, diesel and jatropha oil based muds for one to seven days. The X-ray (CT) scanner equipped with image acquisition and processing software, *volume graphics studio max 2.2 software* was used for the scanning and processing of the core samples. The description and mode of operation of the scanner are detailed in 3.3.1.1 and the preparation of the materials and *shale core sample* are detailed in Section 3.3.2 The procedure employed in conducting the experiment is described in Section 3.3.3

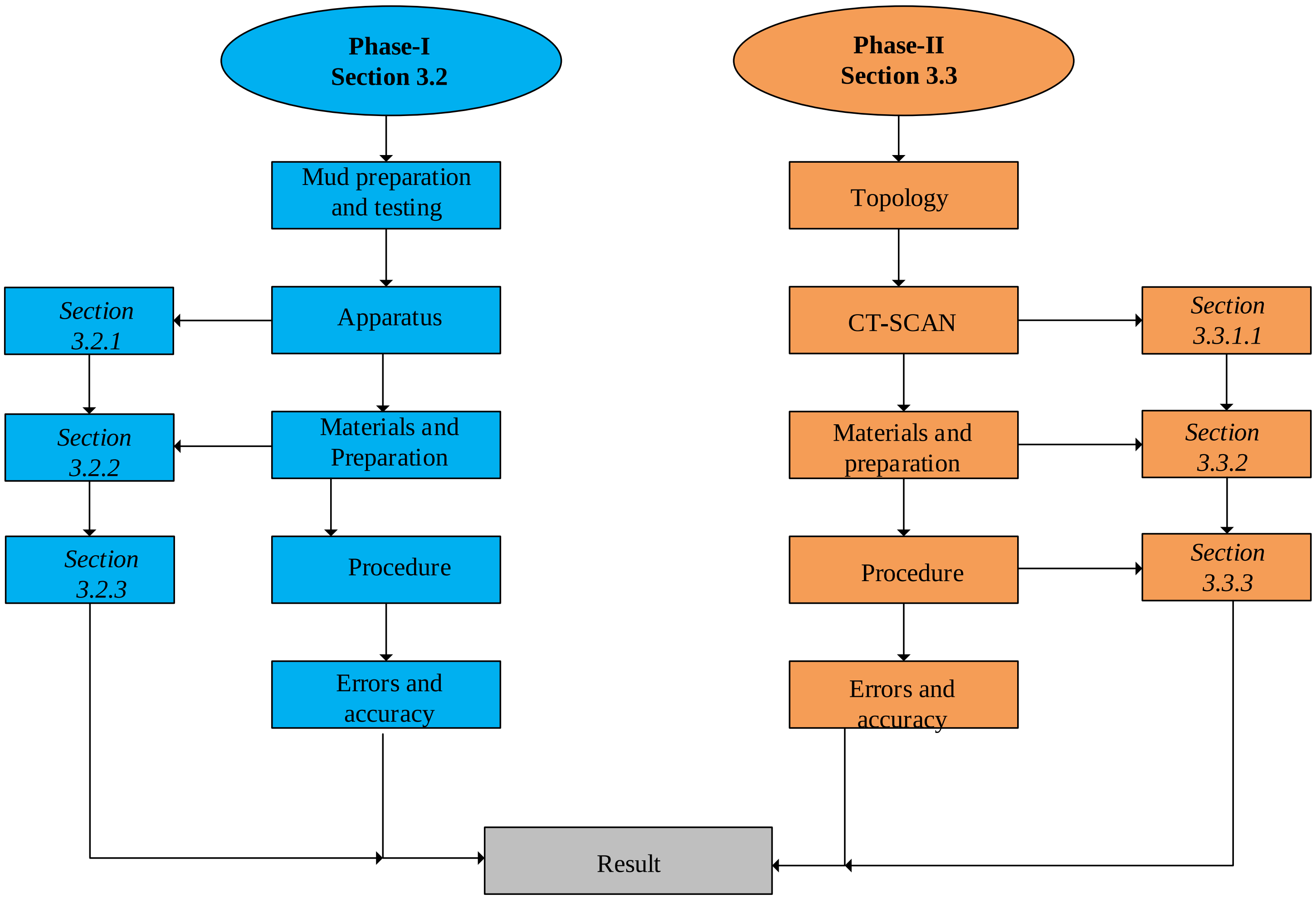

Figure 3.1: Structure and sequence of experimental investigation

3.2 Phase -I: Mud formulation

3.2.1 Apparatus

3.2.1.1 Mud balance

The mud balance is the instrument generally used for the determination of the density of drilling mud. The density of drilling fluid must be controlled to provide adequate hydrostatic head to prevent influx of formation fluids but not so high as to cause loss of circulation or adversely affect the drilling rate and damage the formation. For this study, the mud balance used is the Ofite model 115-00 shown in Figure 3.2.

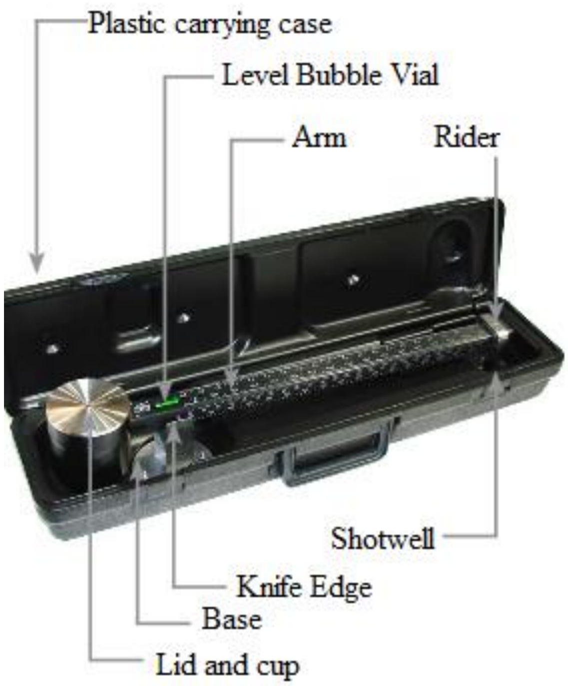

Figure 3.2: Mud Balance (http://www.ofite.com/instructions/115-00.pdf accessed on 19/06/2014)

The mud balance consists of a constant volume cup with lever arm and rider calibrated to directly read the density of drilling mud in pounds per gallon (ppg), pounds per cubic feet (lb/ft^{3}) and pressure gradient in psi/1000ft. The arm is graduated and allows accurate measurements within ±0.1 pounds per gallon or ±0.01 specific gravity. The balance is designed such that the drilling mud holding cup at one end of the beam is balanced by a fixed counterweight at the other end with a sliding-weight rider free to move along a graduated scale.

A bubble-level is mounted on the beam to allow for accurate balancing though attachments for extending the range of the balance may be used when necessary.

The balance was initially calibrated with fresh water to read 8.33 lb/gal or 62.4lb/ft^3,1.00g/ml or 1000kg/m^3 at 21 ˚ C. The calibration was carried out in the following order:

i. The lid was removed from the cup and the cup was completely filled with water.
ii. The lid was replaced and wiped dry.
iii. The balance arm was replaced on the base with knife-edge resting on the fulcrum.
iv. The level vial was centred when the rider was set on 8.33. If it does not centre , then adjust the balancing screw or amount of the lead shot in the well at the end of the graduated arm as required.

3.2.1.2 8-Speed viscometer

The viscometer is the instrument used both in the field and laboratory for the measurement of the rheological properties of fluids. In drilling fluids, the rheological properties measured are plastic viscosity, apparent viscosity, yield point and gel strength. The Ofite 8-Speed model 800 electronic viscometer shown in Figure 3.3 was used for these rheological measurements. The viscometer determines the flow characteristics of drilling fluids in terms of shear rate and shear stress over various times and temperature ranges at atmospheric pressure. The eight precisely regulated test speeds (shear rates in RPM) are 3(gel), 6, 30, 60, 100, 200, 300, and 600. The speed is easily changed with a control knob and shear stress values are displayed on a lighted magnified dial for easy reading. The speed accuracy is 0.1rpm

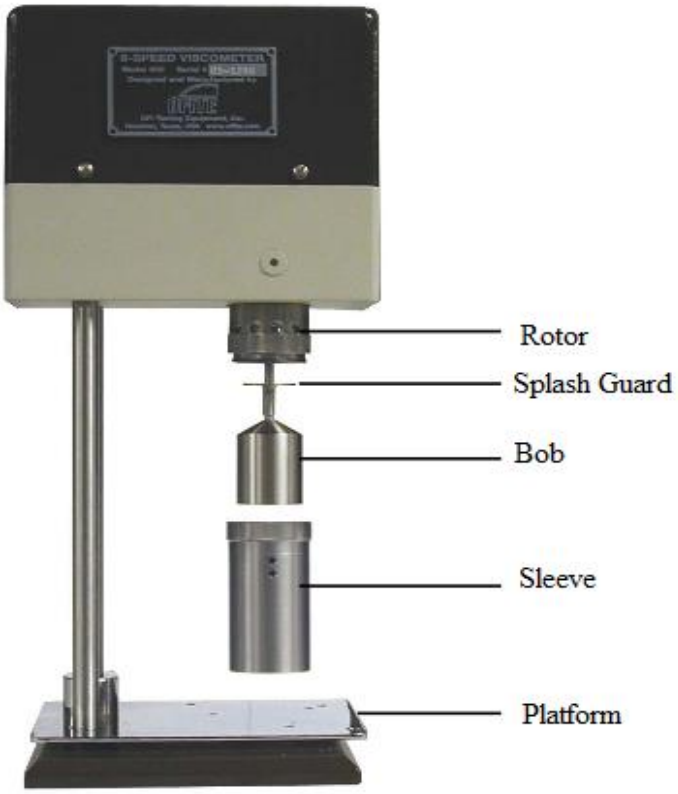

Figure 3.3: 8 -Speed Viscometer ((http://www.ofite.com/instructions/130-10.pdf. accessed on 19/06/2014)

The viscometer was calibrated using the calibration instrument and the calibration check kit supplied together with the instrument. The calibration was done in the following order:

i. The certified calibration fluid was chosen using the temperature-viscosity chart supplied with the calibration fluid to cover the viscosity range of interest ensuring that the lot number on the chart matches with the lot number on the fluid container.
ii. The viscometer bob, sleeve and cup were cleaned and dried. The viscometer and the calibration fluid were placed side-by-side on the counter top in a room with a constant temperature variation of less than 5 ˚F ± 2.5 ˚ F (-15 ˚C ± -16.4 ˚C) allowing the viscometer and the fluid to stand for at least two hours to equilibrate.
iii. The viscometer was operated in air for two to four minutes to loosen up the bearings and gears. The rotor sleeve was observed for excessive wobbling and to be replaced if necessary.
iv. The cup was filled to the scribed line with the calibration fluid and placed on the viscometer stage moving the stage upward until the fluid level was to the fill line on the sleeve. The sleeve was not allowed to be immersed in fluid above the fill line.

v. The thermometer capable of ±0.1 ˚ C was placed into the fluid and held or tape it in place to prevent breakage. The viscometer was operated at low speed setting until the thermometer reading becomes stable within ±0.1 ˚ C per 30seconds taking of the temperature reading.

vi. At a stabilised temperature, the viscometer was operated at 600 RPM and then at 300 the dial readings were recorded to the nearest 0.5 dial unit.

vii. With the temperature-viscosity chart, the certified viscosity is determined to the nearest 0.5 centipoise. The 300 RPM reading was compared to the standard viscosity and recorded and any deviation was noted. The 600 RPM reading was divided by the calibration standard of 1.98, and compared it to the standard viscosity and recorded and deviation was noted.

viii. Deviations exceeding 1.5 dial units were not acceptable. If the deviation exceeds this tolerance, the viscometer will require adjustments.

3.2.1.3 High Temperature-High Pressure Filter Press.

The High Temperature High Pressure Filter press is designed for the evaluation of the filtration characteristics of drilling fluids, cement slurries, fracturing fluids and completion fluids under elevated temperatures and pressures. The evaluation of fluids under HTHP conditions similar to downhole environment is of paramount importance as filtration behaviour and wall cake building characteristics of permeable formations vary with environment. The Ofite model 170-00, 175 ml single-capped test cell was used in this study.

The complete assembly of the HTHP Filter Press as shown in Figure 3.4 consists of the following: controlled pressure source (CO_2), high pressure test cell, heating jacket and a suitable stand

- Controlled pressure source carbon dioxide (CO_2)

The HTHP filter press is pressurised with small carbon dioxide bulbs which contain 10cm^3 of the CO_2 and weighs 8g. The bulbs are pressurised to approximately 1000psi and contain plenty of carrier gas to run a complete 30-minute filtration test, if running the standard API 500psi differential test, which is usually 600psi on the top manifold and 100psi on the bottom or back pressure

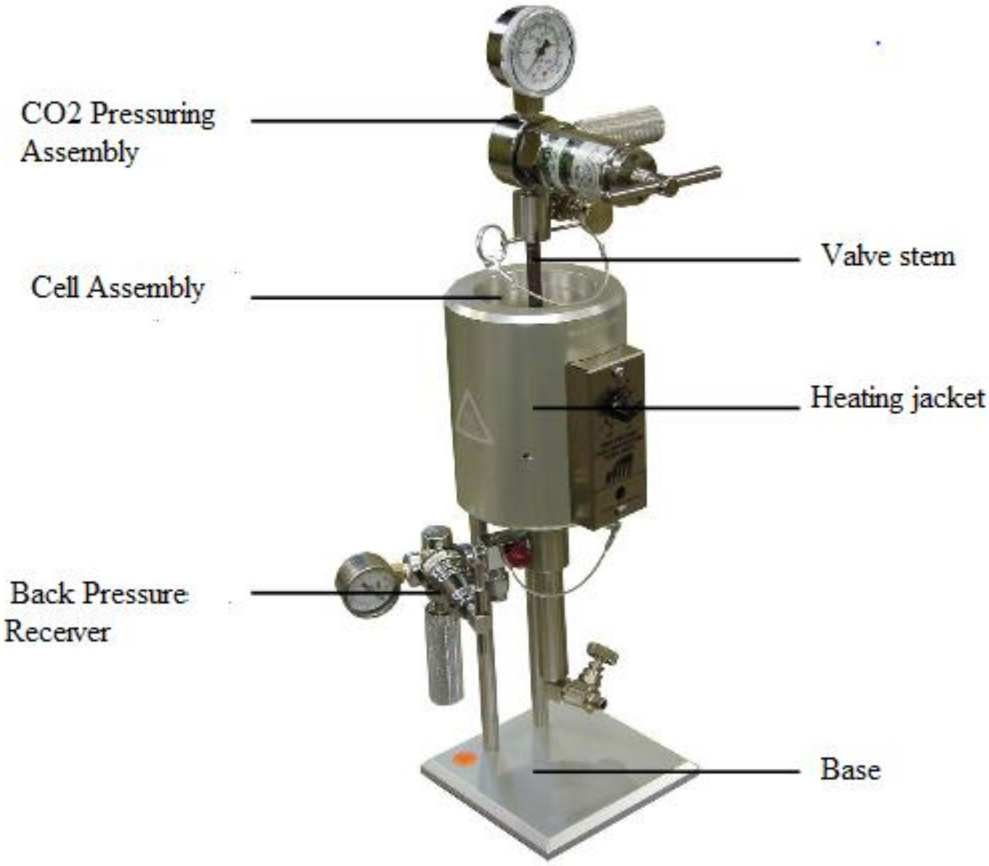

Figure 3.4: HTHP Filter Press (http://www.ofite.com/publications/instructions/170-170-00-instructions/file accessed on 20/06/2014)

- High pressure test cell

The mud sample is held in the test cell. The HTHP pressure test cell or assembly can handle standard filtration tests at elevated temperatures and pressures. The maximum pressure rating is 2000 psi and the maximum temperature is 400˚F (204˚C)

- Heating jacket

The heating jacket is responsible for heating and maintaining the temperature of the test cell throughout the test. The actual temperature of the heating jacket is measured with a stem thermometer and the thermometer is placed into the cell body.

- Suitable stand

The filter press is equipped with a suitable stand or base. This offers the HTHP press stability on the work surface.

3.2.1.4 Electrical stability tester (EST)

The electrical stability (ES) of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. The electrical stability is measured using an electrical stability tester. The Fann Model 23E EST shown in Figure 3.5 was used for the electrical stability measurement. The components of the EST include the testing unit, an electrode probe, two calibration standards, a power cord, batteries and a plastic carrying case.

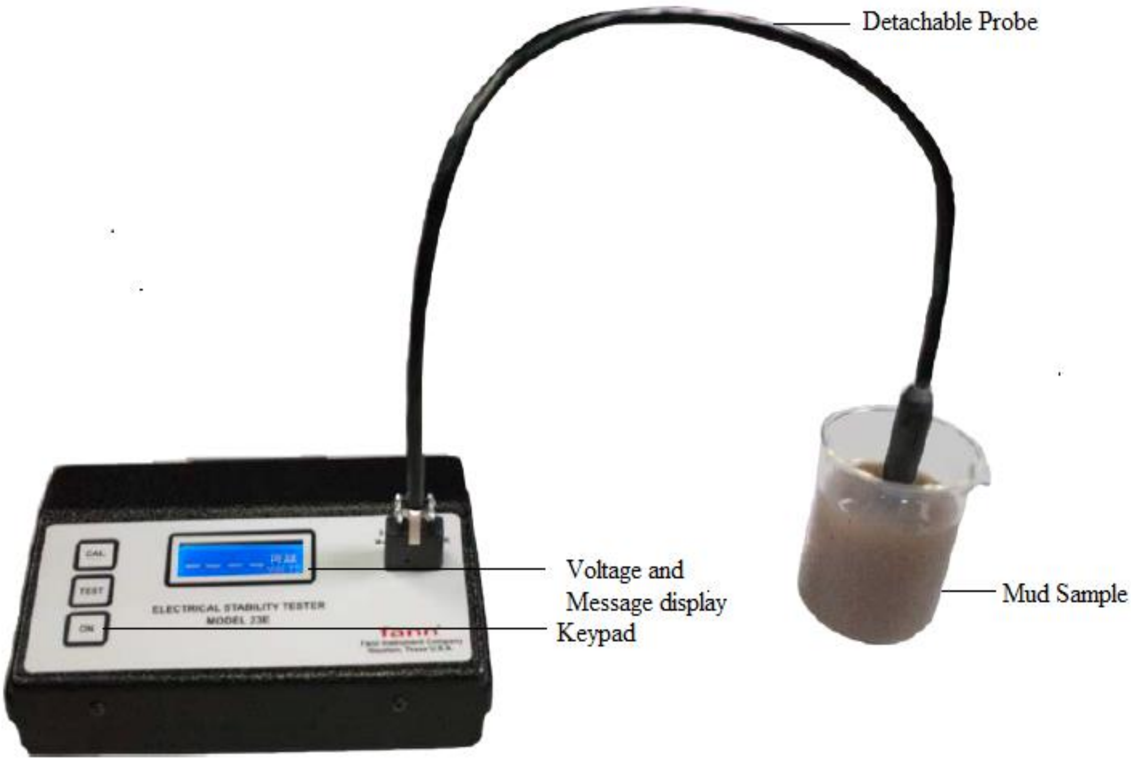

Figure 3.5: Fann Model 23E Electrical Stability Tester Probe immersed in mud sample (http://www.fann.com/public1/pubsdata/Manuals/Electrical%20Stability%20Tester%2023E.pdf accessed on 20/06/2014)

Prior to the test, the EST was pre-checked using the calibration standards, in this case the 610 and 1952 ±2% standards in the following order:

- One of the calibration standards, 610 was inserted into the probe socket (in place of the probe)

- The test button was pushed and released to start the automatic voltage ramp. The ramp stopped ramping when the voltage was within 2% of the standard calibration voltage. This was repeated using the other calibration standard of 1952 V.
- If the instrument does not read the peak voltage of the calibration standards within 2% of the standard calibration voltage, it means that either the calibration standards or equipment is malfunctioning and requires assistance from manufacturer.

- **The Probe check**
  i. Inspect the electrode probe and cable for evidence of damage
  ii. Ensure that the entire electrode gap is free of deposits and that the connector to the instrument is clean and dry
  iii. Connect the probe to the electrical stability meter and run the voltage ramp test in air. The peak voltage should ramp to >2000 volts. If not the electrode probe and connector may need to be cleaned or replaced.
  iv. Repeat the voltage ramp test with the electrode probe in tap water. The ES reading should not exceed 3V. If the ES exceeds 3V, either clean or replace the electrode.

3.2.1.5 Hamilton Beach mixer and cup

Drilling fluid formulations involve series of mixing or shearing at different times to achieve a homogeneous mixture. The Hamilton Beach mixer single-spindle model 936 model as shown in Figure 3.6 was used for all the mixing and shearing in this experimental study. The mixer is equipped with a mixing cup which contains the materials to be mixed. The 3-speed switch which is low, medium and high is used to control the mixing speed. The low speed is equivalent to 10,000 rpm, while the medium and the high speed are 14,000 and 17,000 rpm respectively

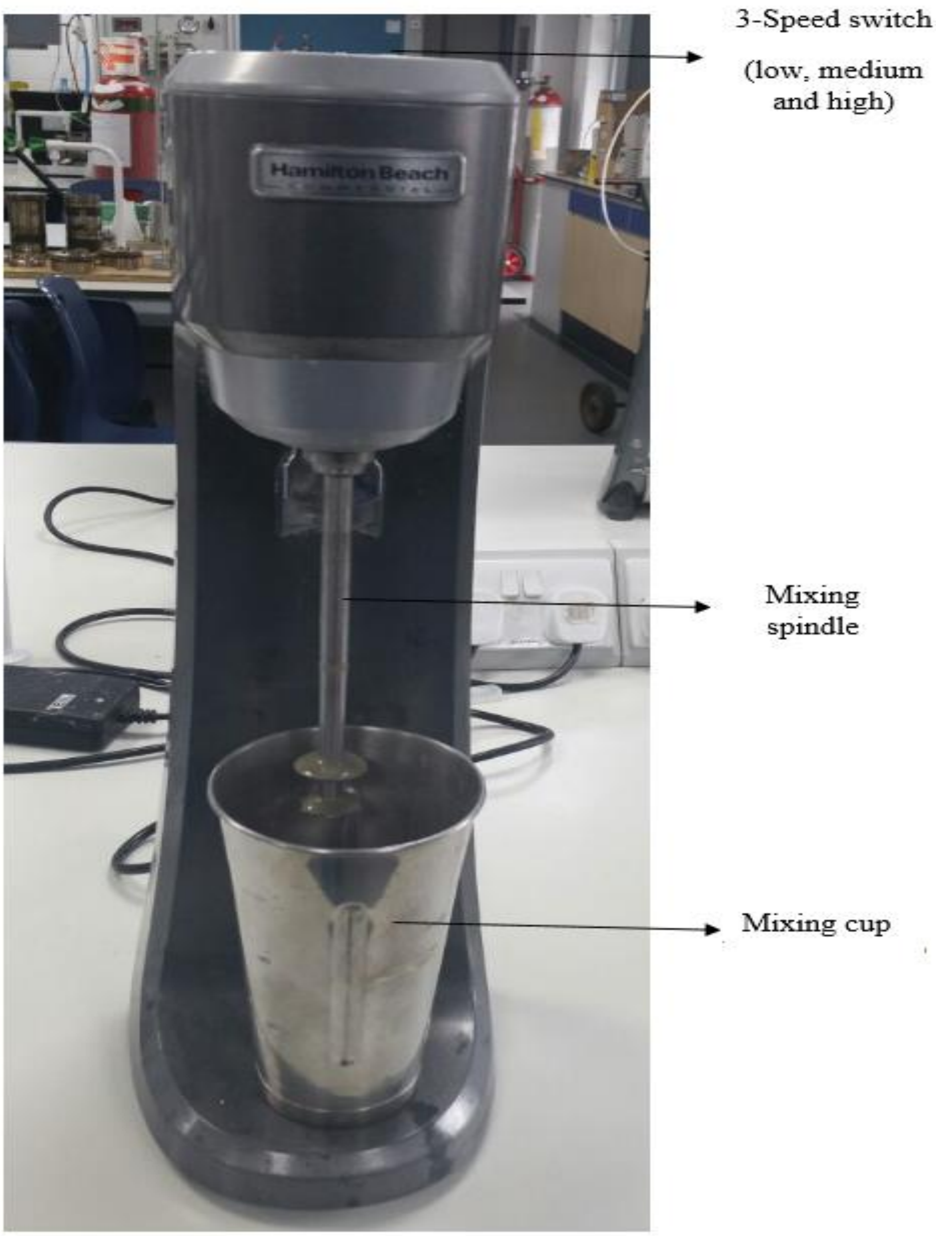

Figure 3.6: Hamilton Beach Mixer and cup

3.2.1.6 Marsh funnel viscometer

There is need to remove any foreign matter or deposits in the drilling mud that may alter experimental measurement. Fann model 200, 1500ml marsh funnel viscometer equipped with measuring cup model 202 shown in Figure 3.7 was used for the screening of the drilling mud prior to any measurement. The Marsh funnel is 6 in. (152.4 mm) in diameter at the top and 12 in. (304.8 mm) long, at the bottom, a smooth-bore tube 2 in. (50.8 mm) long having an inside diameter of 3/16 in. is attached in such a way that there is no constriction at the joint. A wire screen with 1/ 16-in. openings, covering one-half of the funnel, is fixed at a level of 3/4 in. (19 mm) below the top of the funnel. This funnel can also be used to make quick field checks of the viscosity of fluids by measuring the time it takes to fill the quart line (946ml) of the cup.

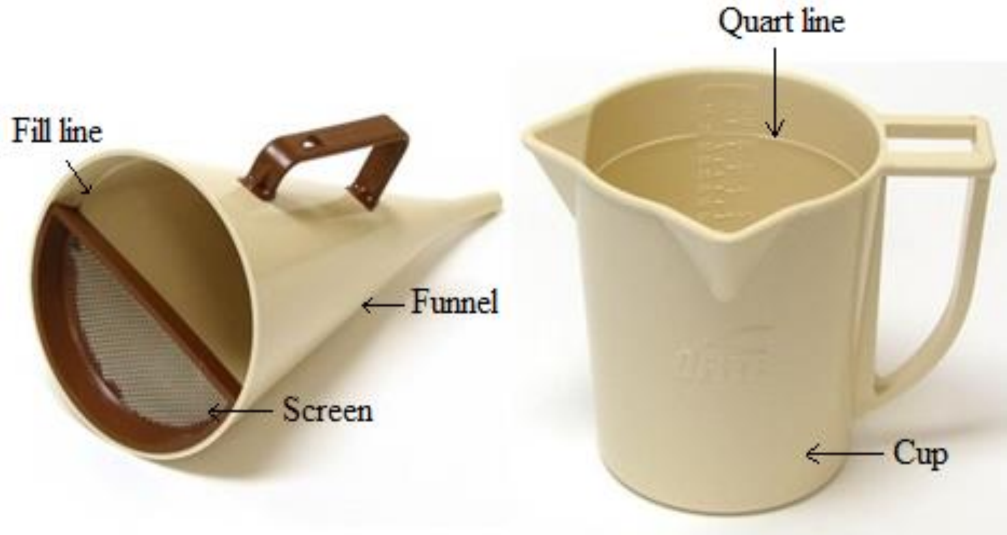

Figure 3.7: Fann Model 200 Marsh Funnel viscometer and measuring cup model 202

3.2.1.7 Analytical weighing balance

Experimental materials were weighed using the Kern –Als analytical weighing scale model, ALS 250-4A. The measuring pan of the balance is housed inside a transparent enclosure to prevent the collection of dust or any other materials that may contaminate the sample. The uniquely designed acrylic air foils allow for a smooth turbulence-free airflow that prevents the balance from fluctuation. These special features ensure accurate weighing results of ± 0.1g

3.2.1.8 pH meter and strips

Potential of hydrogen (pH) as was mentioned in Section 2.2.2 is a measure of the acidity or alkalinity of a solution. The pH of the mud was measured using pH meter and Strips. The pH meter is an electronic device utilising glass electrodes to measure a potential difference and indicate directly by dial reading of the pH of a sample. In this experiment, the pH was first calibrated with pH 4 and pH 7 buffer solutions before testing the pH of distilled water and the mud samples.

The pH paper strips have dyes absorbed to display certain colours in certain pH range from 0 to 14 with 7 as neutral. Readings less than 7 indicate acid solution, whilst readings higher than 7 indicate alkaline or base solutions. The pH test strip is dipped into the mud sample and allowed to stand for 15 seconds and the colour developed is compared with the colour chart on the test strip package. The pH is read as the closest colour match.

3.2.1.9 Digital thermometer and stop watch

Temperature measurements were carried out using a digital thermometer with a stainless steel sensor probe. The simple liquid crystal display (LCD) makes it easy to read and measures in Fahrenheit or Celsius. The temperature range is -50 ˚ C to 300 ˚ C or -58 ˚ F to 572˚ F**.**

The experimental analysis involved a series of tests for which accurate timing was essential. A Sper scientific 810012 digital water – resistant stop watch was used for all the timings. The stop-watch has 0.01 second precision with lap counter for the first 30minutes with alarm and hourly chime.

3.2.2 Materials and Preparation

3.2.2.1 Materials and functions

Mud has been introduced in the previous chapter as a mixture of fluids and solids which enables it to function well in drilling operations. The base fluid and solid components including the additives used in mud formulation have also been discussed in section 2.4. Table 3.1 highlights the basic materials and their functions that were used in this experimental investigation. The weighting agent, barite is a naturally occurring barium sulfate ($BaSO_4$) with a specific gravity of 4.2. The colour of barite varies from white to light shades of grey, red and brown depending on trace elements present. The organophilic clay bentonite is a white free flowing powder which is oil dispersible. Figure 3.8 shows a sample of diesel and jatropha oils whilst Figure 3.9 show a sample of the barite and organophilic bentonite used in this investigation.

Table 3.1: Experimental materials and functions

Materials	**Functions**
Diesel oil	Base fluid in oil-based mud and remains the continuous phase of the mud.
Jatropha oil	Vegetable oil base fluid used as an alternative to diesel oil for environmental concerns.
Water	Dispersed phase in the invert emulsion and continuous phase in the water based mud
Barite	Weighting agent used to increase the density of the drilling mud
Bentonite	Viscosifier used to maintain the viscosity of the drilling mud
VersacleanVB,	Primary emulsifier used to allow oil and water used to achieve a homogeneous mixture
Egg yolk	Used as a non-toxic primary emulsifier in line with environmental compatibility.
Calcium chloride	This is used as brine in the water phase, which is important for balancing the salinity of mud and shale formation, thereby preventing water loss into the shale layers.
Calcium oxide / Lime	This is used as a source of calcium and to increase alkalinity. Increased alkalinity stabilises clay suspension, improves the solubility of various additives and reduces corrosion of drill pipe and casing.

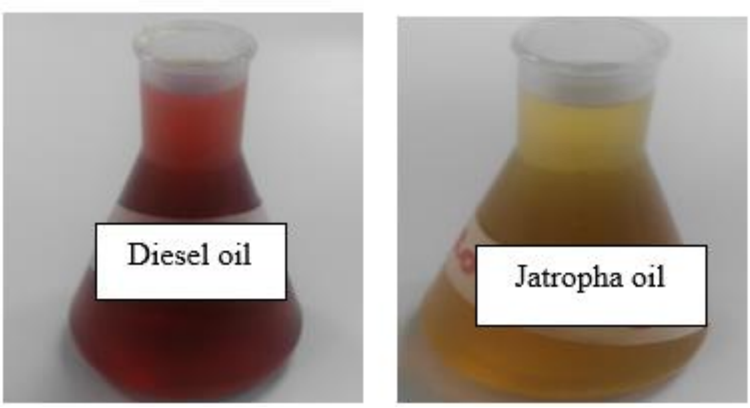

Figure 3.8: Sample of Diesel and Jatropha oils

Figure 3.9: Sample of Barite and Bentonite

3.2.2.2 Testing of experimental materials

Testing of experimental materials is a very important step in mud formulation. It is essential to ascertain the suitability of the materials when satisfying the requirements for mud formulation. Once the mud formulation is determined, testing of the materials to be used is the second step as shown in the mud formulation flow chart in Figure 3.11. In this investigation, the rheological properties and densities of diesel and jatropha oils were analysed. The density was measured using the mud balance described in Section 3.2.1.1, whilst the rheological properties were measured with the 8-speed viscometer described in Section 3.2.1.2 This was followed by testing of the basic requirement of the filtrate volume and yield point / plastic viscosity ratio of bentonite according to API 13A (2010**)** Specification for Drilling Fluids-Specifications and Testing. The rheological property was measured with 8-speed viscometer while the filtration property was measured with the high temperature-high pressure filter press described in Section 3.2.1.3 The results are presented in Section 4.2.1.

3.2.2.3 Preparation of the egg yolk

Two crates of British large size eggs containing ten eggs each were purchased from a local store. Each egg weighed 63g. The eggs were stored at room temperature of 25˚C. The separation of the egg yolk from the albumen (egg white) was carried out individually. The egg, to be separated was held in the upright position, with a receiving plastic container placed underneath. At this point, the shell was careful broken across the centre, allowing the albumen to drain into the receiving container. To remove adhering fragments of the albumen, the egg yolk was rolled on a filter paper and allowed to flow into a plastic container. The volume of each separated egg yolk was 15ml, this was determined using a plastic syringe. Figure 3.10 shows a stack of carefully separated egg yolk and the standard emulsifier versaclean VB

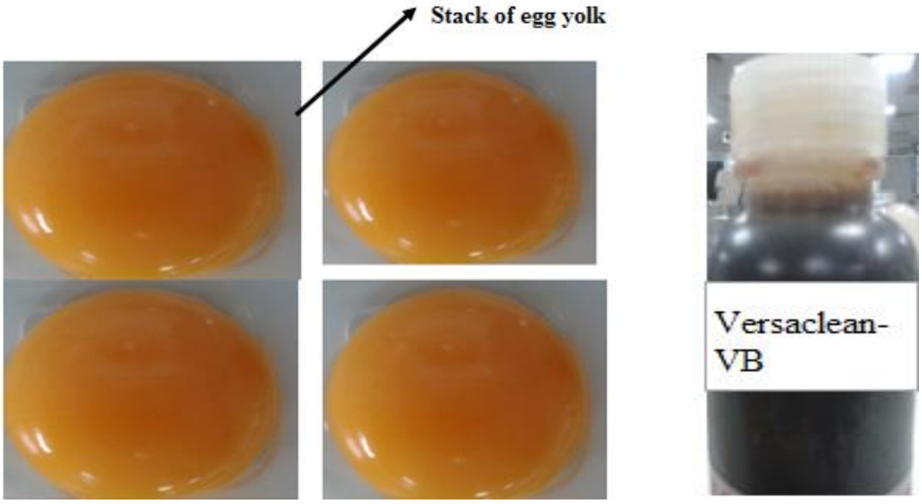

Figure 3.10: (a) Stack of Separated egg yolk (b) VersacleanVB

3.2.3 Procedure

The formulation of mud was conducted in the following sequence:

1. Determination of mud formulation: This was to ascertain the formulation to be conducted.
2. Testing of experimental materials as previously discussed in Section 3.2.2.2 is to ascertain the suitability of the materials for the experiment once the formulation is determined.

3. Preliminary mud formulation and tests in this investigation involved all the mud formulation and tests prior to the formulation of the determined mud formulation
4. Satisfactory result of the preliminary tests is followed by the mud formulation.
5. Mud formulation in this investigation involved the emulsifying ability of egg yolk and 50/50 OWR mud.
6. Mud formulation using egg yolk as an emulsifier to demonstrate its suitability as substitute to a standard emulsifier in this case versacleanVB.
7. Mud formulation of 50/50 OWR to demonstrate the feasibility of a low OWR mud with vegetable oil continuous phase.
8. This is followed by the testing of the properties, analysis and discussion of results and the economic consideration.

The sequence of this experimental investigation is summarised in the flow chart shown in Figure 3.11. The experiment was conducted in accordance with the *API Recommended Practice 13b-2 for Field Testing of Oil-based Drilling Fluids, Fourth Edition.*

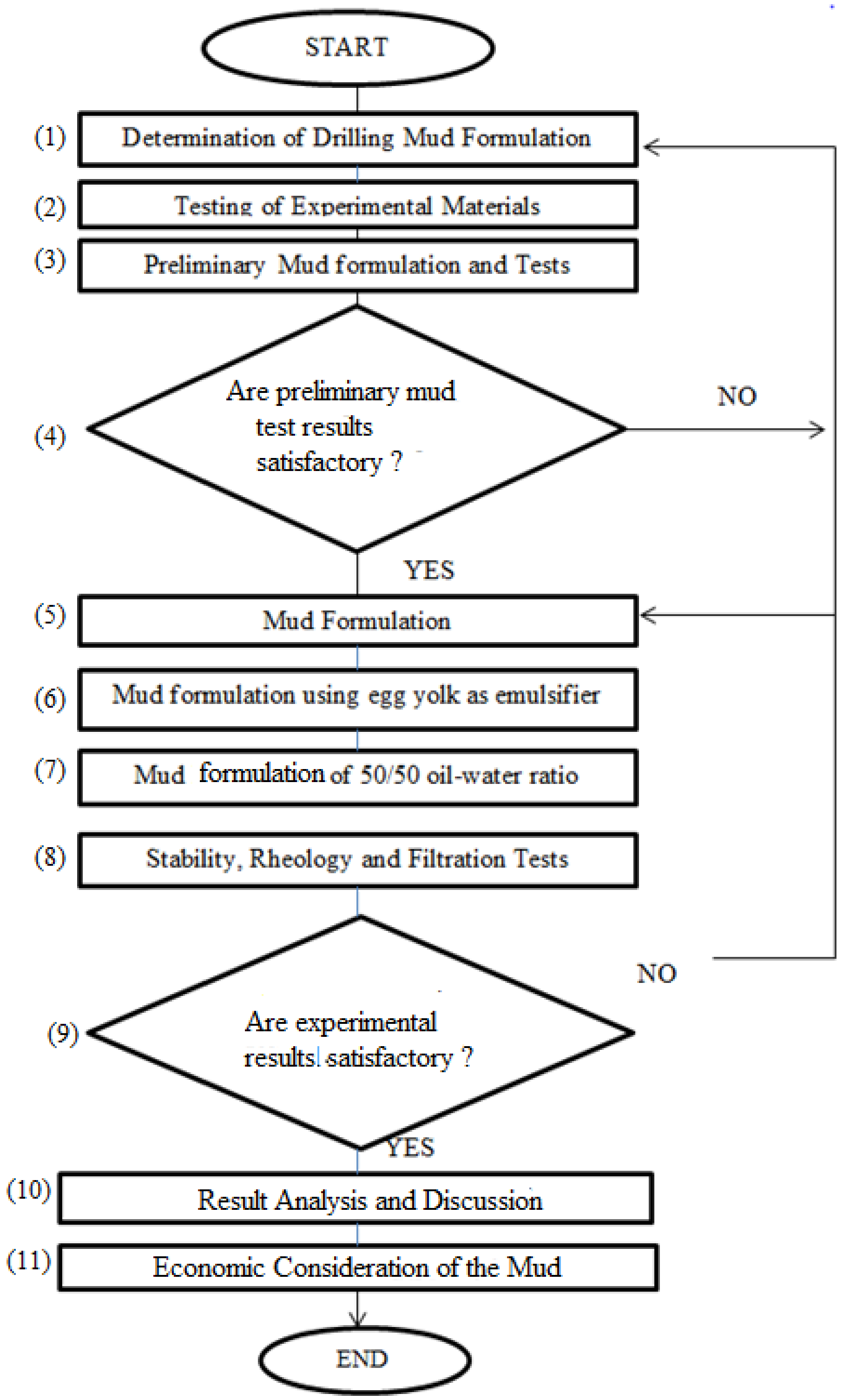

Figure 3.11: Mud formulation flow chart

3.2.3.1 Preliminary mud formulation and tests

The mud formulation determined for this investigation is the utilisation of egg yolk as a non-toxic emulsifier and a low oil-water ratio mud of 50/50 to bridge the gap in the trend of vegetable oil-based mud. However, preliminary mud formulations were conducted before the determined mud was produced. This involved the formulation of one laboratory barrel (350ml) each of four muds of OWR of 90/10, 80/20,70/30 and 60/40. These OWR were adopted for this preliminary test because an invert emulsion can be formulated from 5 - 50% of water as discussed in Chapter 2. Table 3.2 shows the composition of the preliminary mud formulation. The volume of egg yolk used was varied at 1,3,5 and 8ml where stability was observed. Lime was also added according to the pH of the mud

Table 3.2: Preliminary mud composition

OWR	90/10	80/20	70/30	60/40
Component	**Quantity**			
Base oil (Jatropha) (ml)	315	280	245	210
Water (ml)	35	70	105	140
Bentonite (g)	10	12	22.5	22.5
Egg yolk(ml)	1,3,5,8	1,3,5,8	1,3,5,8	1,3,5,8
Calcium chloride (g)	4	4	4	4
Lime (calcium oxide)(g)	8	10	12	3

The formulation was conducted in the following order:

90/10 OWR was formulated using 315 ml of jatropha oil and 35ml of water to make up the 350 ml laboratory barrel. Using a beaker, 315 ml of Jatropha oil and 35ml of water were measured and poured into a mud mixing cup. A transparent glass syringe was used to introduce 1ml of egg yolk into the mixing cup to emulsify the water and the continuous phase oil. This was then mixed for five minutes using the Hamilton beach mixer (Figure 3.6 in Section 3.2.1.5). The weighing balance was used to measure 10g of bentonite, 4g of calcium chloride and 8g of calcium oxide, these materials were introduced individually into the mixing cup and fixed to mixer for thorough mixing for 30 minutes. At the end of the mixing, the mud was poured into a plastic beaker in preparation for the electrical stability test. This was repeated as per Table 3.2 for 80/20, 70/30 and 60/40,

- Electrical stability test.

The electrical stability of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. The stability test was conducted to ascertain the emulsifying ability of egg yolk prior to using it on the final experimental 50/50 mud. This is because stability is a very vital property of invert emulsion

The electrical stability test was carried out using the Fann electrical stability tester (Figure 3.5 in Section 3.2.1.4). The test was conducted at the recommended temperature of 48.9 ˚C The electrode was thoroughly cleaned, wiped dry and then swirled in the base oil used for the mud formulation and then cleaned and dried. The mud sample was placed in a glass beaker and maintained at a temperature of 48.9 ˚ C. The sample was hand-stirred using the electrode probe for 10 seconds to ensure uniform composition and temperature of the sample. The electrode probe was positioned not to touch the bottom or sides of the container and the electrode surfaces completely covered by the sample. The test button was pushed and released for the automatic voltage ramp. The electrode was not moved during the measurement. At the stop of the ramp indicating voltage breakdown, the electrical stability reading was taken in volt. The test was repeated and the average of the readings was recorded as the electrical stability. The stability test was repeated increasing the egg yolk to 3, 5 and 8 ml. respectively In the same order, the test was repeated for the OWR of 80/20, 70/30 and 60/40. The results are presented in Section 4.2.1.

3.2.3.1.1 Formulation of diesel oil-based mud

As part of the preliminary mud formulation, a diesel oil-based mud of the same OWR (90/10, 80/20, 70/30, 60/40 and 50/50) were also formulated with the same composition shown in Table 3.2, but changing the base fluid to diesel oil and emulsifier with versaclean *VB* for comparison of the filtration properties of Jatropha oil-based mud.

3.2.3.1.2 Formulation of water-based mud

350 ml of water-based mud was also prepared using 22.5 g of bentonite and 4ml of sodium hydroxide for the pH control. This was prepared for phase II of this investigation for shale – fluid interaction. The mud composition is shown in appendix C1

3.2.3.2 Mud formulation using egg yolk as a non-toxic emulsifier

With the affirmation of the emulsifying ability of egg yolk from the preliminary test result, 70/30 OWR formulation was repeated using the composition shown in Table 3.3. This formulation is used for complete mud tests – stability, density, rheology and filtration for comparison with mud formulation using the standard emulsifier *versacleanVB.* The formulation was conducted in same order as in the preliminary formulation of Section 3.2.3.1. Similarly, another mud formulation was conducted using the composition in Table 3.3 with the egg yolk, replaced with *versacleanVB.*

Table 3.3: Mud composition

Component	**Quantity**
Base oil (Jatropha oil)	245ml
Water	105ml
Bentonite	22.5g
VersacleanVB emulsifier	8ml
Calcium Chloride	4g
Lime (Calcium Oxide)	12g

3.2.3.3 Testing of mud properties

Following the formulation of the mud, the basic properties of the mud were tested to ascertain the suitability for drilling operation. Properties tested include mud weight, electrical stability, rheology and filtration.

i. **Mud weight test**

The weight of the formulated mud was measured using the mud balance. The mud balance was thoroughly cleaned and dried to avoid any irregularities in the reading. While in the upright position; the cup was filled with the mud to the brim and covered with the lid firmly seated, while allowing some of the mud to expel through the vent to release any trapped air or gas. The outside of the cup was wiped and dried of any mud. The beam was placed on the base support and balanced by moving the rider along the graduated scale until balance was achieved when the bubble was centered. At this point the mud weight was read in pounds per gallon (ppg). The weight was 8ppg and pH measured with pH strip was 5.7. The density was increased to 10ppg by adding 117.6 g of barite , while the pH was adjusted to 9.5 using calcium oxide . The mud was further mixed for 30 minutes. The barite was added using the following relationship:

$$1470\left(\frac{w2-w1}{35-w2}\right) \qquad (3.1)$$

Where:

1470 = density (lb/bbl) of barite

35 = density (lb//gal) of barite

W1 = initial mud weight

W2= final mud weight

ii. **Electrical stability test**

The electrical stability test was conducted as in Section 3.2.3.1. However, the test was additionally repeated at a temperature of 120 ˚ C to examine the effect of temperature on stability. at elevated temperature.

iii. **Rheology test**

The plastic viscosity, yield point and apparent viscosity of the formulated mud samples were analysed after aging the mud for 16 hours. The 8-speed viscometer was used at the following dial settings 600, 300, 200, 100, 60,30,6 and 3 (in revolutions per minute). The mud was allowed to gel for 10 seconds and 10 minutes respectively to take measurement for the gel strength. The plastic viscosity, apparent viscosity and yield point were evaluated using the following relationships:

Plastic viscosity (PV) = dial reading for 600 rpm – dial reading for 300 rpm (3.2)

Yield point (YP) = dial reading for 300rpm – PV (3.3)

Apparent Viscosity (AV) = 600rpm / 2 (3.4)

The *PV* and *AV* were measured in centipoise (cP) while the yield point was measured in

(Ib/100 ft^2). This test was conducted at various temperatures of 50, 70, 100 and 120 ˚C to check any variation with temperature

iv. **Filtration test**

Low fluid loss is essential for wellbore stability. The filtration property was examined using the high temperature high pressure (HTHP) filter press (Figure 3.4 Section 3.2.1.3). The test was conducted at 120 ˚ C and 500 psi and the filtrate was collected at the end of 30 minutes in the following order:

- The heating power cord was connected to an appropriate power source and a dial – type metal thermometer was placed into the well and preheated to 10 ˚ F (6 ˚ C) above the desired temperature of 120 ˚ C. A pilot light comes on when the heating jacket is the desired temperature as indicated by the thermostat control knob. At this point, the needle valves on the test cell are closed by turning them counter-clockwise.

- The mud sample is stirred for 10 minutes with the viscometer at a high speed, the cell body is then inverted and the mud sample carefully poured into the cell. A space of 0.5 inches (13mm) was maintained below the O-ring groove to allow for heat expansion of the fluid.
- An O-ring was placed in the cell and another in the cap recess. A circle of filter paper was placed on top of the cell O-ring and slowly pushed the cell cap into the cell, ensuring that the arrow on the cell cap lined up with the arrow on the cell body.
- The cap locking screws and the valve stems were tightened. The cell was then placed in the heating jacket with the outlet or filter side of the cell pointed down. The cell and the heating jacket were rotated so that the pin in the bottom of the heating well was seated into the hole in the bottom of the test cell. This was to anchor the cell in the well and prevent it from rotating as the valve stems are opened and closed. The thermometer from the heating jacket was transferred to the thermometer well within the cell.
- The pressure assembly was connected to the top valve stem and locked in place with the safety pin. The back pressure receiver was placed on the bottom valve stem and also locked in place with safety the pin.
- Keeping the valve stem closed, the top and bottom regulators were adjusted to the recommended back pressure of 100 psi ensuring that the ball and bleeder valves were all closed. The top valve stem was opened a ½ turn to pressurise the sample. The pressure was maintained on the fluid until the temperature has stabilised.
- The heating time was not allowed to exceed one hour and thereafter the pressure on the top pressure unit was increased to 500 PSI (3448 kPa) more than the back pressure. The bottom stem was the opened ½ turn to initiate filtration.
- The filtrate at 10 seconds, 1 minute, 7.5 minutes and 30 minutes were collected, while maintaining the test temperature within ±5 ˚ F (±3 ˚ C). The 10 second collection point is precautionary as a fluid with little filtration properties may fill up the 15ml receiver tube almost immediately.
- At the end of the test, the top and bottom valve stems were closed to seal off the cell. The regulator T-screws was turned counter-clockwise to close off the flow of the pressurised gas. The outlet valve on the back pressure was opened to collect all of the remaining filtrate into the graduated cylinder.

- The filter press has a filtration area of 3.55in^2 (22.9cm^2), therefore the total filtrate volume collected was corrected to a standard filtration test area of 7.1 in^2 (45.8 cm^2) by doubling the filtrate volume collected in 30 minutes and recorded with the temperature, pressure and time.
- The primary assembly was disconnected by lifting the lock ring and the pressure assembly was slipped off the cell coupling. The cell was removed from the heating jacket and allowed to cool to room temperature.
- To save the filter cake, the cooled cell was kept upright with the outlet side down. Then The inlet valve stem was slowly opened to bleed off pressure from the cell body.
- The filter cake was washed on the paper with a gentle stream of water, measured and thickness reported to the nearest 1/32 inch (0.8mm)
- The apparatus was thoroughly cleaned and dried.

3.2.3.4 Formulation of 50/50 oil-water ratio invert emulsion mud

A major focus of this investigation is to formulate a low oil-water ratio mud of 50/50. This was made using the same procedure of preliminary mud formulation in Section 3.2.3.1. However, in order to get the right composition for this mud, four mud samples were made because of the high water content, excessive viscosity was observed. At the first instance, on the addition of the standard 22.5 g of bentonite, there was no flow. The quantity of the bentonite was reduced to half (11.3 g) then further reduced to 5.7 g without any significant flow. The fourth attempt was made with 3g, at this point flow occurred that would allow for the mud properties tests The composition used for the final sample is shown in Table 3.4.

The properties, stability, density, rheology and filtration were tested as described in Section 3.2.3.3. The 12ml of egg yolk stated was confirmed to be the best after six different quantities was assessed. The result is shown in Table 4.7 of Section 4.2.4.

Table 3.4: Mud Composition of 50/50 invert emulsion mud**.**

Component	Component
Base oil (Jatropha oil)	175ml
Water	175ml
Bentonite	3g
Egg yolk emulsifier	12ml
Calcium Chloride	4g
Lime(Calcium Oxide)	3g

3.2.3.5 Mud formulation after six months of aging of egg yolk

To ascertain if egg yolk loses its emulsifying ability overtime, eggs were aged for six months at 25 ˚ C and then used for mud formulation. The electrical stability test was repeated for mud samples of OWR 70/30 and 50/50 and were compared to the pre- aged samples

3.2.4 Errors and Accuracy

i. The arm of the mud balance is graduated and allows accurate measurements within ±0.1 pounds per gallon or ±0.01 specific gravity
ii. The speed accuracy of the 8-speed viscometer is 0.1rpm
iii. The electrical stability tester allows accurate measurement of ±2% of reading
iv. The filtrate of the high temperature-high pressure filter press was collected while maintaining the test temperature to within ±3 ˚C (±5 ˚ F)

3.3 Phase-II: Topology (Shale-fluid interaction)

As unconventional shale is the reservoir of interest for this investigation, the shale –fluid interaction by immersion test was conducted. Observations of the interaction of the core samples with water, jatropha and diesel oil-based muds were noted from one to seven days. The Phoenix x-ray computed tomography (CT) scanner equipped with image acquisition and processing software, (volume graphics studio max 2.2 software) was used for the scanning and processing of the core samples. The detailed description, mode of operation of the scanner and step by step method employed to conduct the experiment are described in this Chapter. Imaging and scanning will be used interchangeably.

3.3.1 Apparatus

3.3.1.1 The Phoenix V|Tome|X s Scanner X-Ray Computerised Tomography- CT scanner)

The v|tome|x s is a CT scanner of versatile high-resolution system for 2D X-ray inspection and 3D computed tomography (micro ct and nano ct) and 3D metrology. The v|tome|x s may is equipped with a 180 kV/15 W high-power nanofocus X-ray tube and a 240 kV/320 W microfocus tube for high flexibility. This unique combination of tubes, allows a very effective and reliable tool for a wide range of applications from extreme high-resolution scans of low absorbing materials to 3D analysis of high absorbing objects. The scanner is equipped with sample manipulator, radiation protection cabinet, two x-ray tubes, high voltage generator, control panel and a control console.

The sample manipulator is a motorised device used for positioning of the sample. It can be moved in 5 directions (x,y,z, rotation and tilt). The X-ray tubes are located to the right of the sample manipulator. The radiation protection cabinet is accessed through a sliding door at the front. The sliding door has an integrated pane of lead glass which allows the system operator a completely clear and safe view of the radiation protection cabinet

The x-rays beams emitted by the x-ray tubes hit the image intensifier (the detector) to the left of the sample manipulator. The x-ray image is converted by the detector to the format that can be displayed on screen. The integrated quality assurance imaging software can then be used to perform a comprehensive on screen analysis of the x ray.

The sample is manually clamped in place, whilst the sliding door is open. After closing the sliding door, the control console is used to adjust the manipulator until the sample is in the correct position to be penetrated by the beams.

This high resolution CT scanner manufactured by Phoenix x-ray in Germany is used in this study and shown in Figure 3.12. The system is equipped with image acquisition and processing software called volume graphics studio max 2.2.

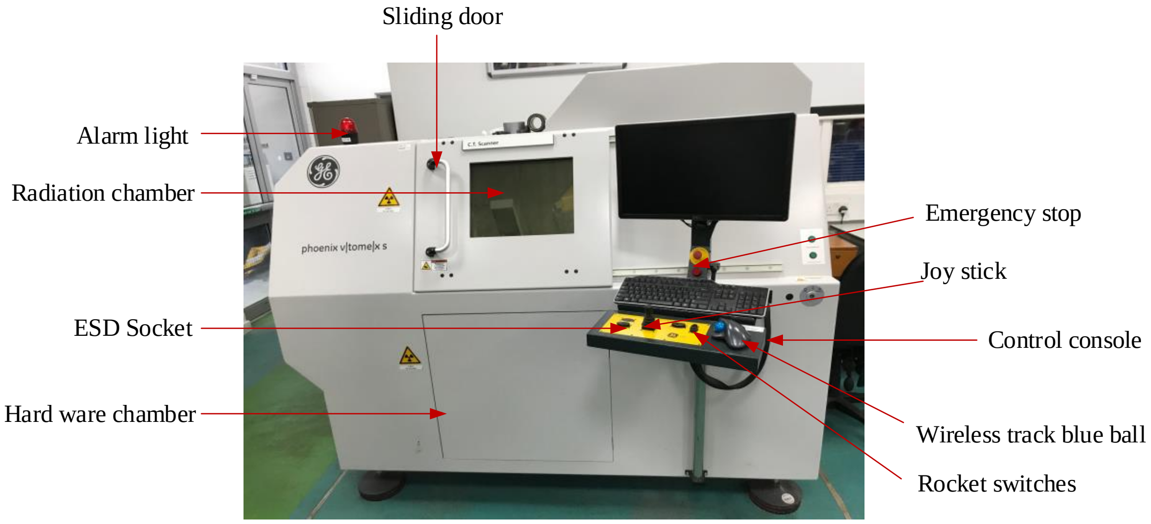

Figure 3.12: The Phoenix V|Tome|X S (Computerised tomography CT-scanner)

The CT- scanner comprises of the following components: radiation chamber, x-ray tubes, detector, control console, wireless track ball and screen

i. *Radiation chamber*

The radiation chamber is an all steel full protection cabinet with integral lead shield. The interior of the radiation cabinet is accessed through the sliding door with a handle. The sliding door has an integrated pane of lead glass which allows the system operator a completely clear and safe view of the radiation protection cabinet.

ii. *X-ray tubes*

The two x-ray tubes, a direct tube and a transmission tube are shown in Figure 3.13. The x-ray tubes, transmission and direct beam are located to the right of the radiation cabinet, opposite the detector. The cathode which generates the X-rays is located inside the tubes, while the beams exit the tube through the target.

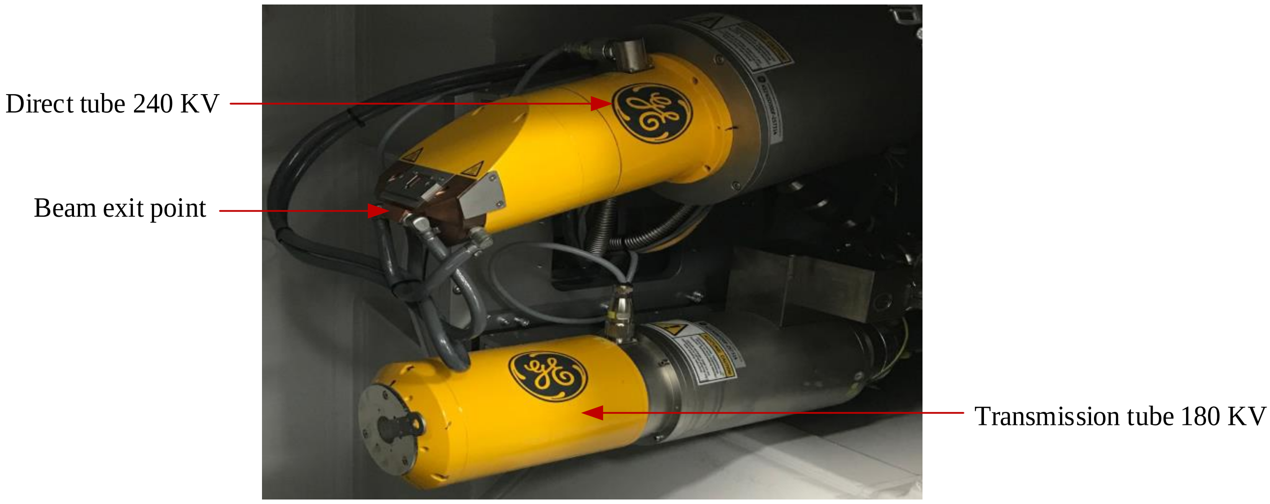

Figure 3.13: X-ray tubes

X-rays are emitted when excited electrons are decelerated upon striking a target. A tungsten filament is heated by passing a current through it (heating current/filament current UH) in the transmission tube until electrons are released. As a result of a difference in potential between filament (cathode) and anode, the electrons travel from cathode to anode, where they strike at one third the speed of light. The filament current is controlled by means of a grid, which is held at a negative potential (voltage UG). The beam escapes through a hole in the anode and is then directed onto an electromagnetic lens by a series of deflecting magnets where it is then collimated and focussed onto a target as shown in Figure 3.14

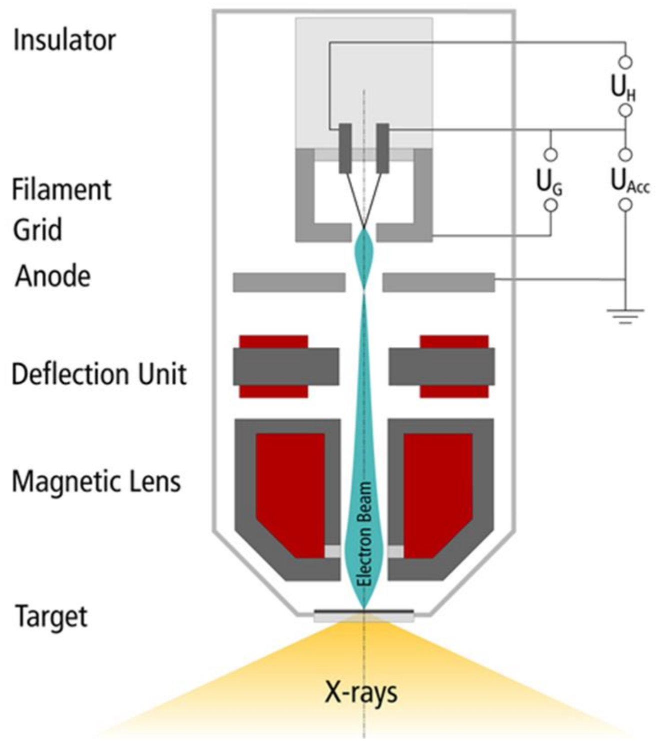

Figure 3.14: Schematic representation of the transmission tube (Zacher et al., 2014)

The transmission target consists of a thin layer of tungsten on a plate of light metal. As the radiation exits the window, this plate is doubled. The diameter of the focal spot dictates the size of the X-ray source, which is only a few microns in size. In the case of transmission targets, the X-ray source is located very close to the outer wall of the microfocus X-ray tube. This position allows the user to bring samples very close to the source ensuring highest magnifications.

The directional tube operates the same way as the transmission target X-ray tube, but due to the cylindrical and massive shape of the direct beam target, it is fit for voltages 10-20 times as high as the ones used for transmission tubes. The X-ray source of the direct beam target tube is located further away from the outer tube wall. This features makes this tube very suitable for the inspection of thicker, larger and denser samples such as castings as shown in Figure 3.15

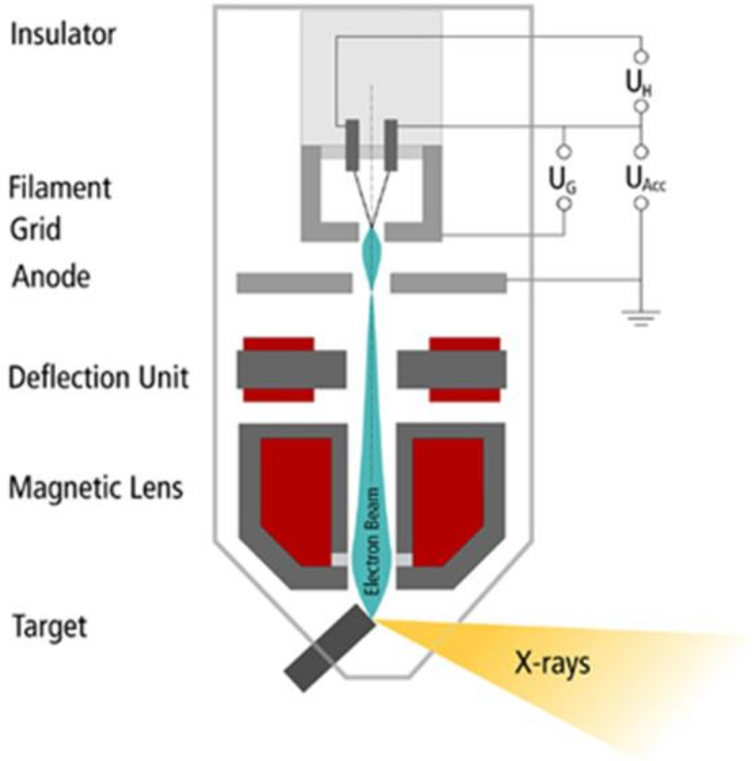

Figure 3.15: Schematic representation of the direct tube (Zacher et al., 2014)

iii. Detector

The detector shown is situated in the radiation protection cabinet to the left opposite of the X-ray tube. As the X-ray penetrates the sample, it hits the detector. The live image information is transmitted to the quality imaging software in real time

iv. Sample Manipulator

During scanning, the sample manipulator moves the sample in a horizontal (x, y) and a vertical (z) direction of the X-ray tube and detector. The sample can also rotate (rot) and ± 45^0 in the beam path. This is to allow for easy manipulation while scanning the sample.

v. Control console

The control console is the operational panel located on the front of the X-ray system beneath the screen. It is fitted with the following elements: key board, mouse rocker switches and joystick which is used to move the sample front and back. *A wireless track blue ball* is fitted on the mouse, which consist of a low battery indicator. The panel is also fitted with an integral Electrostatic Discharge (ESD) socket which diverts electrostatic discharge to prevent damage of sensitive components.

vi. Screen

The screen is a thin film transistor (TFT) monitor, located to the front of the system, above the control console. It is used for real time visualisation of displayed x-ray images.

3.3.2 Materials and preparation

3.3.2.1 Test shale sample and preparation

The core samples used in the present study are Marcellus shale of the Middle Devonian-age which can be found in the Pennsylvania, New York, Ohio and West of Virginia. It is a black, low density carbonaceous (organic) rich shale. Figure 3.16 shows the test sample before the 3D representation on the CT scanner.

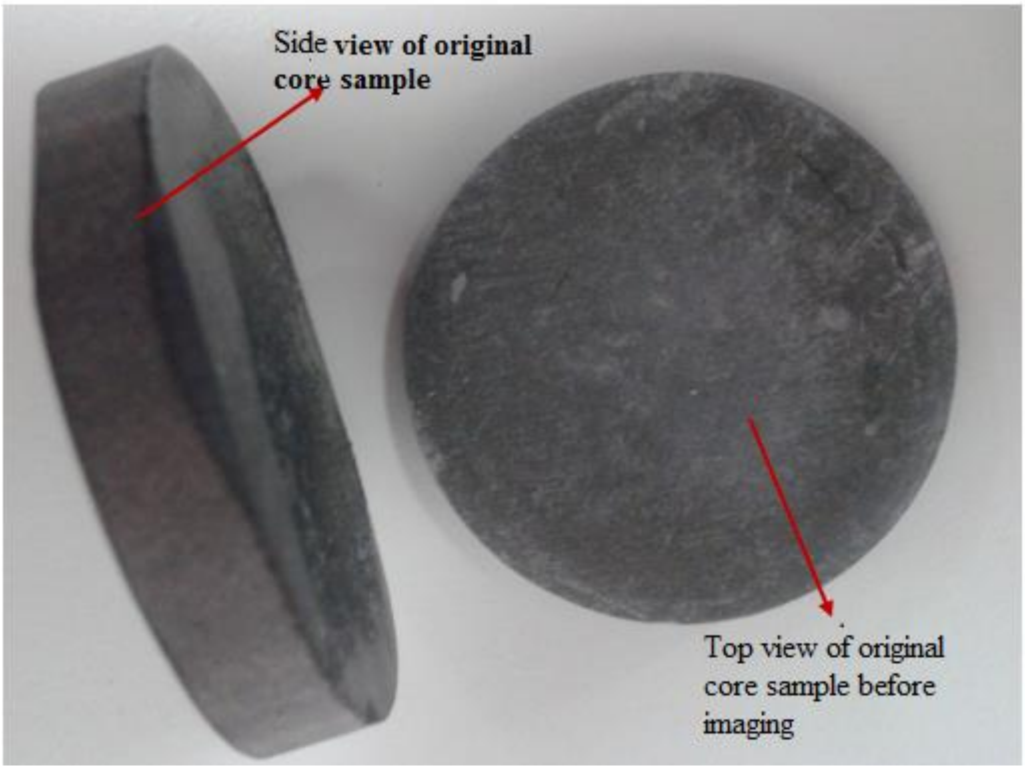

Figure 3.16: Original shale test sample before 3D representation on CT scanner

The size of the specimen must be small and properly shaped. This is to obtain the most efficient use of the detector to acquire the highest possible resolution, considering both the detector coverage during the sample rotation and the relation between sample size and resolution. Six small spherical core sample of 37.5 mm diameter with a thickness of 3.1 mm are drilled out of a larger core using a diamond core drill bit. The samples were weighed and conditioned in the oven at 50 ˚ C for 24 hours. The initial weight was 14.83 g of all the six samples. At the end of 24hours in the oven, the samples were re-weighed three times giving an average weight of 14.28kg. The conditioning of six samples was to avoid the immersion of a particular sample more than once into a mud sample. The six samples were used for observation for one and seven- days immersion tests in the three mud samples- water-based, diesel and jatropha oil-based muds. After this conditioning, samples are ready to be imaged. Each was aligned vertically onto the sample stage for best resolution and acquisition.

3.3.3 Procedure

3.3.3.1: CT Scanner data acquisition

The components of the CT scanner, showing the basic data acquisition process is shown in Figure 3.17.

The basic principle consists of an X-ray source, a sample manipulator stage and a detector. The X-ray source generates the beams, while the sample manipulator positions and rotates the sample with high precision. The generated attenuated profiles are collected by the detector as raw information for reconstruction.

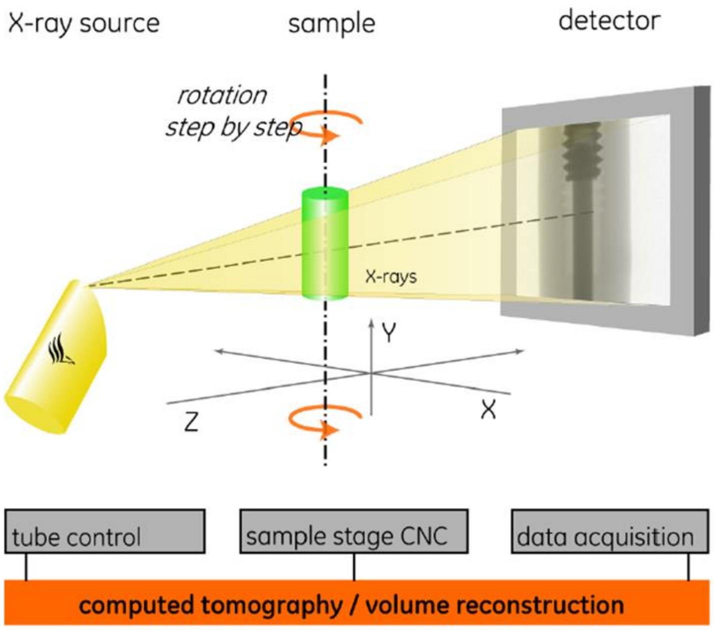

Figure 3.17: Schematic Set-up of CT scanner data acquisition process (Zacher et al., 2014)

The scanner uses an industrial tube to generate a bundle of X-rays as a cone beam. During the scanning, the sample has to be imaged at least every half degree and rotated 360 degrees for a comprehensive set of radiographic profiles for the reconstruction

3.3.3.2: Imaging of sample with CT Scanner.

The samples were scanned using the following parameters:

- *Positioning of Sample*

The sample must be properly positioned using the control panel. It must be ensured that the sample fills the field of view as fully as possible while retaining a band of clear air to the left and right of the specimen throughout the rotation. Quick scans are taken for every 45^o to ensure that edges of the sample are more than 20-25 pixels from the edge of the detector at each view. The sample has to be regulated and aligned properly by the sample manipulator, to achieve the highest possible resolution

- *Sensor Calibration*

The energy of X-ray has to be decided by tuning the voltage. A value of 80kV is sufficient for good imaging. The resultant current is around 80µA. Correction images are then acquired to remove any homogeneity in the background images. These are images with only air between source and detector. Two of the correction images are required, one acquired while the X-rays is on and the other with the X-rays switched off. Table 3.5 shows the imaging parameters of the shale sample.

Table 3.5: Shale sample imaging parameters

Geometry	
Magnification	91.278049
Acquisition	
Number of images	1776
Detector	
Type	Dxr-250 rt
Timing	1000ms
Average	5
Skip frames	1
X-ray	
Voltage	100 kV
Current	111µA
Tube mode	1

3.3.3.3 Immersion test

The immersion test is one of the most efficient methods of evaluating fluid-rock interaction and fracture development, as it directly shows the interaction between the fluid and the shale. As the name implies, the test involves immersion of selected shale samples into chosen fluids over a period of time. Observations are then made to see if there are changes and developments in the structure of the shale sample. The test was conducted for one and seven days. Figure 3.18 shows the 3D representation of shale core sample before immersion in the mud

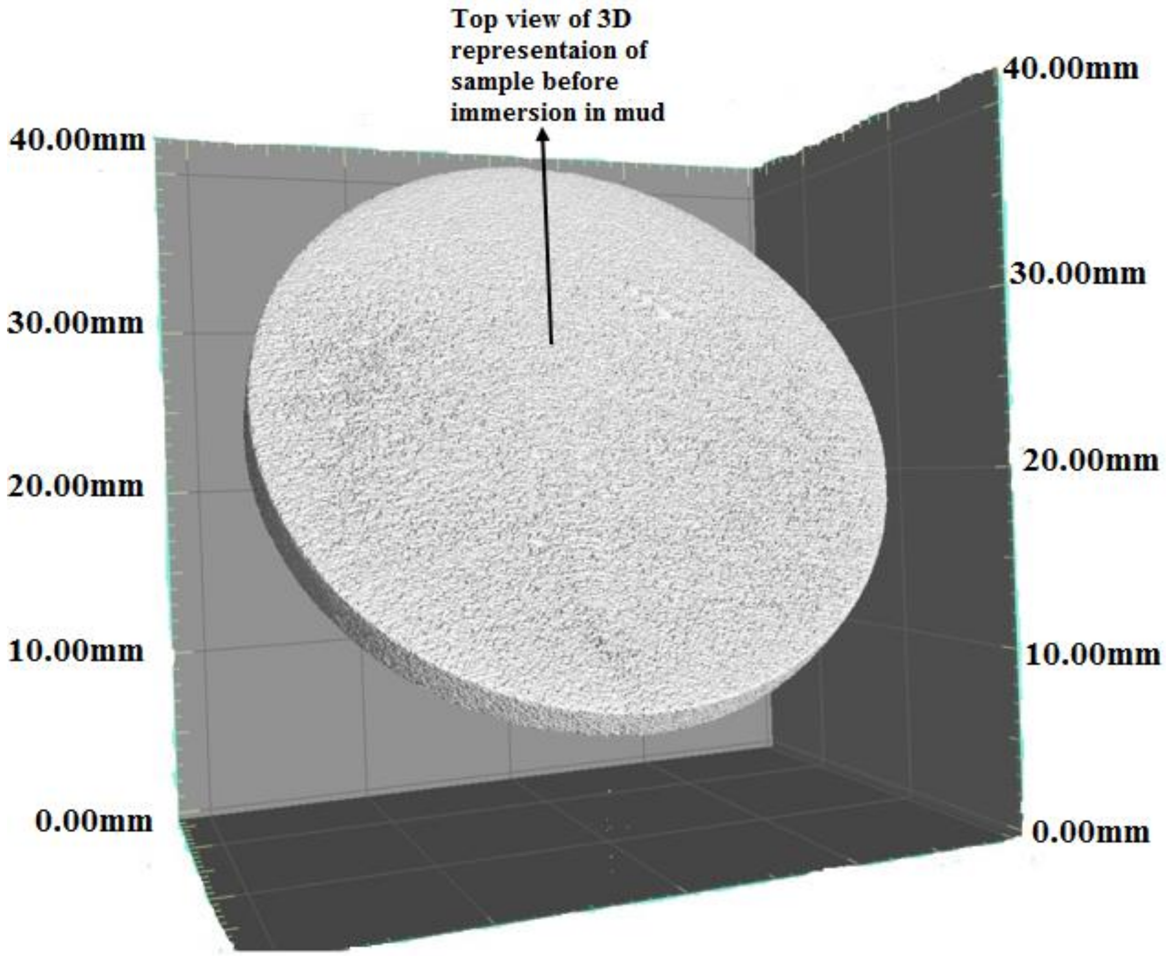

Figure 3.18: 3D representation of shale core sample before immersion in mud

i. One-day immersion in water-based, diesel and jatropha oil-based muds.

Following the imaging of the samples, three samples were immersed one for each of the three mud samples - water–based mud, 50/50 oil-water ratio diesel and jatropha oil-based invert emulsion muds. At the end of the day, the samples were re-scanned and the acquired images are presented in Section 4.3.2

ii. Seven-day immersion in water-based, diesel and jatropha oil-based muds.

To further investigate the extent, of the shale –fluid interaction, the immersion test was repeated leaving the samples immersed for seven days using similar shale sample size as shown in Figure 3.18. In the same manner as in the one-day test, the samples were re-scanned at the end of the seventh day. The percentage variation in volume distribution of the test core samples was analysed using VG Studio Max 2.2 software discussed in Section 2.6

3.3.4: Errors and accuracy of the Phoenix V|Tome|X s scanner

Resolution deteriorates during scanning by contamination of the sample, column, or apertures. This is as a result of interaction of the beam with organic volatiles and causes them to polymerise. Polymerisation occurs primarily where the beam is the most intense (crossover points), such as at the apertures and on the sample. Organic volatiles result from oil diffusion pumps, rubber vacuum seals, vacuum grease, and fingerprints, as well as the sample itself.

3.4 Chapter summary

This section summarises the experimental work carried out in the two phases of the present study

Phase I - Mud formulation and testing

- The emulsifying ability of egg yolk was compared to that of a conventional emulsifier *VersacleanVB*
- 50/50 oil-water invert emuslaion was formulated and the properties compared to that of 70/30 OWR which is commonly used.
- Properties of diesel and jatropha oil-based muds were compared.

Phase II- Topology- Shale-fluid interaction

- Shale-fluid interaction was investigated by immersion tests using Marcellus shale samples in water-based, diesel oil and jatropha oil-based muds for one and seven days. This was to ascertain any development or enlargement of fractures in the shale samples to indicate the level of interaction with the different mud samples.

- The shale core samples were scanned before and after the immersion test using the CT scan and captured images were analysed with the Volume Graphics (VG) Studio Max 2.2 software.

Chapter 4

Results and Discussion

4.1 Introduction

This chapter presents the results obtained from this experimental investigation conducted as outlined in the previous chapters shown in the work flow chart of Figure 3.1. This is in accordance with the main focus of this investigation in utilising egg yolk as non-toxic emulsifier in a low oil –water ratio invert emulsion with a vegetable oil continuous phase. A low oil-water ratio mud capable of reducing fluid loss for wellbore stability was investigated in the topology (shale-fluid interaction) and the economic benefit of this invert emulsion was also considered. The results are classified into two phases in accordance with the experimental sequence.

- **Phase I:** Mud formulation and testing: Section 4.2 discusses the formulation of an invert emulsion mud utilising egg yolk as an emulsifier and a low oil water ratio mud of 50/50. The result of the testing of experimental materials is presented in Section 4.2.1 followed by the results of the preliminary mud formulation in Section 4.2.2. The emulsifying ability of egg yolk and the low fluid loss of the 50/50 invert emulsion are detailed in Section 4.2.3 and 4.24. The qualitative results have been illustrated in relation to non-toxic and compatible additives and also low fluid loss which is beneficial to wellbore stability.
- **Phase II:** Topology (shale-fluid interaction). Shale-fluid interaction was investigated qualitatively by immersion test for one and seven days using the CT scanner imaging, and for analysis with the VG studio max 2.2 software as was detailed in Section 3.3.3.

4.2 Phase I- Mud formulation

4.2.1 Experimental materials

The first stage of the mud formulation was the preparation of experimental materials which involved the testing of experimental materials, in this case the properties of the diesel and jatropha oils and the basic physical requirement of bentonite. Tables 4.1 shows the density and rheological properties of diesel and jatropha oils. Figure 4.1 represents the viscosities of diesel and jatropha oils.

Table 4.1: Rheological properties and density of diesel andjJatropha oils

Dial Setting (RPM) 50 ˚ C	**Dial Reading**	
	Diesel oil	**Jatropha oil**
600	8	147
300	5	77
200	3	52
100	2	25
60	2	16
30	2	16
6	2	2
3	1	2
Gel Strength (10 seconds) (Ib/100 ft^2)	1	2
Gel Strength (10 minute) (Ib/100 ft^2)	1	2
Apparent Viscosity (cP)	4	73.5
Plastic Viscosity(cP)	3	70
Yield Point (Ib/100 ft^2)	2	7
Density (ppg)	7.0	7.8

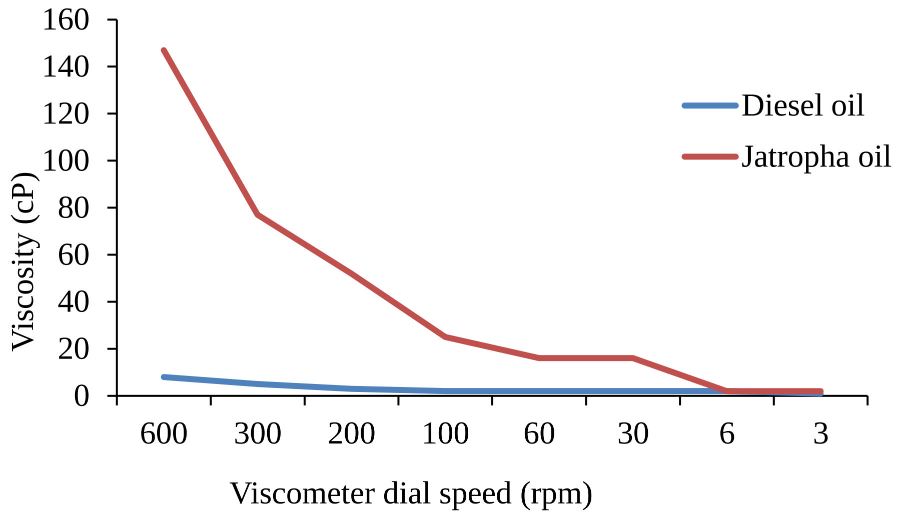

Figure 4.1: Comparative analysis of viscosity of diesel and jatropha oils

From the results, there is no significant variation in the densities of the two oils, with 7ppg for diesel and 7.8ppg for Jatropha. However, Jatropha exhibited a higher viscosity compared to diesel oil. High viscosity has been noted as a challenge in this trend of vegetable oil-based muds, however, in the present study the issue of high viscosity was managed by guided empiricism.

Table 4.2 shows the measured value of the basic physical requirement of the organophilic bentonite used in this investigation as measured against the API standard.

Table 4.2: Basic Physical Requirement of Bentonite

Requirement	**Measured Value**	**API Standard**
600	61	30 (minimum)
300	52	Not applicable
Plastic Viscosity	9	Not applicable
Yield Point	43	Not applicable
Yield Point / Plastic viscosity Ratio	4.8	6(maximum)
Filtrate volume	13.5	16(maximum)

The values from the measured parameters as shown above were within the acceptable range of API standard. The organophilic clay bentonite as mentioned in Section 3.2.2.1 is a white free flowing powder which is oil dispersible suitable for oil-based mud.

4.2.2 Preliminary mud formulation and tests

Invert emulsion mud of OWR of 90/10, 80/20, 70/30 and 60/40 were formulated, prior to the formulation of the determined 50/50 oil-water ratio. The electrical stability test was first conducted to ascertain the emulsifying ability of egg yolk. The electrical stability values obtained are shown in Table 4.3

Table 4.3: Electrical Stability

Egg yolk (ml)	**Electrical Stability (V)**			
	90/10	**80/20**	**70/30**	**60/40**
1	492	291	232	187
3	568	350	285	225
5	573	389	361	257
8	615	421	398	315
Required range	(Above 600)	300-400	200-300	200-300

The test was conducted four times using varied volumes of the egg yolk of 1, 3,5 and 8 ml. This was because, the first formulation of OWR of 90/10 could not achieve the estimated electrical stability value required of mud within that range. According to Lyons and Plisga (2011) the estimated requirement of electrical stability value of OWR of the range 88/12-92/8 is above 600V. The stability value of 615V was attained when 8ml of the egg yolk was added to the mud. At this point, the emulsifying ability of egg yolk was established. In the same order the electrical stability value requirement for 80/20 is 400-500V, 70/30 is 300-400V while 60/40 was accepted with same range of 65/35-75-25, which is 200-300V.

This qualitative result of the emulsifying ability of egg yolk for vegetable oil-based mud has answered one of the research questions in line with Apaleke et.al., (2012a) of the need to replace toxic additives with less toxic and organic ones, also for compatibility with the vegetable oil continuous phase. Bakhtyar and Gagnon (2012), also noted that a primary emulsifier – *emulS50* gave the strongest biomarker response in a toxicity assessment of individual ingredients of a synthetic based mud. The emulsifying ability of egg yolk is a step in the right direction for non-toxic additives compatible with vegetable oil-based mud

4.2.3 Mud formulation ssing egg yolk as a non-toxic emulsifier

The emulsifying ability of egg yolk as a non- toxic emulsifier in an environmentally friendly invert emulsion drilling mud using jatropha oil as a base fluid was evaluated. This was to support the drive for non-toxic and compatible additives with the vegetable oil continuous phase. The electrical stability values of the mud emulsified by egg yolk and the mud emulsified by *versaclean –VB* is presented in Table 4.4 and graphically illustrated in Figure 4.2

Table 4.4: Electrical Stability values Using egg yolk and versclean-VB emulsifier

Temperature (˚C)	**Mud Electrical Stability (V) with Egg yolk**	**Mud Electrical Stability (V) with Versaclean VB**
48.9	398	201
120	289	188

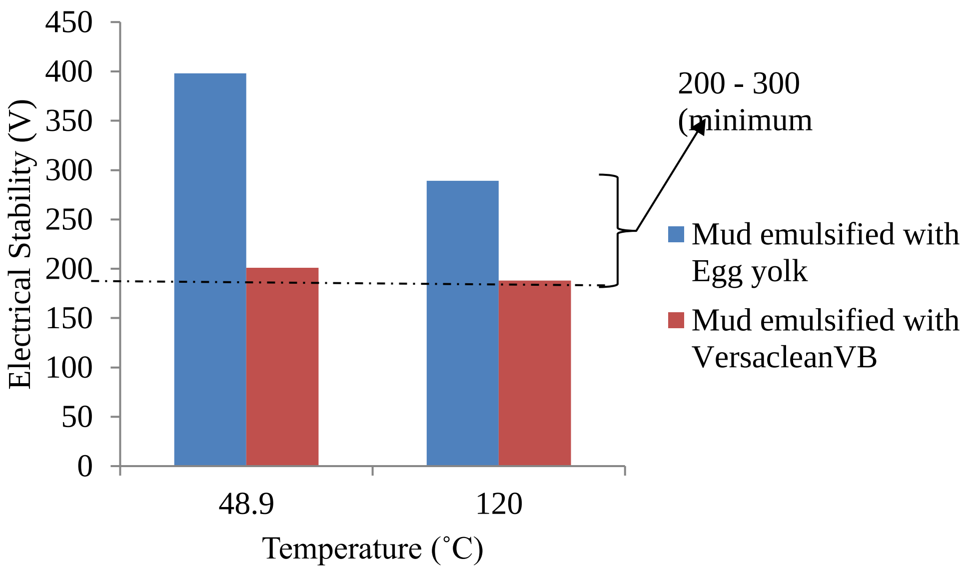

Figure 4.2: Comparative analysis of the electrical stability using egg yolk and versacleanVB

The electrical stability of the mud emulsified with egg yolk at 48.9 ºC was 398V and decreased to 289V at 120 ˚ C. In the same order the electrical stability of the mud emulsified with versacleanVB was 201V at 48.9 ˚ C and decreased to 188V at 120 ˚ C. This indicates that the mud was more stable with the egg yolk than with the *versaclean VB* emulsifier implying compatibility with the vegetable oil continuous phase as shown in Figure 4.3. In line with compatible additives and the replacement of toxic additives with non-toxic ones for environmental concerns, egg yolk has demonstrated a potential as an emulsifier substitute for vegetable oil based mud. Apaleke et.al. (2012a) emphasised the need of replacement of toxic additives compatible with vegetable oil continuous phase. Also, in toxicity assessment of individual ingredients of synthetic based drilling muds, the emulsifier caused the strongest biomarker responses. The primary emulsifier (Emul S50) followed by the fluid loss agent (LSL 50) caused the strongest biochemical responses in fish (Bakhtyar and Gagnon 2012). The electrical stability values were within the acceptable range of 200-300 volts of oil-based mud of mud weight of 8-10 ppg with oil-water ratio 65/35-75/25 (Lyons and Plisga, 2011)

Figure 4.3: Mud emulsified with egg yolk and (b) mud emulsified by versacleanVB

From Figure 4.3, it can be seen that the mud emulsified with egg yolk showed a full stable mud compared to the mud emulsified by *versaclean VB* with evidence of slight phase separation and some bubbles of oil on top indicating a low-stable emulsion. In the same order, the measured mud weight and the rheological properties of the mud emulsified by egg yolk and *versaclean VB* are presented in Tables 4.5 and 4.6 respectively and graphically shown in Figure 4.4

Table 4.5: Rheology variation with temperature for mud emulsified with egg yolk

Dial Setting (RPM)	**50 ºC**	**70 ºC**	**100 ºC**	**120ºC**
600	173	160	64	49
300	121	109	43	32
200	90	75	29	22
100	58	46	23	19
60	37	30	21	15
30	25	22	17	13
6	21	15	14	11
3	14	13	12	10
Gel Strength (10seconds) (Ib/100 ft^2)	14	12	10	11
Gel Strength (10minute) (Ib/100 ft^2)	15	13	11	12
Apparent Viscosity (cP)	85.5	80	32	24.5
Plastic Viscosity (cP)	52	51	21	17
Yield Point (Ib/100 ft^2)	69	58	22	25
Mud weight (ppg)	10	10	10	10

Table 4.6: Rheology aariation with temperature for mud emulsified with versacleanVB

Viscometer Dial Setting (RPM)	**50 ˚ C**	**70 ˚ C**	**100 ˚ C**	**120 ˚ C**
600	168	157	62	47
300	119	107	42	30
200	89	72	26	20
100	53	42	20	16
60	31	27	17	13
30	20	19	13	11
6	15	10	11	9
3	13	9	10	10
Gel Strength,10seconds (Ib/100 ft^2)	13	11	10	11
Gel Strength,10minute (Ib/100 ft^2)	14	12	11	12
Apparent Viscosity (cP)	84	78.5	31	23.5
Plastic Viscosity (cP)	49	50	20	17
Yield Point (Ib/100 ft^2)	70	57	22	13
Mud weight (ppg)	10	10	10	10

Figures 4.4 illustrates the variation in viscosities with temperatures of the muds formulated using egg yolk (EY) and *versacleanVB* as emulsifiers respectively. There is no significant variation in the viscosity of the mud emulsified by egg yolk and that of versacleanVB. This further indicates the suitability of egg yolk as potential substitute for a conventional emulsifier

The viscosity of each of the muds steadily decreased as the temperature increased from 50 ˚ C to 120 ˚ C.,the viscosity was highest at 50 ˚ C and lowest at 120 ˚ C. At 50 ˚ C and viscometer dial speed of 600 the viscosities were 173 and 168 cP for the mud emulsified with egg yolk and the mud emulsified with *versacleanVB* respectively. At increased temperature of 120 ˚ C, at the same dial speed of 600, the viscosities of the mud samples were reduced to 49 and 47cP respectively.

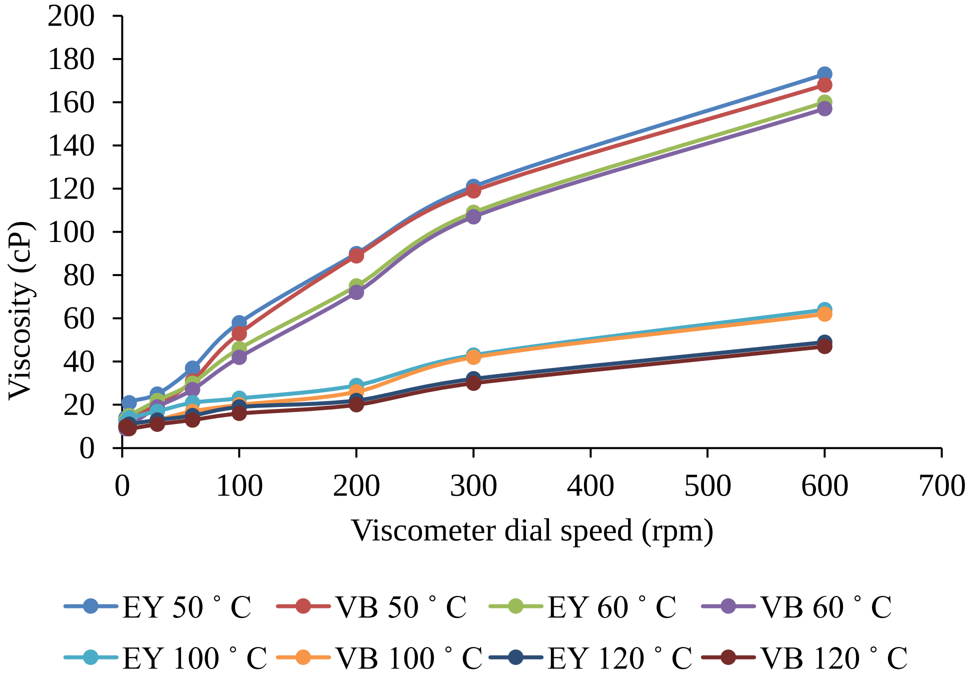

Figure 4.4: Viscosity Variations with Temperature of the Mud emulsified by Egg yolk (EY) and standard emulsifier (versacleanVB**)**

4.2.4 Formulation of 50/50 oil water ratio invert emulsion mud

50/50 OWR mud was formulated and the properties were compared with that of 70/30 OWR mud. The sample of the 50/50 and 70/30 OWR muds is shown in Figure 4.5 The electrical stability was conducted six times to get an optimum value due to the high water content of the mud. This was necessary to maintain stability when downhole water is encountered. The result of the six stability test is shown in Table 4.7

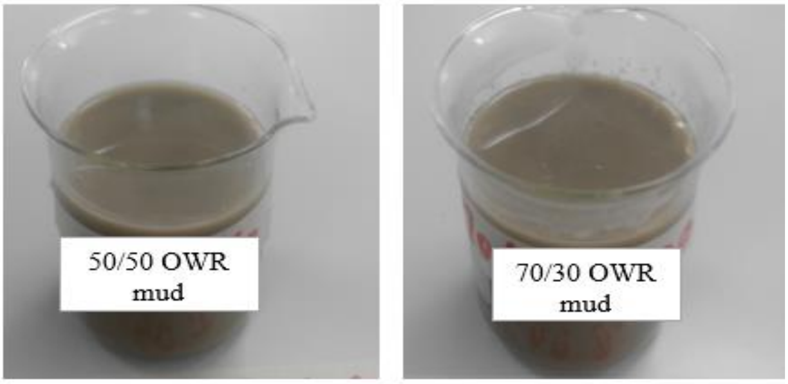

Figure 4.5: Sample of 50/50 and 70/30 OWR muds

The electrical stability was conducted six times to get an optimum value due to the high water content of the mud. This was necessary to maintain stability when downhole water is encountered. The result of the six stability test are shown in Table 4.7

Table 4.7: Electrical Stability of 50/50 OWR

Egg yolk (ml)	Electrical Stability (V)
1	192
3	238
5	263
8	301
10	338
12	353

4.2.4.1 Electrical Stability values of 50/50 and 70/30 OWR muds

The result of electrical stability test, rheological and filtration properties of the 50/50 and 70/30 OWR muds is presented in Table 4.8

Table 4.8: Electrical Stability Values of 50/50 and 70/30 OWR muds

Temperature (ºC)	**Mud Electrical Stability (V) 50/50 OWR Mud**	**Mud Electrical Stability (V) 70/30 OWR**
48.9	353	480
120	258	393

Figure 4.6 compares the electrical stability of the 50/50 and 70/30 OWR muds. The electrical stability of the 50/50 OWR mud compared favourably with 70/30 OWR mud without much variation. The electrical stability values at 48.9 ˚ C were 353 v and 480 v for the mud of 50/50 and 70/30 OWR muds respectively. However, the electrical stability of the 50/50 OWR mud decreased to 258V at 120 ˚ C. In the same order the electrical stability of the 70/30 OWR mud decreased to 393V. The values were within the estimated range of the estimated electrical stability of 200-300 volts for oil-based mud of mud weight of 8-10 ppg of OWR 65/35-75/25.

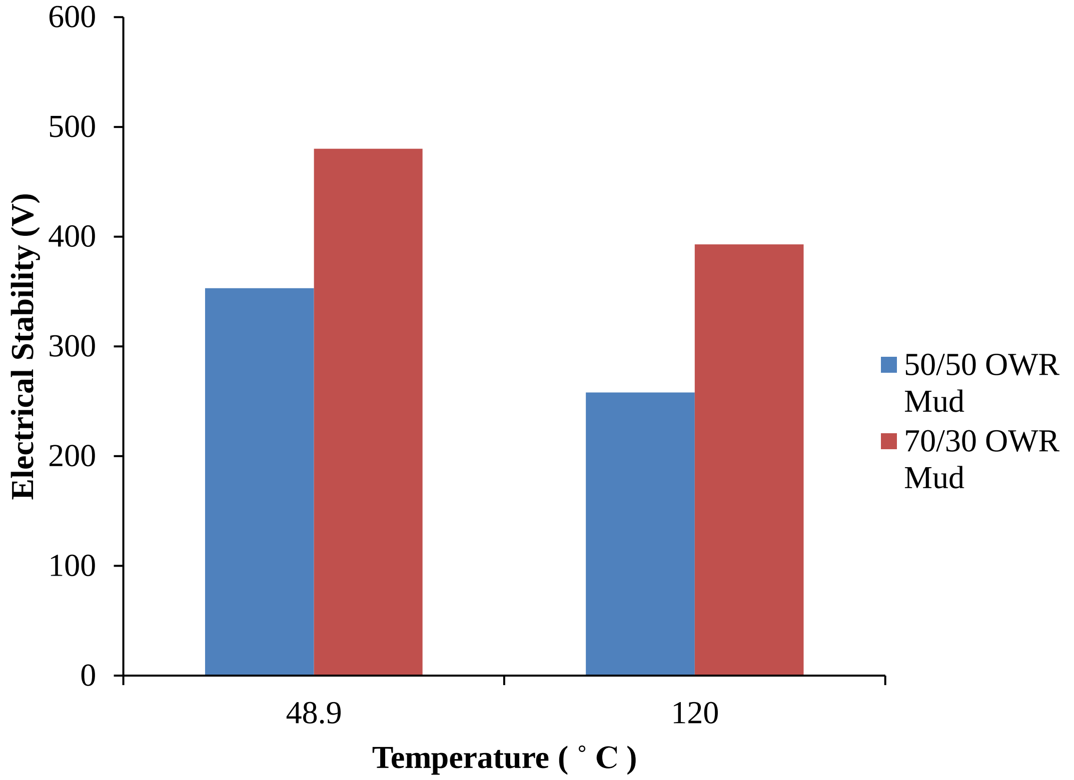

Figure 4.6: Comparative analysis of the electrical stability of 50/50 and 70/30 OWR muds

This qualitative results have shown that a stable low OWR of 50/50 emulsified with egg yolk is feasible with a vegetable oil continuous phase**.** This has also answered one of the research questions of achieving a low oil-water ratio mud with a vegetable oil continuous phase.

4.2.4.2 Rheological Properties

Rheological properties of 50/50 and 70/30 OWR muds were compared as shown in Table 4.9

Table 4.9: Rheological Behaviour of 50/50 and 70/30 OWR muds

Viscometer Dial Setting (RPM) 50 ºC	**Dial Reading**	
	50/50 OWR Mud	**70/30 OWR Mud**
600	89	79
300	53	48
200	38	40
100	23	36
60	15	16
30	9	11
6	3	9
3	2	6
Gel Strength,10 seconds (Ib/100 ft^2)	3	6
Gel Strength,10 minute (Ib/100 ft^2)	4	7
Apparent Viscosity (cP	44.5	39.5
Plastic Viscosity(cP)	36	31
Yield Point (Ib/100 ft^2)	17	17

Figure 4.7 compared the viscosities of the 50/50 and 70/30 OWR mud samples.

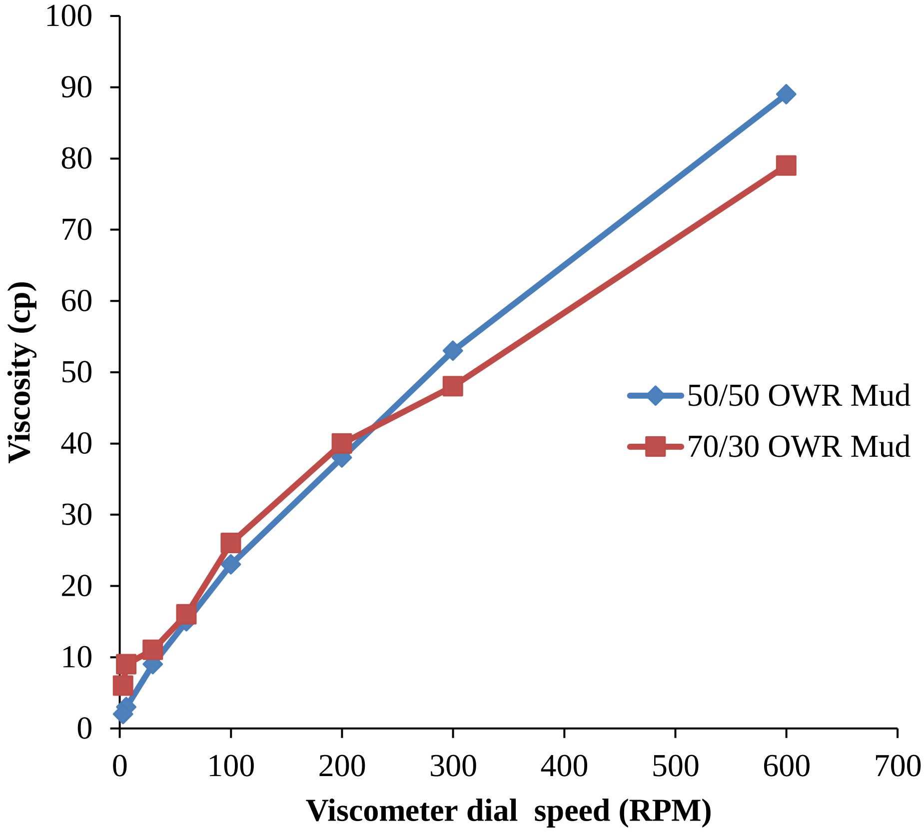

Figure 4.7: Comparative analysis of the viscosity 50/50 and 70/30 OWR muds

There was no significant variation in the plastic viscosity of the 50/50 and 70/30 OWR muds. As shown in Table 4.9, the plastic viscosity of the 50/50 OWR mud was 36cp and a yield point of 17 Ib/100 ft^2 while that of 70/30 OWR mud had plastic viscosity of 31cp and yield point of 17 Ib/100 ft^2. The 10 seconds and 10 minutes' gel strength of the 50/50 OWR mud was 3 Ib/100 ft^2 and 4 Ib/100 ft^2 while that of 70/30 OWR mud was 6 Ib/100 ft^2 and 7 Ib/100 ft^2 respectively.

4.2.4.2.1 Gel Strength

The gelling characteristics of drilling fluid has been described as the ability of the mud to suspend cuttings when circulation is stopped. The gel strength of the 50/50 and 70/30 OWR muds were compared, as represented in Figure 4.8. The 10 seconds and 10 minutes gel strength of the 50/50 OWR mud were 3 and 4 Ib/100 ft^2 respectively, while that of 70/30 OWR mud were 6 and 7 Ib/100 ft^2. Both muds exhibited a low -flat gel profile, which is desirable. This indicates that the muds will remain pump-able if left static in the hole with time. This further suggests the suitability of the 50/50 OWR mud compared to 70/30 OWR mud. The comparison of the properties of the 50/50 mud to that of the 70/30 mud was to ascertain its suitability, because the 70/30 mud is the widely used mud.

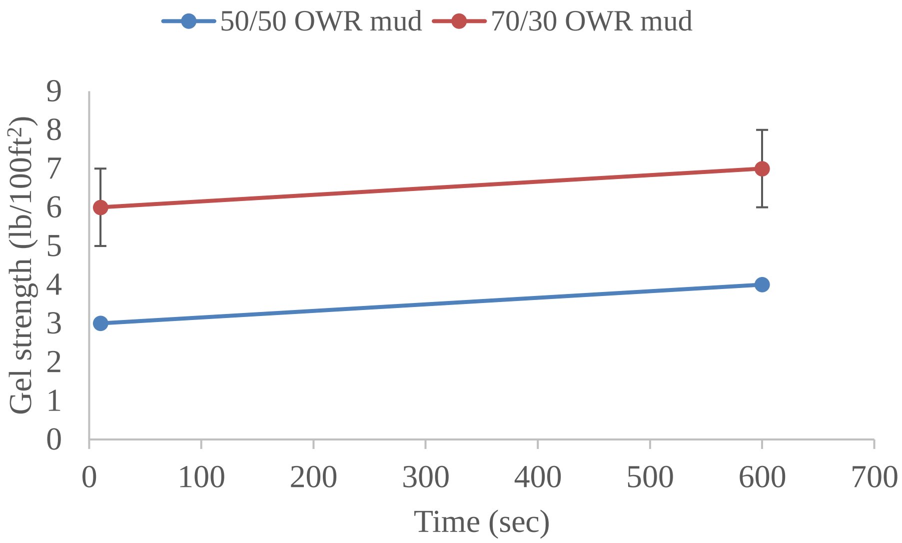

Figure 4.8: Comparative analysis of gel strength of 50/50 and 70/30 OWR Muds.

4.2.4.3 Filtration

The presence of an emulsion phase is important to reduce fluid loss; therefore, it is expected that oil-water ratio will affect fluid loss (Aston et.al.,2002). The 30 minutes fluid loss for the 50/50 OWR mud was 6ml and that of the 70/30 mud was 12ml as shown in Figure 4.9. The lower filtrate volume is due to the ability of emulsion droplets to provide thin filter cake (mud cake) while drilling Amanullah, (2005) noted that Duratone used as Fluid-loss control additive in mineral oil based muds is not suitable for vegetable oil-based mud; hence the need for a suitable fluid loss additive for vegetable oil-based muds. While the industry works on a suitable fluid loss for vegetable oil, a low oil-water ratio mud of 50/50 has the potential for a low fluid loss. The filtration property of the 50/50 and 70/30 OWR muds were compared as shown in Table 4.10 and illustrated in Figure 4.9

Table 4.10: Fluid Loss of 50/50 and 70/30 OWR Mud at 120 ºC and 500 Psi

30 minutes	50/50 OWR mud	70/30 OWR mud
Fluid loss (ml)	6	12

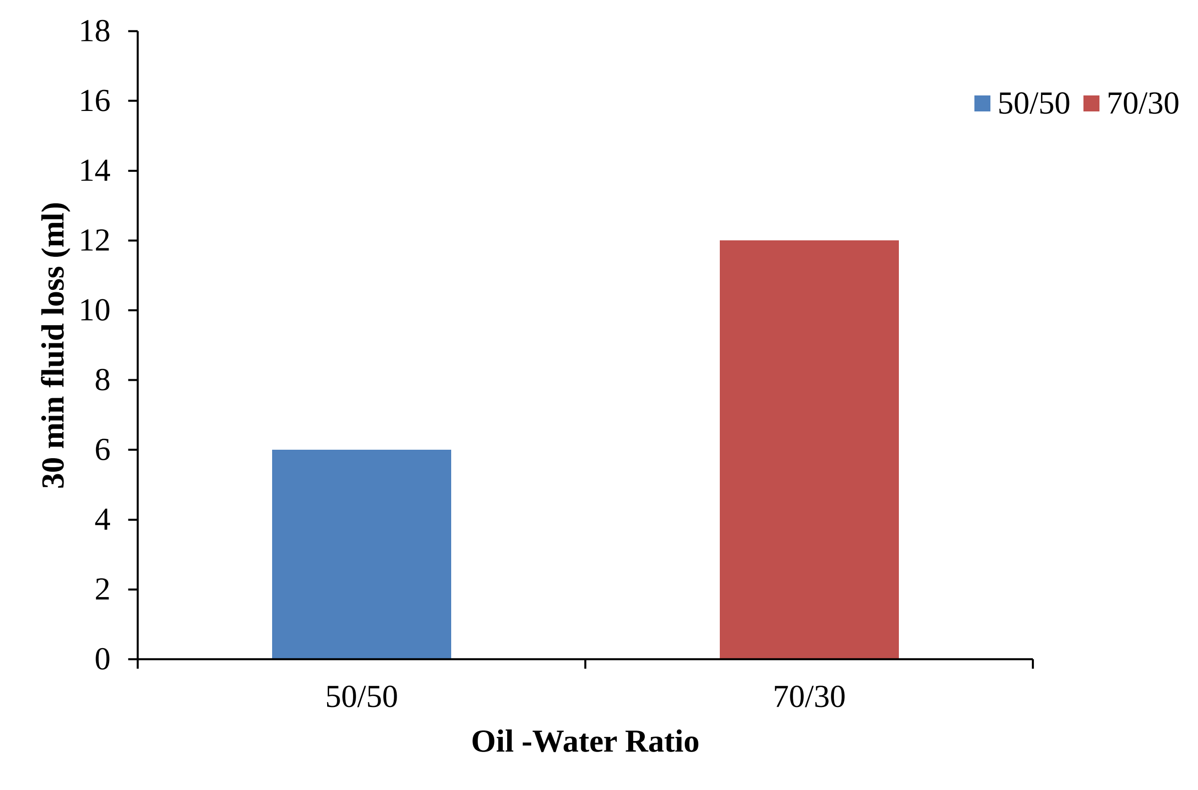

Figure 4.9: Comparative Analysis of the Fluid loss of 50/50 and 70/30 OWR Mud after 30 minutes

The 50/50 OWR mud had a thin filter cake of 1.7 mm while the 70/30 mud gave 3.2 mm as show in Figures 4.10 a and 4.10 b. The thin but soft filter cake of the 50/50 OWR mud was below 2mm as required of a mud cake. This is desirable as it controls loss of filtrate into the formation while drilling but also allows the hydrocarbons to flow back during production. A thick filter cake could result in a stuck pipe, high torque and drag. There was no fluid loss additive in the mud formulation. Higher water content clearly improves the filtration property. These results have shown that a 50/50 OWR invert emulsion mud with vegetable oil continuous phase is feasible and compatible with drilling mud requirements. Despite the higher water content, the electrical stability and rheological behaviour of the 50/50 OWR mud compared favourably with the usual 70/30 OWR mud while achieving a lower fluid loss which is essential for wellbore stability.

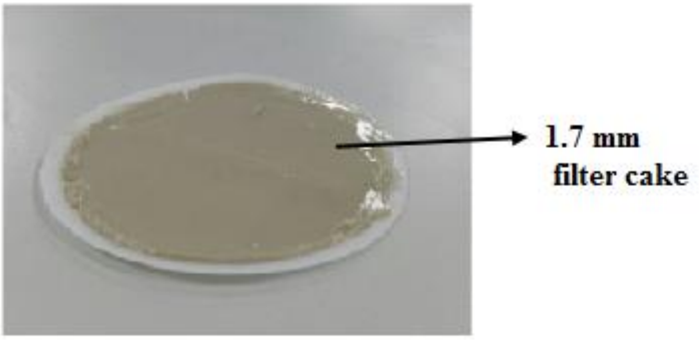

Figure 4.10 a: Filter cake of 50/50 OWR Mud

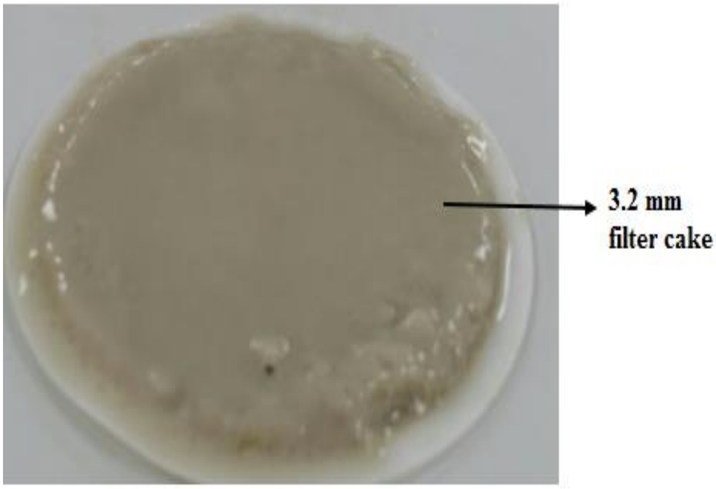

Figure 4.10b: Filter cake of 70//30 OWR Mud

For full comparison, the filtration property of diesel and jatropha oil-based muds of the following OWR 90/10, 80/20 and 60/40 were also evaluated as shown in Table 4.10. All tests were done three times and the mean values taken. The was no significant variation in the filtrate volumes of the DOBM and JOBM. The 50/50 OWR filtrate volume was the least, which is more beneficial to wellbore stability.

Table 4.11: Fluid Loss of Diesel and Jatropha muds for various OWR at 120 ˚ C and 500 Psi

	Fluid loss (ml)	
OWR	**DOBM**	**JOBM**
90/10	15	16
80/20	13	14
70/30	10	12
60/40	7	9
50/50	5	6

Figure 4.11 represents full comparative analysis of fluid loss variation with various OWR ranging from 90/10 to 50/50 for diesel and jatropha oil-based muds.

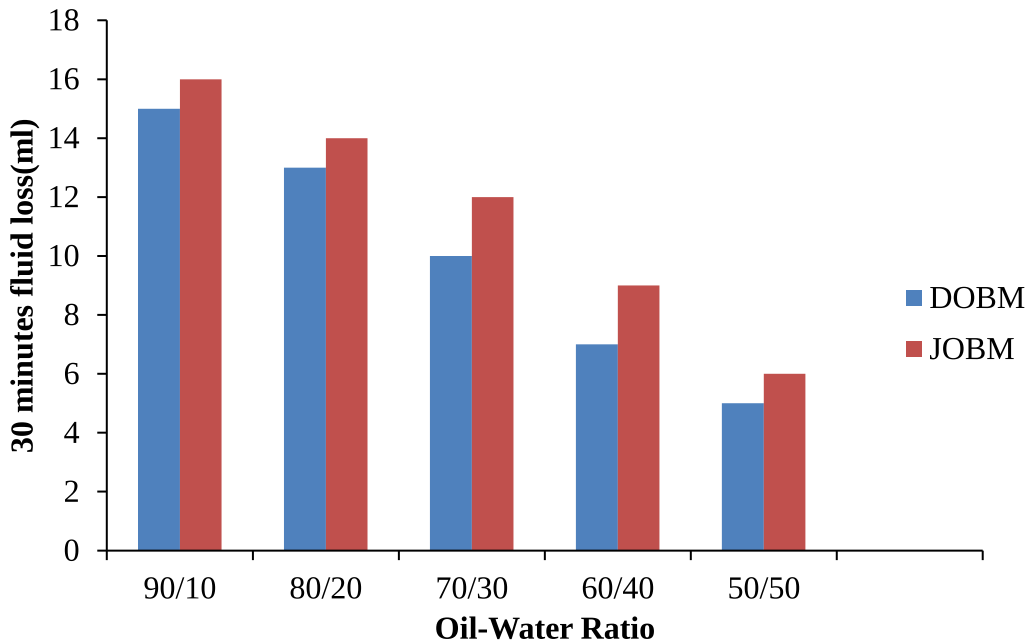

Figure 4.11: Comparative Analysis fluid loss variations of various OWR of DOBM and JOBM

The result clearly indicates that fluid loss decreases as the as the OWR decreases. As shown in Figure 4.11, 30 minutes fluid loss for OWR 90/10 was 15 ml and 16 ml for diesel oil-based mud and jatropha oil-based mud respectively. The fluid loss gradually decreased with 50/50 OWR losing 5 ml and 6 ml for diesel and jatropha oil-based muds respectively. This result confirms the findings of Aston et.al. (2002) with diesel oil-based mud giving 15.2 ml fluid loss for 90/10 OWR Mud and 5 ml for 55/45. The low filtrate exhibited by the 50/50 OWR indicates a good emulsion which is desirable for stable drilling operations. The thin filter cake of 1.7 mm which was below 2.mm as required of a mud cake controls loss of filtrate and further indicates the suitability of the 50/50 OWR for drilling operation

4.2.4.4 Electrical Stability(ES) after aging of egg for six months

To ascertain if egg yolk loses its emulsifying ability overtime, eggs were aged for 6 months at 25 ˚ C and the electrical stability test was repeated for mud samples of OWR 70/30 and 50/50. These results were then compared to the electrical stability values before aging as shown in Table 4.12. There was no significant reduction in the electrical stability values obtained after aging the eggs. This has indicated that aging appears to have no effect on the emulsifying ability of egg yolk. This is beneficial to storage.

Table 4.12: Comparative analysis of electrical stability values of 50/50 and 70/30 OWR muds before and after aging of eggs for 6 Months.

Temperature (ºC)	**ES (V) initial**	**ES (V) post aging of egg**	**ES (V) initial**	**ES (V) post aging of egg**
	50/50	50/50	70/30	70/30
48.9	353	351	480	473
120	258	255	393	388

Figure 4.12 represents the comparative analysis of the electrical stability values before and after aging of the egg for 6 months @ different temperatures.

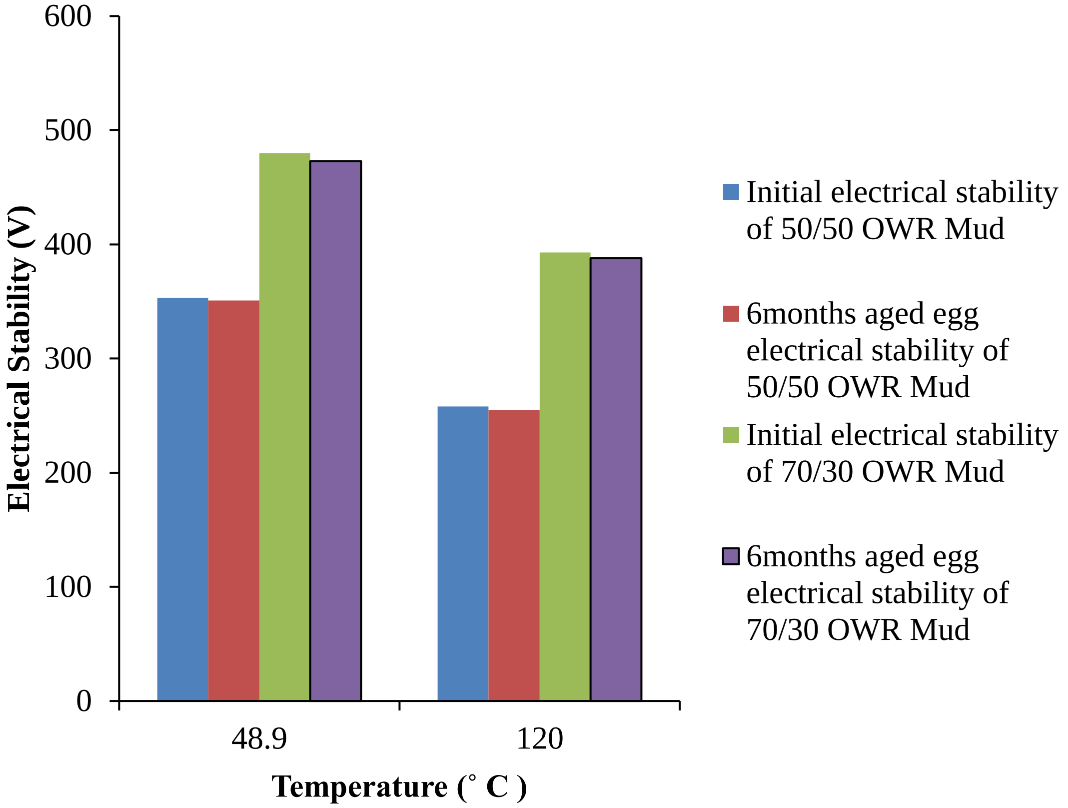

Figure 4.12: Comparative Analysis of the Electrical Stability Values before and after aging of egg for 6 months at different temperatures

There was no significant variation in the electrical stability values before and after aging of the egg. For the 50/50 OWR mud the electrical stability value at 48.9 ˚ C before aging was 353 V and 351 V after aging. At 120 ˚ C the stability value was 258 V and 255 V after aging. In the same order the electrical stability of the 70/30 OWR mud was 480V before aging and 473 V after aging while at 120 ˚ C stability value was 393 V before aging and 388 V after aging. This results have shown that the aging of egg does not affect the emulsifying ability of egg yolk. On this premise, eggs could be purchased at lower prices at seasonal offers and stored overtime. It is also economical to acquire eggs that have past sell date in the stores.

4.2.4.5 Effect of Electrical Stability on Rheological Properties - PV, YP and AV

Emulsion droplets behave as fine solid particles, which contributes to enhanced rheological properties because the drops are coated with solid particles. (Jha et.al 2014) The effect of electrical stability on rheological properties (plastic viscosity, yield point and apparent viscosity) of the muds emulsified by egg yolk and verscleanVB respectively were analysed.

Figure 4.13 illustrates the variation in plastic viscosity with emulsion stability. At low temperatures and high stability, the plastic viscosity was high. This could be attributed to the oil forming a mechanical barrier around the water droplets coating the water droplets and thereby raising the viscosity of the oil continuous phase. In the same order, at high temperature, the plastic viscosity decreased as the stability decreased as result of the breakdown of the emulsion. This is also applicable to the yield point and apparent viscosity as represented in Figures 4.14 and 4.15 respectively. There was no significant variation in the behaviour of these rheological properties with stability in the mud emulsified by egg yolk and versacleanVB. This indicates the suitability of egg yolk as a potential substitute emulsifier for vegetable oil-based invert emulsion mud.

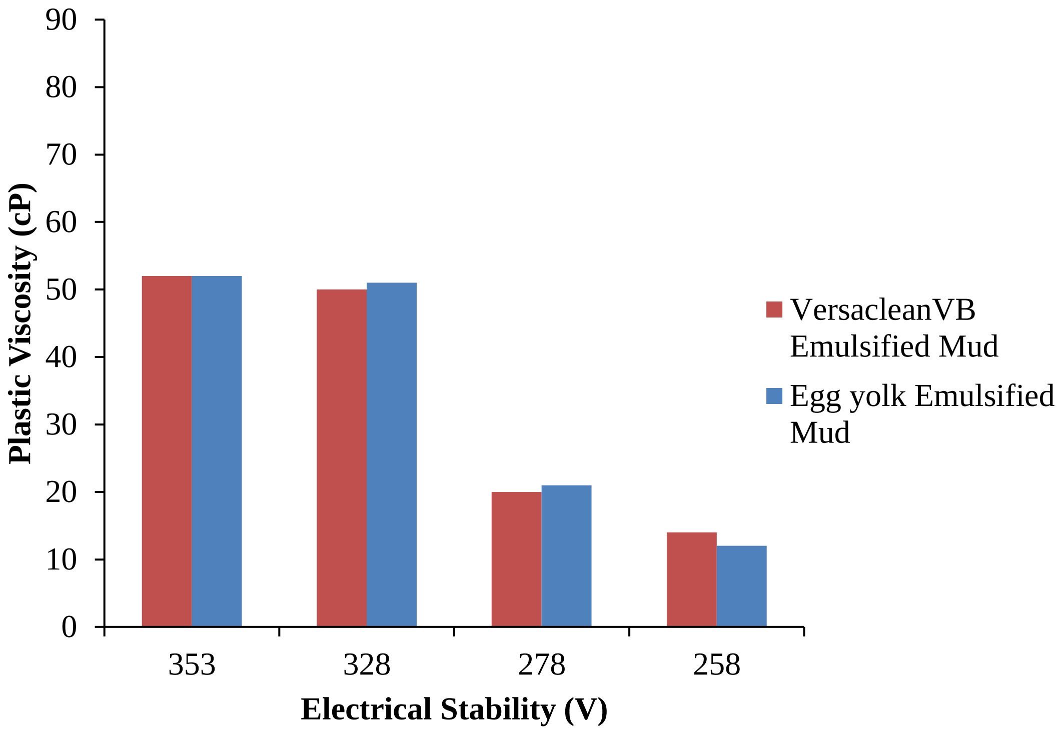

Figure 4.13: Plastic Viscosity variations with electrical stability

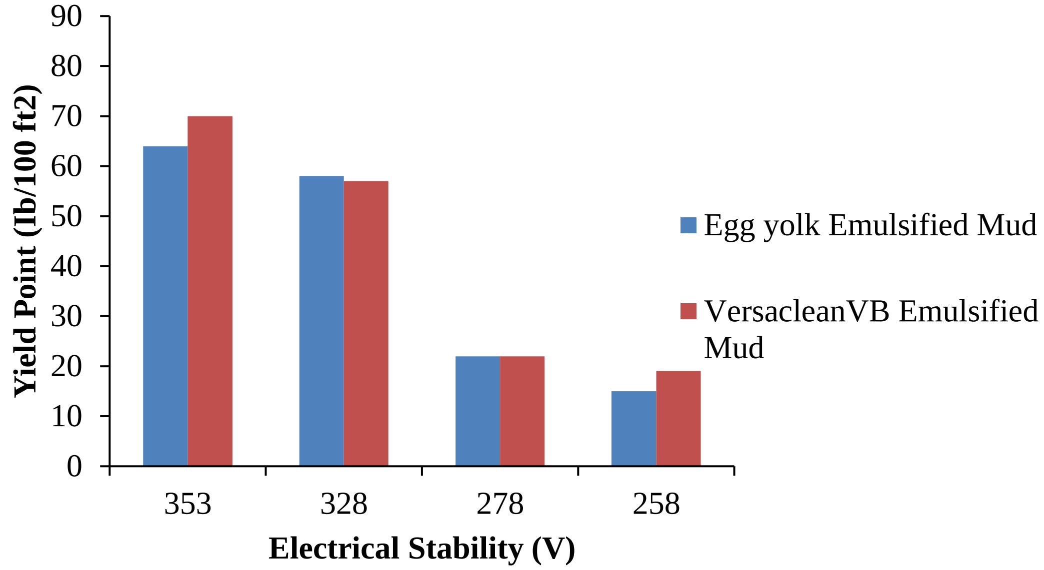

Figure 4.14: Yield Point variations with electrical stability

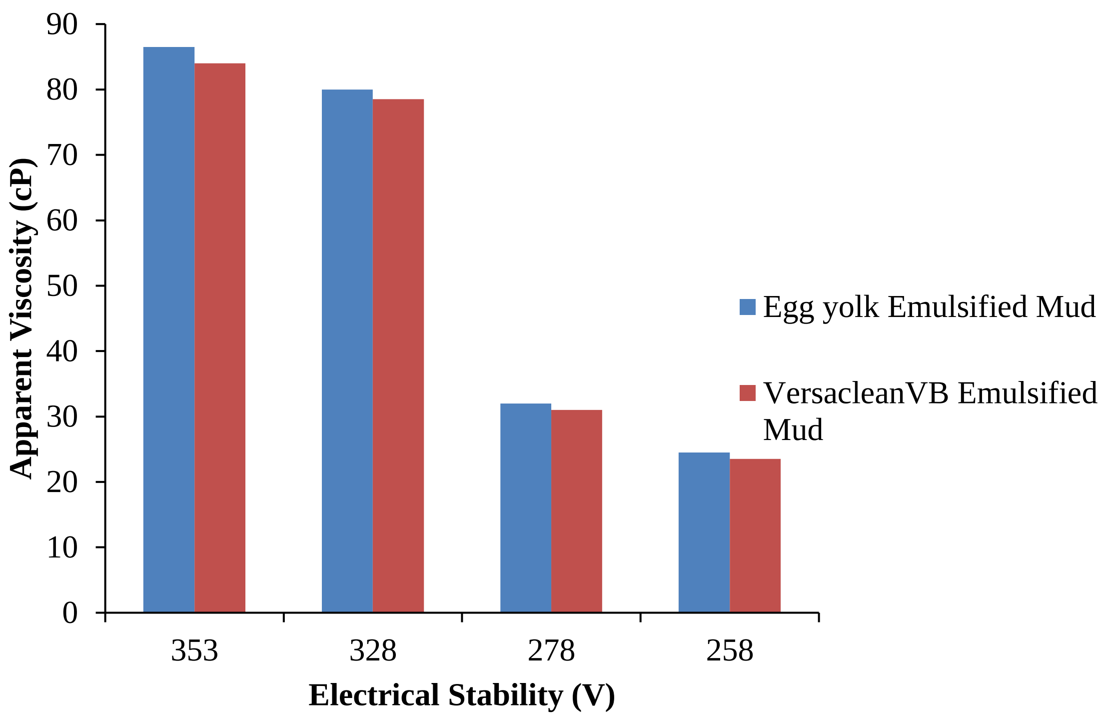

Figure 4.15: Apparent Viscosity variations with electrical stability

4.3 Phase II- Topology (Shale-Fluid Interaction)

4.3.1 Immersion test and CT imaging analysis

Shale-fluid interaction by immersion testing was investigated. The CT scanner imaging analysis was used for the acquisition and analysis of the shale sample. The VG studio max 2.2 was used for the analysis of the acquired images. The shale sample used was Marcellus shale. Six test shale samples were immersed into three mud samples- water-based mud, diesel and jatropha oil-based muds and observed for one and seven days.

4.3.2 Comparative analysis of one and seven days immersion test

Interactions between shale and drilling mud and their consequences on wellbore stability were evaluated. This was undertaken over one and seven days period of immersion in water based mud, 50/50 oil-water ratio diesel and jatropha oil- based muds to ascertain the level of interaction with mud.

Due to heterogeneous nature of shale, it behaves in various ways on encounter with fluids and other substances. However, the focus of this study is on the level of shale interaction with the mud. The original shale sample as was shown in Figure 3.18 has no fractures, therefore any resulting fractures was caused by the interaction with the mud. Figure 4.16 represents a CT image from the one-day immersion test in water-based mud. The different interactions with the core sample on immersion has been represented with colour marks. The thickness of the concentration of the colour marks indicates the extent of the interaction with the mud which is graphically shown in Figure 4.17

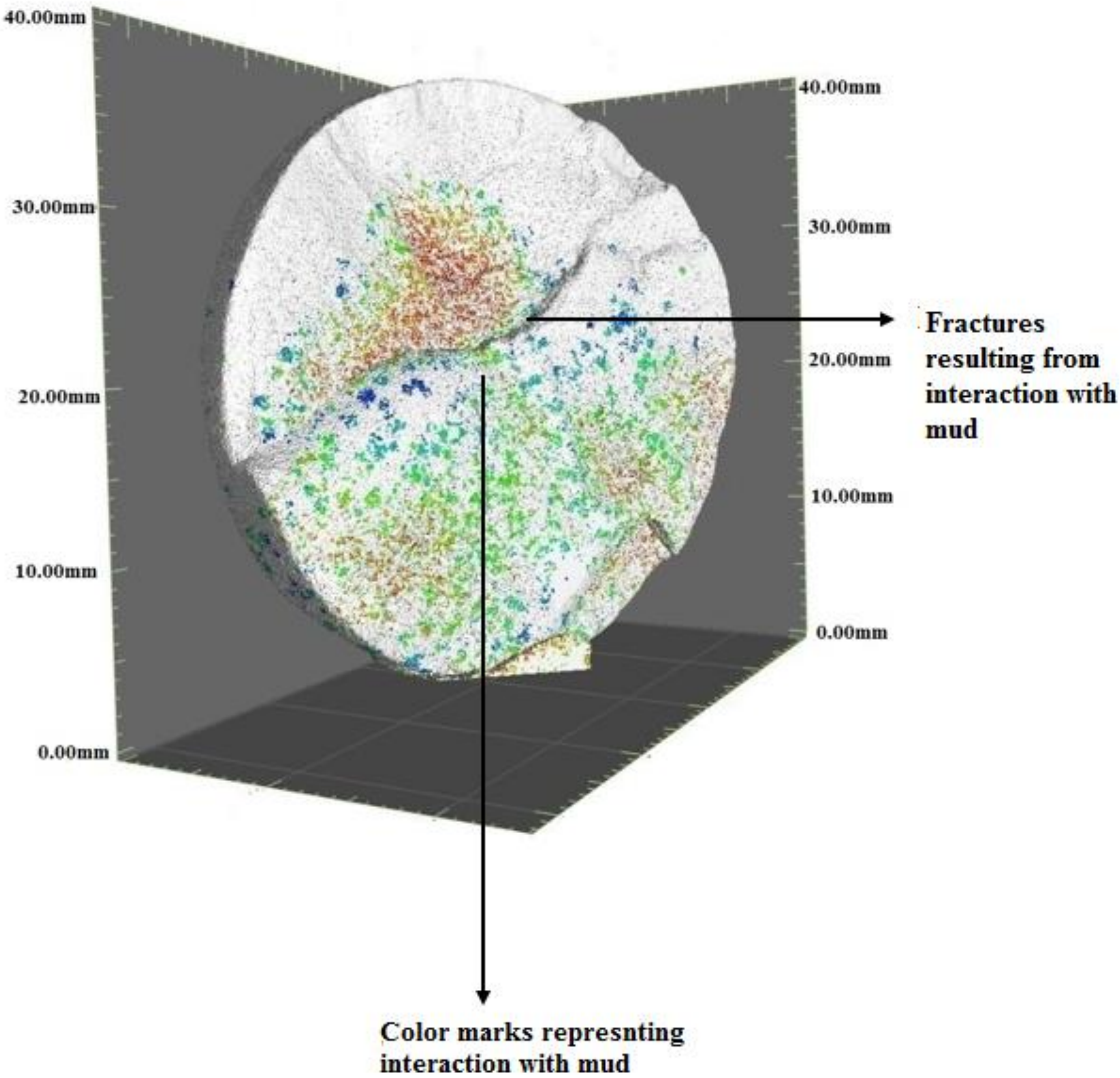

Figure 4.16: One-day Immersion in Water- Based Mud

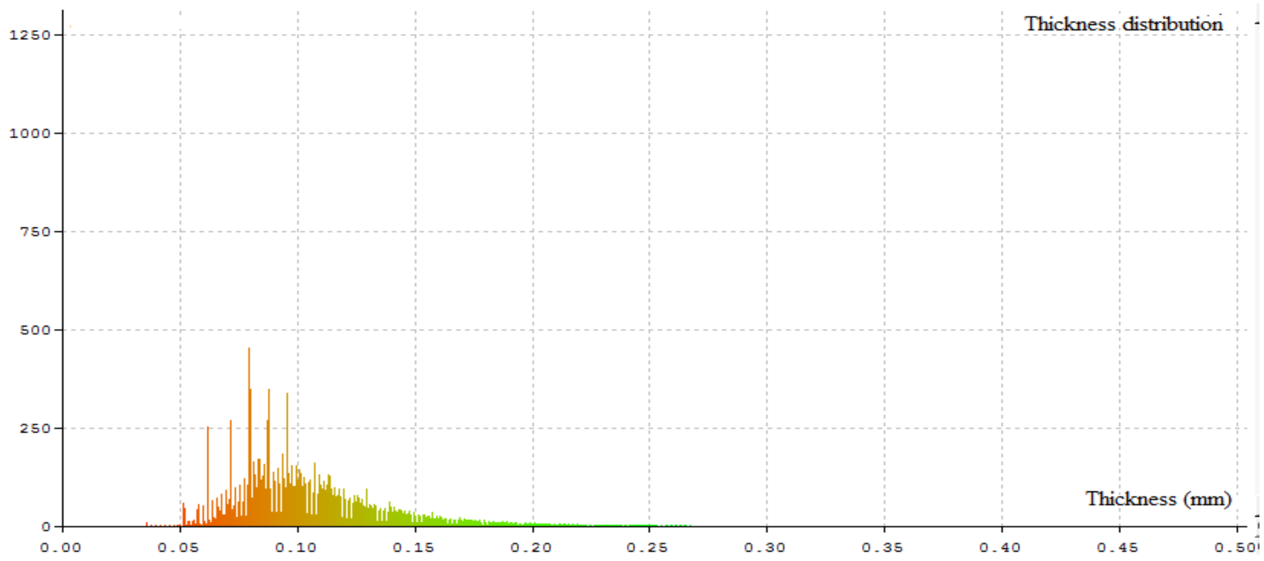

Figure 4.17: Thickness distribution of the interaction (one -day immersion) of water based mud

The volume analysis after one-day immersion was 3872.35mm^3 compared to the initial volume of 3502.21 mm^3 indicating an increase in volume by 370.14mm^3 as shown in the defect volume distribution in Figure 4.18

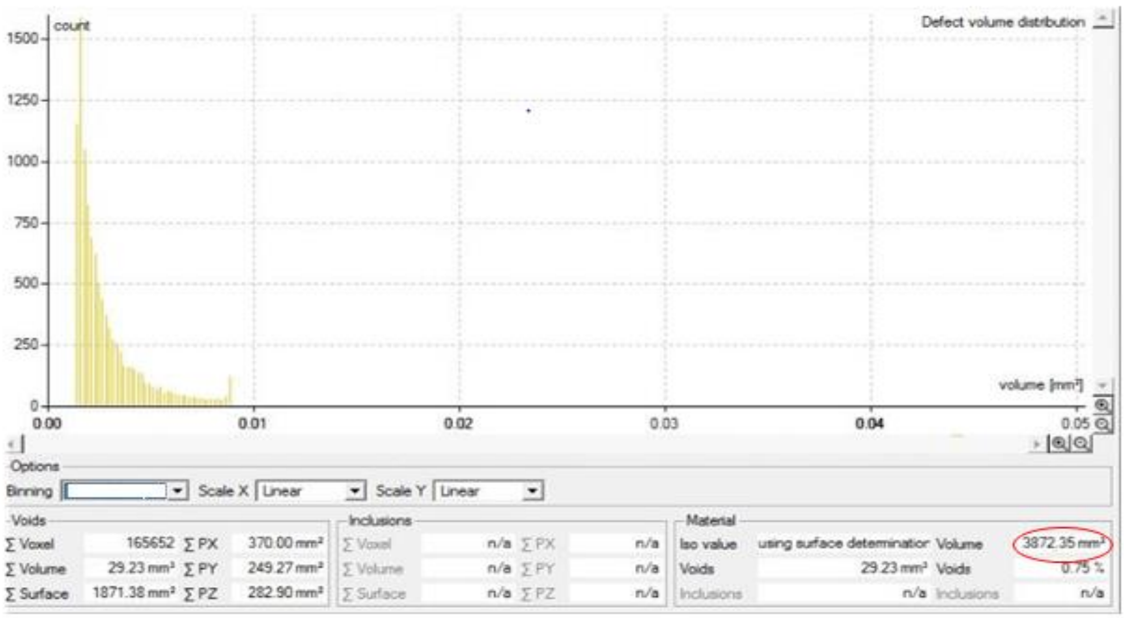

Figure 4.18: Defect volume distribution of core sample in one-day immersion in water- based mud

Figure 4.19 represents the seven-day immersion showing significant interaction with the mud as represented with the concentration colour marks.

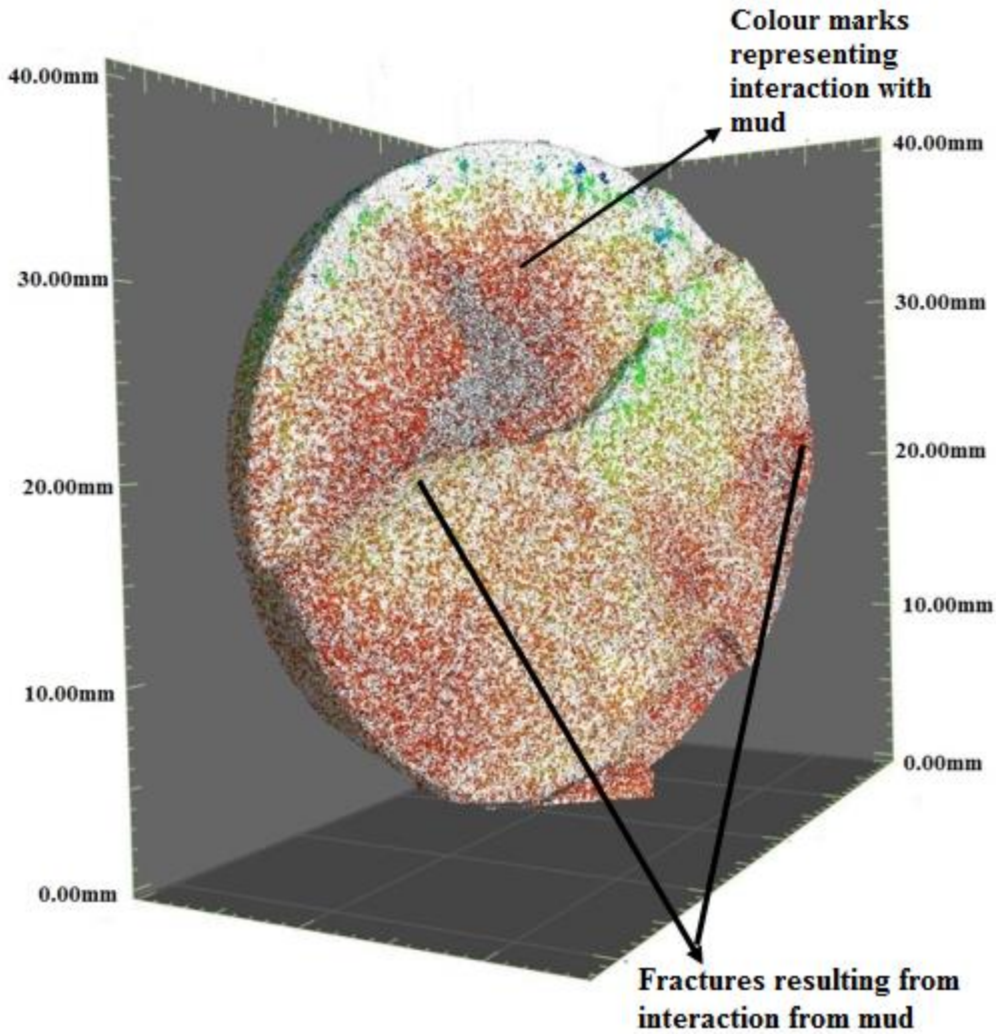

Figure 4.19: Seven-days immersion in water- based mud

Figure 4.20 shows the thickness distribution of interaction after seven days immersion in water-based mud.

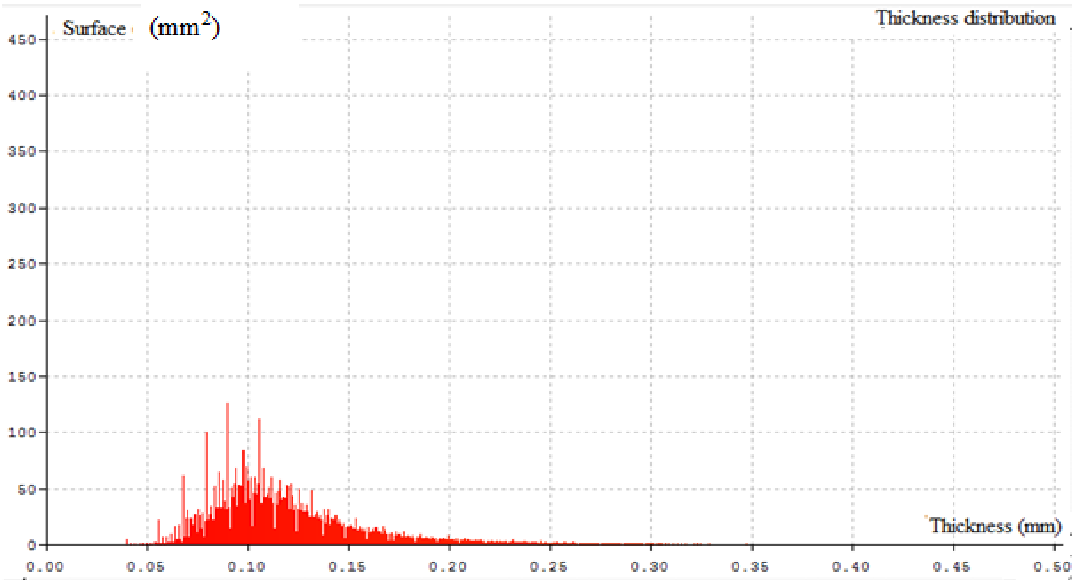

Figure 4.20: Thickness distribution of the interaction (seven days immersion)

The defect volume distribution was 4306.19mm^3 as shown in Figure 4.21. This is high compared to the volume of the test core sample of 3502.21 mm^3 giving significant increase by 803.98mm^3

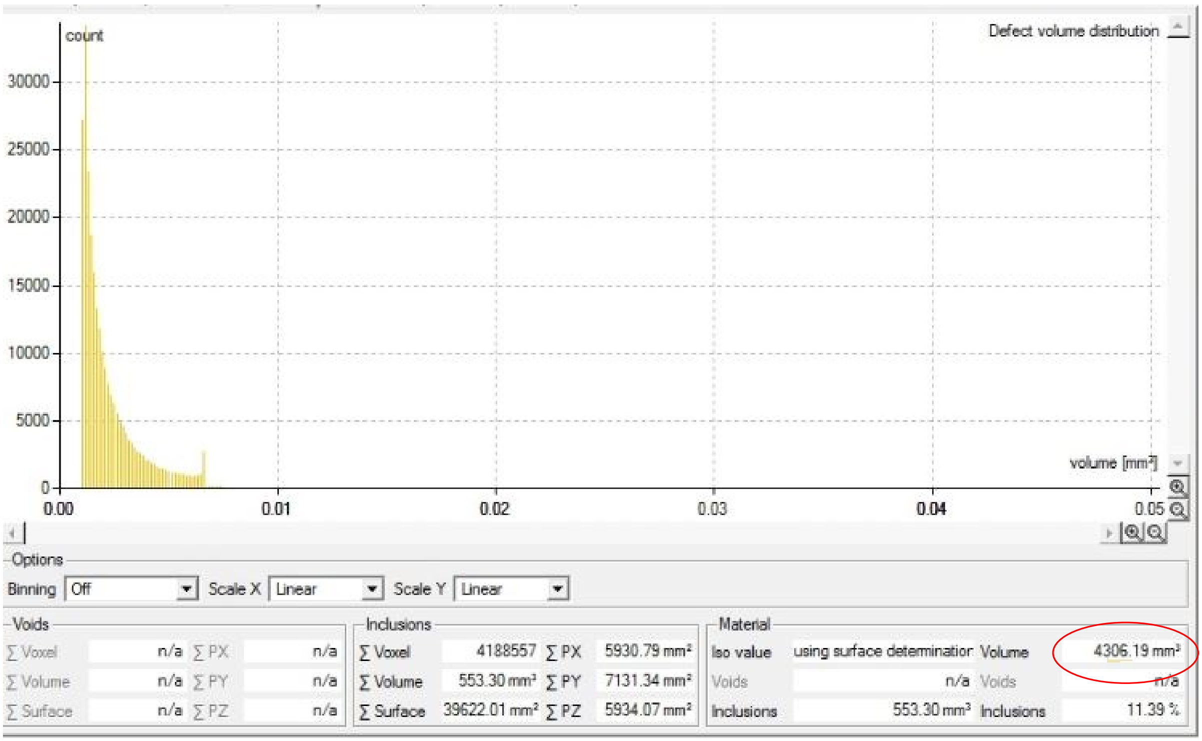

Figure 4.21: Defect volume distribution of core sample in seven-day immersion in water-based mud

Comparatively, the one to seven-day immersion in diesel and Jatropha oil-based muds exhibited no enlargement or development of new fractures in the core sample as shown in Figure 4.22. The insignificant level of interaction with fluid could not reflect on a thickness distribution chart.

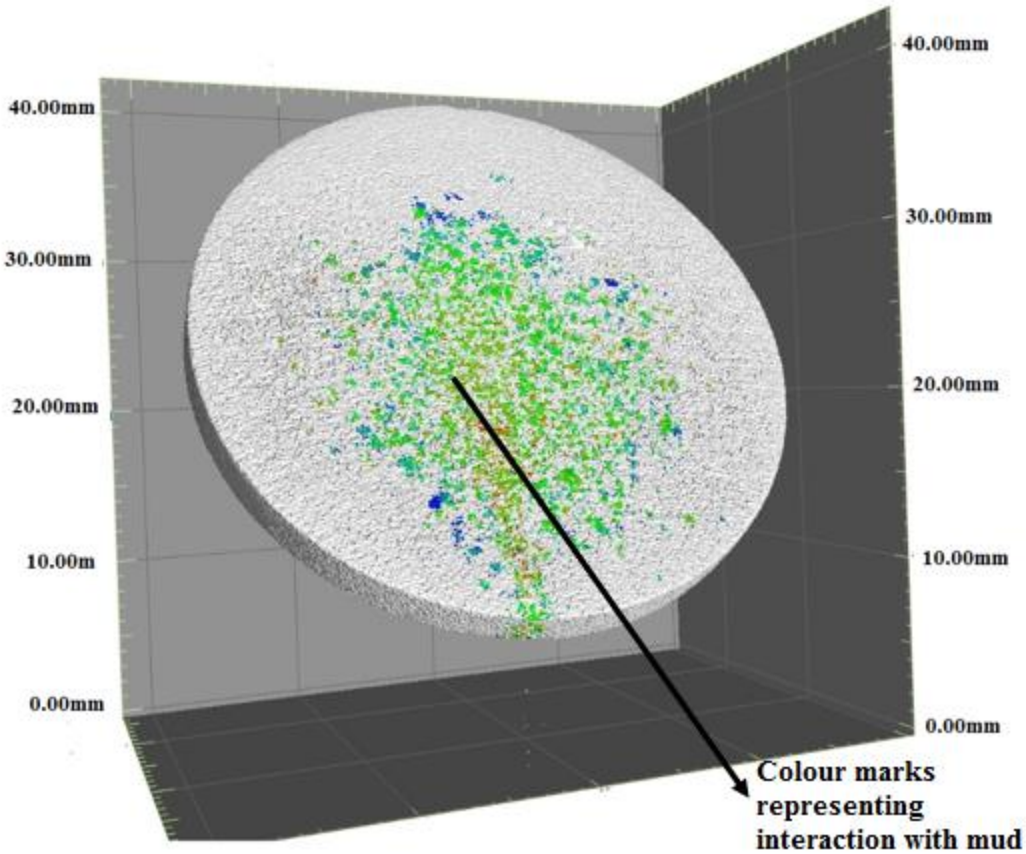

Figure 4.22: One day to seven -days immersion in diesel and jatropha oil-based muds.

The level of interaction with the diesel and jatropha muds was low with a defect volume distribution of 3533.16mm^3 and 3590.75mm^3 at the end of one-day and seven days immersion respectively compared to the initial volume of 3502.21mm^3 before the immersion. This is represented in the defect volume distribution Figure 4.23

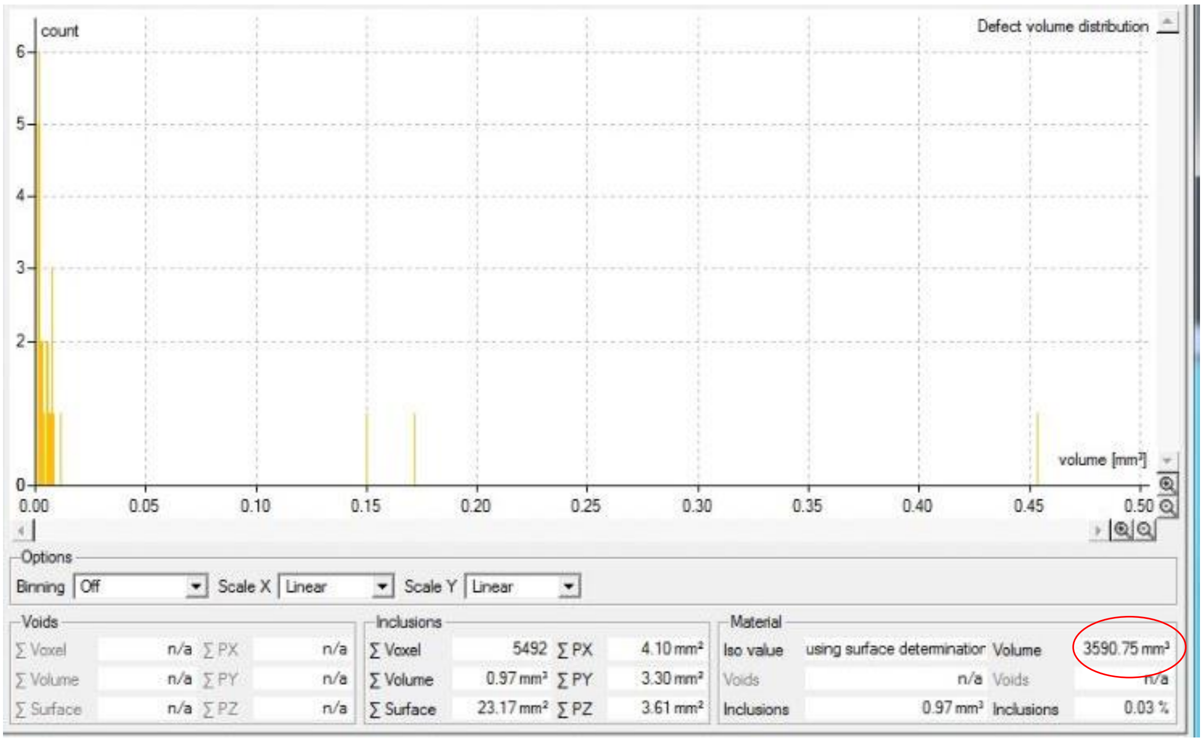

Figure 4.23: Defect volume distribution of one day to seven -days immersion in diesel and jatropha oil-based muds.

Table 4.13 represent the percentage variation in the defect volume distribution. The volume increase was 0.88% (30.95mm^3) and 2.53% (88.54mm^3) compared to 11% (370.14mm^3) and 23% (803.98mm^3) obtained in the water-based mud. This is graphically shown in Figure 4.24. There was no variation in appearance of shale exposed to diesel and jatropha oil based muds. This indicates that Jatropha oil-based mud could be categorised as a relatively environmentally friendly substitute to diesel mud for drilling shale formations while maintaining wellbore stability.

Table 4.13: Percentage variation in volume distribution of test core samples

Mud Samples	**Test Core Sample (mm^3)**	**1-Day Immersion (mm^3)**	**Percentage change in Volume (%)**	**7-Day Immersion (mm^3)**	**Percentage change in Volume (%)**
Water-Based Mud	3502.21	3872.35	11.0	4306.19	23.0
Jatropha Oil-Based Mud	3502.21	3533.16	0.88	3590.75	2.53
Diesel Oil-Based Mud	3502.21	3533.16	0.88	3590.75	2.53

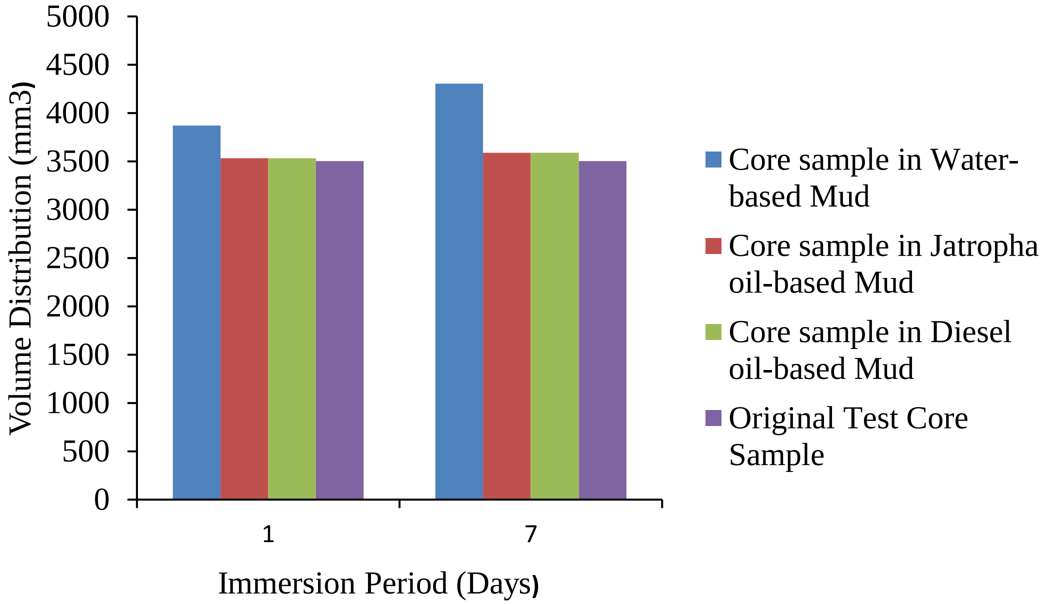

Figure 4.24: Comparison of volume distribution of the core sample in the mud samples

4.4 Economic consideration of vegetable oil-based invert emulsion

4.4.1 Introduction

The choice of any drilling fluid for a specific well depends on three key factors - cost, technical competence and environmental compatibility. Economic consideration of drilling fluid is required before any drilling operation. Sometimes drilling fluid may be technically viable and environmentally compatible but may not be used for drilling operation if it is not affordable at an economic cost. This economic consideration of vegetable oil-based invert emulsion consists of comparing both the cost of formulation and the cost of disposal after the drilling operation, to include all the benefits which may not be necessarily monetary such as social and environmental benefits. In this comparison, as many factors as possible are measured in the same cash units. The cash unit in this evaluation is the US dollar ($), since most of the international businesses in the petroleum industry are quoted in US dollars

This chapter considers the economic viability of invert emulsion using jatropha oil in 50/50 oil-water ratio with egg yolk as a non-toxic emulsifier. This was compared with the conventional diesel oil-based mud using a standard emulsifier –*VersacleanVB.* The comparison was based on the cost of formulation and disposal of one barrel of the mud. In this evaluation, the economic parameters considered are the base oil and the emulsifier which are substituted in the mud formulation.

4.4.2 Profitability analysis of vegetable oil-based invert emulsion

In order to evaluate the economic viability of the 50/50 invert emulsion, the physico-chemical properties of diesel and jatropha oils were evaluated, as they help in early estimation of the mud composition and behaviour. These properties include the following: *specific gravity flash point fire point pour point, kinematic viscosity,* a*niline point and cloud point*. According to Amanullah (2005), vegetable oil-based mud will have several advantages over the mineral oil-based mud due to the higher flash point, fire point, and high biodegradability. Higher flash and fire points indicate better fire resistant capacity and minimum chances of causing operational problems associated with low flash and low fire points. The high flash and fire point also ensure enhanced safety in handling, storage and transportation. The aniline point is the lowest temperature at which equal volumes of aniline ($C_6H_5NH_2$) and the oil will form a single phase. It determines if the base oil will damage components of the drilling assembly. Table 4.14

compares the properties of jatropha and diesel oils as base oils in oil-based mud. The properties were ranked in the order of compatibility. The pour point, flash point and kinematic viscosity of diesel oil were compatible whilst the fire point was not compatible. Comparatively, the pour point, flash point and fire point of jatropha oil were compatible but the kinematic viscosity was not compatible.

Table 4.14: Comparison of properties of datropha and diesel oils as base oils used in oil-based muds

Property	Aniline Point (˚ C)	Pour Point (˚ C)	Flash Point (˚ C)	Fire Point (˚ C)	Kinematic Viscosity @40^{O}C, cST	Aromatic Content (%)
Required Properties of Base Oil	> 65	<Ambient temperature	> 66	> 80	2.3 -3.5	4-8
Diesel Oil	< 49	- 6	65	78	2.86	
Jatropha Oil	64-65	6	214	256	36.92	

Key: **Green**- Compatible**, Red-** Not compatible, **White** –Not determined

Furthermore, diesel and jatropha oils were evaluated in relative to some objectives such as availability, cost effectiveness, environmental compatibility, rheological and filtration properties and ranked in low (poor), medium (fair), and high (good) as shown in Table 4.15

Table 4.15: Evaluation matrix of diesel and jatropha oils

Base Oil	**Rheological Properties**	**Filtration Properties**	**Cost Effectiveness**	**Availability**	**Environmental Compatibility**
Diesel	High	High	Low	High	Low
Jatropha	Medium	High	High	High	High

4.4.3 Cost comparison for formulation of 50/50 OWR diesel and jatropha oil- based muds

The cost of formulation of one barrel of 50/50 OWR invert emulsion mud was formulated using diesel and jatropha oils respectively and then compared. The comparison was based on the cost of the base oil and the emulsifier, which are the two items substituted in the formulation. Not all the benefits of vegetable oil based mud are measurable in financial terms considering all the environmental benefits. It is also difficult to find accurate comparative data for total cost of formulation of diesel oil-based and vegetable oil-based muds.With the available data, comparison of the formulation cost of one barrel of diesel oil-based mud and vegetable oil-based mud is shown in Table 4.16. The unit price of an egg from a local store containing 15 ml (0.000094 bbl) of egg yolk was $0.11, therefore the price of 1 bbl was $1170. The price of diesel was US$ 3.11 per US gallon (US$ 130.62 / bbl). As at the time of this study, the price of jatropha oil from the supplier (Fairy Group Export on 01/03/2016) was US$ 400 per metric ton (US$ 54.8 / bbl)

Table 4.16: Cost comparison for formulation of 1bbl of 50/50 OWR diesel and jatropha oil based muds

Cost Parameter	Diesel Oil-Based Mud	Jatropha Oil-Based Mud
Cost of base oil (US$/bbl)	130.62	54.8
Required volume of base oil (bbl)	0.50	0.50
Cost of base oil per required volume (US$/bbl)	65.31	27.4
Cost of Mud Additive - emulsifier (US$/bbl)	1.5	1170
Required volume of Mud Additive(emulsifier) (bbl)	0.000075	0.000075
Cost of Mud Additive per required volume (US$/bbl)	18	0.09
Total cost of formulation of 1barrel (US$/bbl)	**65.31**	**27.49**

4.4.4 Cost comparison of management and disposal of diesel oil-based and vegetable oil-based muds

As was mentioned in Section 2.7, the choice of disposal method of used mud and drilled cuttings largely depends on the type of generated cuttings, cost of treatment and disposal. Oil-based mud cuttings from diesel and mineral oils pose a complex and costly waste management challenge and cannot be discharged on-site. Waste streams high in hydrocarbons ranging from 10-40 % like oil-based mud are candidates for thermal treatment technology. Cost of thermal treatment of oily waste ranges from $75 to $150 per ton with labour being a large component. (Bansel and Sugiarto, 1999) Diesel oil-based muds pose greater risk than vegetable oil based mud through skin irritation and effects of inhalation amongst other hazards.The added transport and disposal costs as well as potential liability issues associated with diesel oil-based mud have restricted the widespread usage making vegetable oil-based mud the preferred alternative.

In 1997 disposal costs reported by offsite commercial disposal facilities for oil-based drilling wastes ranged from $0 to $57/bbl and for water based drilling waste ranged from $0.20 to $14.70. Most operators charge transportation cost by hour typically $55.00/hr to $175.00/hr. Others use a per-load or per container basis for instance in one case $1.00/bbl to $3.00bbl (Puder and Veil 2006). Not all the benefits of management and disposal of vegetable oil based mud are measurable in financial terms considering all the environmental benefits. It is also difficult to find accurate comparative data for total drilling waste management costs for diesel oil-based and vegetable oil-based muds. With the available data, comparison of the disposal cost of one barrel of diesel oil-based mud and vegetable oil-based mud is shown in Table 4.17. With the disposal cost of $57/bbl for oil-based wastes and $14.70 for water based wastes, a disposal cost of $28.5/bbl has been assumed for vegetable oil-based mud wastes.

Table 4.17: Cost comparison of management and disposal of diesel oil-based and vegetable oil-based muds

Cost Parameter	**Diesel Oil-Based Mud**	**Jatropha Oil-Based Mud**
Cost of transportation of drilling wastes to disposal site (US$/bbl)	3.0	3.0
Cost of Commercial disposal of drilling wastes (US$/bbl)	57.0	28.50
Total cost of disposal of 1barrel (US$/bbl)	**60.0**	**31.50**

4.4.5 Cost evaluation analysis

Comparison of the cost of formulation of one barrel of invert emulsion mud based on base oil and emulsifier showed that Jatropha oil-based mud at the cost of $27.49/bbl is cheaper than diesel oil-based mud at $65.31/bbl as illustrated in Figure 4.25. In the same order, the cost of disposal of drilling wastes of jatropha oil-based mud at $31.50 is lower than that of diesel oil-based mud at $60.00, as shown in Figure 4.26, suggesting a potential saving of 57.91% of the $65.31 cost of formulation and 47.5 % of $60 of the cost of disposal of the conventional diesel oil-based mud. This has the potential to equate to saving of $37.82 per barrel of invert emulsion formulated and $28.50 per barrel disposed. Based on these costs of formulation and disposal, jatropha oil-based invert emulsion is more economical than diesel oil-based mud as shown in Figure 4.27.

There is no standard cost for drilling mud in a well. The cost of the mud usually depends on the depth and the complexity of the well. However, in oilfield the rule of thumb is the allocation of 5% of the drilling budget to drilling fluids (AIG 2015). The cost can rise up to 15% in difficult drilling situations and formations. Allowances are usually given for losses to the formation, solid control equipment like desanders, desilter and shale shakers. In this case $37.82 is saved per barrel formulated for any operation and $28.50 per barrel disposed. The cuttings generated using diesel oil-based muds need special treatment before discharging them to prevent contamination of water with free oil. The use of more water in the formulation of 50/50 OWR invert emulsion mud means reduced cost of formulation. While drilling through shale, the water activity of the invert emulsion fluid is maintained at a lower level than the water activity of the shale creating an osmotic pressure that drives the flow of water from the shale to the invert emulsion fluid thereby preventing shale hydration and less fluid loss which are essential for wellbore stability. High wellbore stability will ensure maximum drilling performance thereby reducing non-productive down-time and economic losses. Based on these comparisons, not only being technically and environmentally compatible vegetable oil-based mud is more economically viable than diesel oil-based mud.

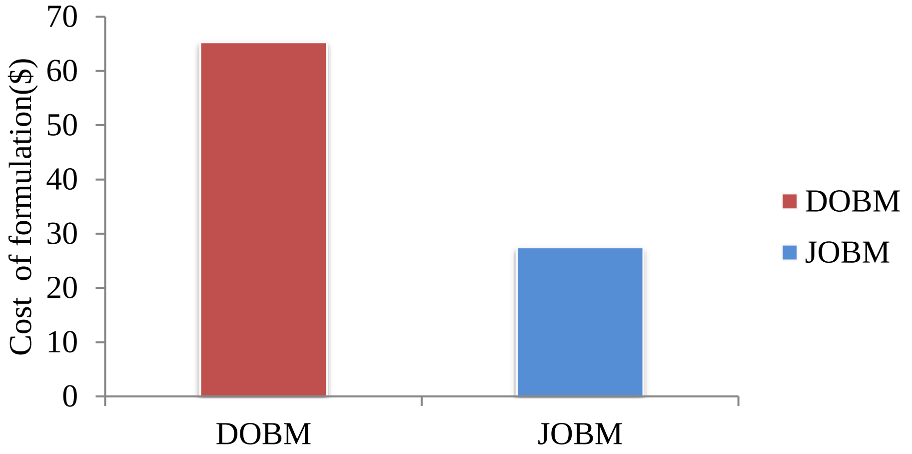

Figure 4.25: Cost comparison for formulation of 1bbl of 50/50 OWR diesel and jatropha oil based muds

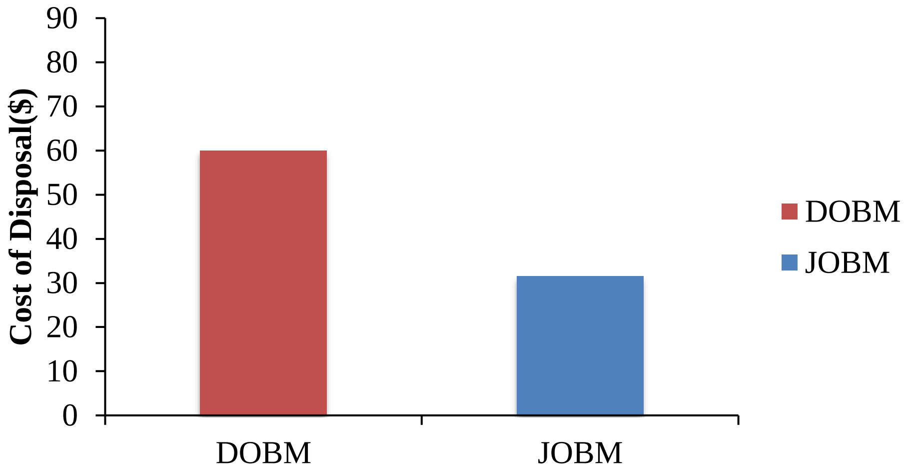

Figure 4.26: Cost comparison of management and disposal of diesel oil-based and jatropha oil-based muds

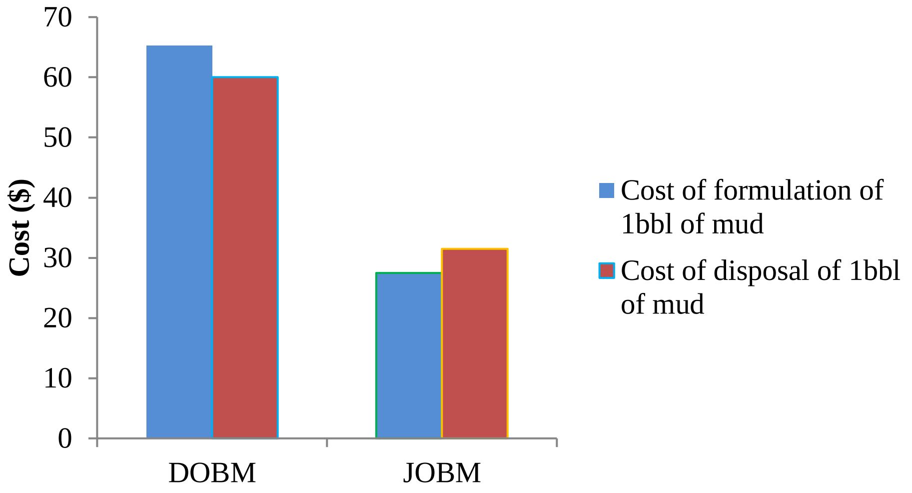

Figure 4.27: Total **c**ost comparison of formulation and disposal of diesel oil-based and jatropha oil-based muds

4.5 Summary

The preceding chapter is summarised as follows:

Phase -I: Mud formulation and testing

- The emulsifying ability of egg yolk was determined and higher stability was achieved with the egg yolk compared to a conventional emulsifier *VersacleanVB*, which supports the proposal for the replacement of toxic additives with non-toxic and organic substitutes and also for compatibility with vegetable oil continuous phase as proposed by Apaleke et al., (2012a)
- A low OWR of 50/50 with stable emulsion, required rheological and filtration properties were achieved. The mud gave a low fluid loss which is suitable for wellbore stability (Aston et al. 2002) The mud was formulated without a fluid loss additive which solved the issue of incompatible additives as noted by Amanullah, (2005), that Duratone used as fluid-loss control additive in mineral oil based muds is not suitable for vegetable oil-based mud.
- The high water content of the mud has the potential of reducing material and disposal costs because a lower quantity of oil will be retained on the drilled cuttings. This was demonstrated in the economic consideration.
- The economic consideration of the mud showed a potential saving of 57.91% of the $65.31 of the cost of formulation and 47.5% of $60 of the cost of disposal of the

conventional diesel oil-based mud. This has the potential to equate to saving of $37.82 per barrel of invert emulsion formulated and $28.50 per barrel disposed based only on the base oil and emulsifier components.

Phase- II: Topology (Shale-fluid interaction)

- Shale-fluid interaction by immersion testing and imaging using the CT scanner revealed significan shale core fracture development after seven days exposure to the water based mud, thus indicating high interaction with the shale sample, which is not good for wellbore stability.
- There was no enlargement or development of new fractures in diesel and jatropha oil based muds. Comparatively, there was no variation in the appearance of the shale exposed to diesel oil based mud and jatropha oil based mud.
- Considering the economic viability of Jatropha oil over diesel oil, this further suggests that Jatropha oil can be used as a diesel alternative for a relatively environmentally friendly invert emulsion, suitable for drilling shale formations while maintaining wellbore stability

Chapter 5

Conclusions and Recommendations

5.1 Conclusions

The aim of this experimental study was to achieve a low oil-water ratio of 50/50 using Jatropha vegetable oil continuous phase capable of reducing fluid loss for increased wellbore stability especially in unconventional shale reservoirs. At the same time, substituting a conventional emulsifier with egg yolk and to work towards the reduction of cost of drilling operations by reducing the cost of formulation and disposal of the conventional diesel oil-based mud. Shale-fluid interaction by immersion test was also studied as a way of ascertaining the impact of drilling fluid on shale which is a major cause of shale and wellbore instability.

A detailed review of drilling fluid, properties and additives, drilling and fracturing in unconventional shale reservoirs was discussed. The trend of vegetable oil-based muds and the challenges including the achievement of a low oil-water ratio and replacement of toxic additives with non-toxic ones was discussed in Chapter 2. The theoretical background of shale –fluid interaction by immersion test using X-ray computerised tomography scanner was also highlighted. A detailed description of the experimental apparatus, materials and procedure used during this experimental study were presented in Chapter 3

The results of the various concluded experiments were presented, interpreted and analysed Chapter 4. The economic consideration of the low oil-water invert emulsion using vegetable oil was also examined to determine the profitability over the conventional diesel oil-based mud.

Based on the findings from the present study, the potential of a low oil-water ratio of 50/50 invert emulsion with Jatropha vegetable oil continuous phase to reduce fluid loss. which is beneficial to wellbore stability, is considerable. Furthermore, the emulsifying ability of egg yolk in vegetable oil-based muds is also considerable. All the work highlighted above led to the conclusions that are listed according to the two phases below:

Phase-1

- The properties and availability of diesel and jatropha oils were successfully evaluated. Jatropha demonstrated comparatively good filtration properties, high flash and fire point to diesel oil. On availability, cost effectiveness and environmental compatibility, diesel oil has high availability, but low cost effectiveness and environmental compatibility while jatropha oil is highly available, cost effective and environmentally compatible. By this evaluation Jatropha oil has demonstrated technically, economically and environmentally viable as a base oil substitute in oil-based muds for difficult formations such as the shale.

- The emulsifying ability of egg yolk was successfully examined. The emulsion stability of the jatropha oil based mud emulsified with egg yolk was higher than when it was emulsified with the standard emulsifier Versaclean VB. Comparatively there was no significant variation in behaviour of the diesel mud emulsified with VersacleanVB and the Jatropha oil based mud emulsified with egg yolk indicating that the potential to substitute egg yolk as an emulsifier in vegetable oil invert emulsion muds is considerable.
- A 50/50 OWR invert emulsion was achieved in the present study and the properties compared favourably with that of 70/30 OWR mud, giving a lower filtrate volume which is conducive for wellbore stability**.** The mud exhibited a low-flat gel profile, which is desirable indicating that the muds will remain pump-able if left static in the borehole for some time. The 50/50 OWR mud is also more economical in terms of mud formulation and disposal. This because it is cheaper to use more water than oil in mud formulation, and the high water content will mean less oil retained on the drilled cuttings, making disposal easier and cheaper as it will require little or no treatment before the disposal

Phase-2

- Increased instability in shale resulting from incompatibility of drilling fluids is the main drive for compatible fluids for drilling shale reservoirs. One and seven- days immersion testing of shale core samples in a water-based mud showed swelling and facture development, while there was no enlargement or development of new fractures when immersed in diesel or jatropha oil based muds. This is an indication of little or no interaction with the mud, which is also conducive for wellbore stability.

- Stringent environmental regulations on the disposal and management of diesel oil-based mud has been the main drive for the replacement with vegetable oil-based muds. The economic consideration of 50/50 oil-water ratio invert emulsion equally showed a potential saving of 57.91% of the $65.31 of the cost of formulation and 47.5% of $60 of the cost of disposal of the conventional diesel oil-based mud. This has the potential to equate to saving of $37.82 per barrel of invert emulsion formulated and $28.50 per barrel disposed.

From the above conclusions in meeting with the aims and objectives of the present study, the three important factors in choice of drilling fluid which are technical competence, cost and environmentally compatibility were also recognised.

5.2 Recommendations for future works

Further investigations may include

- Invert emulsions have the ability to reduce filtrate loss to the formation due to the ability of emulsion droplets to provide thin filter cake during drilling as demonstrated in this novel 50/50 OWR invert emulsion mud. Invert emulsion mud has been characterised by several mud properties, but an area for future work is to study the characterisation of invert emulsion by the emulsion droplet size. This is to ascertain the effect of emulsion droplet size on fluid loss. Study area will include the effect of emulsion droplet size on fluid loss on various oil-water ratio invert emulsion muds.
- One of the major advantages of invert emulsion is high rate of penetration, with a vegetable oil continuous phase associated with relatively high viscosity which could affect the rate of penetration. Study area could include comparison of penetration rates using different nozzle diameter.
- On Shale fluid interaction, investigation should include the effect of fluid chemistry and exposure time on shale strength.

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Appendices

Appendix A

Publications and Conferences

1 **Ihenacho, P.C**., Burby,M., Nasr, G.G and Enyi,G.C., 2016 Utilisation of Egg Yolk as a Non-Toxic Emulsifier in Environmentally Friendly Invert Emulsion Drilling Mud. *Journal of Petroleum Engineering & Technology 2016:6 (1): 41-49p*

2 **Ihenacho, P.C.,** Burby, M., Nasr, G.G and Enyi,G.C. 2016 50/50 Oil-Water Ratio Invert Emulsion Drilling Mud Using Vegetable Oil as Continuous Phase. *International Journal of Chemical, Molecular, Nuclear and Metallurgical Engineering. Vol.10, No3, 2016*

3 **Ihenacho, P.C.,** Burby, M., Nasr, G.G and Enyi,G.C. 2016 50/50 Oil-Water Ratio Invert Emulsion Drilling Mud Using Vegetable Oil as Continuous Phase. *18th International Conference on Oil, Gas and Petrochemical Engineering. Rome, Italy March 21-22,2016*

4 **Ihenacho, P.C.,** Burby,M., Nasr, G.G. and Enyi,G.C. (2017) Economic Evaluation of Environmentally Friendly Vegetable Oil-Based Invert Emulsion Drilling Mud**.** *Journal of Engineering Technology. Journal of Engineering Technology (ISSN:0747-9964) Vol 6, issue 2, July,2017, pp776-783*

5 **Ihenacho, P.C**., Burby,M. and Nasr,G.G Shale-Fluid Interaction in Environmentally Friendly Invert Emulsion Using an X-Ray Computed Tomography (CT) Scan. *Petroleum Science PETROSCI-2016-0355* (Under Review)

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Utilization of Egg Yolk as A Non-Toxic Emulsifier in Environmentally Friendly Invert Emulsion Drilling Mud

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Abstract

Vegetable oil such as Jatropha has been shown to be a comparable alternative to diesel and mineral oils for environmentally friendly oil based drilling mud. However compatibility of the vegetable oils with the usual chemical additives is still a major challenge that calls for compatible and non-toxic substitutes. This study examined the suitability of hen egg yolk as a non-toxic emulsifier in invert emulsion drilling mud using vegetable oil continuous phase. 8 ml of the egg yolk which was separated from the albumen was added dropwise as a primary emulsifier to a 70/30 oil-water ratio invert emulsion drilling mud formulated with Jatropha vegetable oil. The electrical stability was tested using electrical stability tester at the recommended test temperature of 48.9°C and later at 120°C to check any variation at elevated temperature. The stability values in volts were 398 and 289 V respectively. A similar formulation using a standard emulsifier (versacleanVB) an amidoamine gave stability values as 201 and 188 V respectively indicating that higher stability was attained with the egg yolk. The viscosities of the mud samples were also tested giving plastic viscosities of 52 and 49 cP at 50°C for the egg yolk and verscleanVB emulsified mud respectively. This study indicates that egg yolk could serve as an emulsifier for vegetable oil based invert emulsion mud in line with non-toxic additives for environmental compatibility.

Keywords: *Environmental compatibility, invert emulsion mud, non-toxic additives, electrical stability, emulsion stability*

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INTRODUCTION

Successful drilling and completion of oil and gas wells to a considerable extent depend on the properties of the drilling fluids. The fluids are supposed to perform certain functions such as maintenance of wellbore stability, minimise fluid loss, lubricate and cool drill bit, clean and transport cuttings out of the wellbore. The choice of fluid depends mainly on the formation lithology, temperature and pressure, cost and logistics, environmental and health considerations. Invert emulsion fluids are usually preferred for drilling highly technical and challenging formations like shale because of the fluids sterling properties such as improved wellbore stability, high lubricity, penetration rate, and greater cleaning abilities with less viscosity. The enhanced shale inhibition ability of invert emulsion fluids is of particular importance in shale formations for multi-stage fracking. Jha *et al.* defined invert emulsion mud as oil based drilling mud to which water is added [1]. According to Wagle *et al.,* while drilling through shale, the water activity of the invert emulsion fluid is maintained at a lower level than the water activity of the shale creating an osmotic pressure that drives the flow of water from the shale to the invert emulsion fluid thereby preventing shale hydration [2]. These properties may help to reduce operation cost; however the use of potentially hazardous base fluids and chemicals like diesel pose several environmental issues associated with the disposal and hazards to personnel. The uncontrolled release of these fluids can result in the contamination of surrounding areas, including sources of drinking water, and can negatively impact natural habitat. It has become imperative for drilling fluids industry to provide oil-based drilling fluids using alternative base oils to diesel and non-toxic additives that will be environmentally friendly while maintaining high technical performance. Vegetable oil such as Jatropha has been shown to be a comparable alternative to diesel oil for environmentally friendly oil based drilling mud. However, compatibility of the vegetable oils with the usual chemical additives is still a major challenge that calls for compatible and non-toxic substitutes.

Within the industry it has been acknowledged that toxic additives are the high performers and need to be replaced with environmentally friendly mud additives. Apaleke *et al.* emphasised the need for careful selection of additives for vegetable oil-based mud stating that additives that will function in base-oil from hydrocarbon or synthetic source may not be functioning well in vegetable grade oil-base medium [3]. The hazardous effect on marine and human life have been reported due to the usage of additives such as defoamers, descalers, thinners, viscosifiers, lubricants, stabilisers, surfactants and corrosion inhibitors. Ferro-chrome lignosulfonate (a thinner and deflocculant) affected the survival and physiological responses of fish eggs and fry. Filtration control additive carboxymethyl cellulose (CMC) causes the death of fish fry at high concentrations (1000–2000 mg/ml) as physiological changes starts at the level of 12–50 mg/ml. According to Apaleke *et al.*, 896 t of drilling mud containing soltex, a fluid loss additive was dumped along the coast of Great Britain, thus contaminated the coast with potentially toxic heavy metals like antimony, arsenic, cadmium, nickel, lead, mercury, vanadium, zinc barium, fluoride and copper [4]. Materials that show good technical performance (stability) at high temperatures are frequently poor biodegraders and those that are most chemically active can show the highest toxicities [5]. In a preliminary test result of vegetable oil-based mud, Amanullah *et al.* stated that duratone used as fluid-loss control additive in mineral oil-based muds is not suitable for vegetable oil-based mud; hence the need for a suitable fluid loss additive for vegetable oil-based muds [6]. In toxicity assessment of individual ingredients of synthetic based drilling muds, the emulsifier caused the strongest biomarker responses. The primary emulsifier (Emul S50) followed by the fluid loss agent (LSL 50) caused the strongest biochemical responses in fish [7].

Zanten *et al.* described emulsions as unstable thermodynamic systems that can be stabilised by the use of surfactants or surface active solids to remain dispersed for any functionally relevant period of time [8]. In order for an emulsion to form, large amounts of energy must be put into the system in the form of shear. This energy is required due to increase in conformational entropy and surface area best explained using a thermodynamic framework. The free energy of the system is represented by:

$$\Delta G = \Delta H - T\Delta S \tag{1}$$

Or,

$$\Delta G = \Delta A - \gamma_{0w} - T\Delta S \tag{2}$$

Where:
G is the Gibbs free energy,
T is the temperature,
S is the entropy,
H is the enthalpy,
A is the interfacial area and
γ is the interfacial tension at the oil-water interface.

If the free energy is less than zero then the process is spontaneous, if it is greater than zero, then energy is required to be input into the system to cause the desired change. The stability of an emulsion can be enhanced by the addition of surfactant or emulsifier to decrease the surface tension making the droplets more stable. Emulsifiers are molecules that have hydrophilic and hydrophobic ends. While the hydrophilic end forms chemical bonds with water, the hydrophobic end forms chemical bonds with oil. According to Growcock *et al.*, water-in-oil emulsion is usually stabilised with a primary emulsifier usually a fatty acid salt [9]. Egg yolk contains some amino acids with a hydrophobic end that is attracted to the oil molecules and a hydrophilic end that is attracted to the water molecule. Lecithin which is a common phospholipid found in egg yolk is an effective emulsifier because of its polar and non-polar properties [10]. Egg yolk is a pseudo-plastic non-Newtonian fluid with a viscosity which depends on the shear forces applied. Its surface tension is 0.044 Nm^{-1} (25°C) while its pH is 6.0 and unlike egg white, increases only slightly to 6.4–6.9 after prolonged storage [11]. The use of non-toxic chemical additives would give a better environmentally friendly oil based mud. This study examined an environmentally friendly invert emulsion oil-based mud with Jatropha oil using hen egg yolk as emulsifier. This can be technically viable as diesel oil-based mud and equally compliant with health, safety and environment standards and regulations. This was compared with a similar mud formulation using a standard primary emulsifier (versacleanVB) an amido amine in place of the egg yolk while other components remain unchanged. VersacleanVB is irritating to the eyes and skin and not suitable for disposal into drains, sewers and watercourses.

MATERIALS AND METHOD

An invert emulsion mud consisting of 350 ml of oil-water ratio 70/30 using Jatropha oil as base fluid and egg yolk as emulsifier was formulated. Hen egg yolk was carefully separated from the albumen (egg white) by rolling it on a filter paper to remove adhering fragments of the albumen and allowed to flow into a suitable plastic container. The densities of the base oil (Jatropha) and water were measured using the mud balance. Using a beaker, 245 ml of Jatropha oil and 105 ml of water were measured. The weighing balance was used to measure the required quantities of calcium chloride and bentonite as shown in the mud composition in Table 1. The calcium chloride was added to the beaker of water. Using the Hamilton beach mixer, the measured materials were thoroughly mixed, adding 8 ml of egg yolk dropwise while mixing for 60 min.

Table 1: Mud Composition.

Component	Quantity
Base oil (Jatropha oil)	245 ml
Water	105 ml
Bentonite	22.5 g
Barite	117.6 g
Egg yolk	8 ml
VersacleanVB	8 ml
Calcium Chloride	4 g
Lime (Calcium Oxide)	12 g

Mud Weight Test

The weight of the formulated mud was measured using the mud balance shown in Figure 1. The mud balance was thoroughly cleaned and dried to avoid any irregularities in the reading. While in the upright position; the cup was filled with the mud to the brim and covered with the lid firmly seated while allowing some of the mud to expel through the vent to release any trapped air or gas. The outside of the cup was wiped and dried of any mud. The beam was placed on the base support and balanced by moving the rider along the graduated scale until balance was achieved when the bubble got under the centreline. At this point the mud weight was read in pounds per gallon (ppg). The weight was 8 ppg and pH measured with pH stripe was 5.7. The density and pH were increased to 10 ppg and 9.5 using barite and calcium oxide respectively. This involved further mixing for 30 min.

Electrical Stability Test

The electrical stability of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. The electrical stability test was carried out using the Fann electrical stability tester shown in Figure 2. The test was conducted at the recommended test temperature of 48.9°C and later at 120°C to check any variation at elevated temperature. The electrode probe was thoroughly cleaned, wiped and swirled in the base oil used for the mud formulation, cleaned and dried. The mud sample was placed in a glass beaker and maintained at a temperature of 48.9°C. The sample was hand-stirred using the electrode probe for 10 sec to ensure uniform composition and temperature of the sample. The electrode probe was positioned not to touch the bottom or sides of the container and the electrode surfaces completely covered by the sample. The test button was pushed and released for the automatic voltage ramp. The electrode probe was never moved during the measurement. At the stop of the ramp indicating voltage breakdown, the electrical stability reading was taken in volt. The test was repeated and the average of the readings was recorded as the electrical stability of the invert emulsion mud. The test was repeated at 120°C to examine the effect of elevated temperature on the stability and recorded as shown in Table 2. A repeat mud formulation was done using a standard primary emulsifier (versacleanVB) an amido amine in place of the egg yolk while other components remained unchanged. Similar tests were conducted to compare the emulsifying ability of egg yolk.

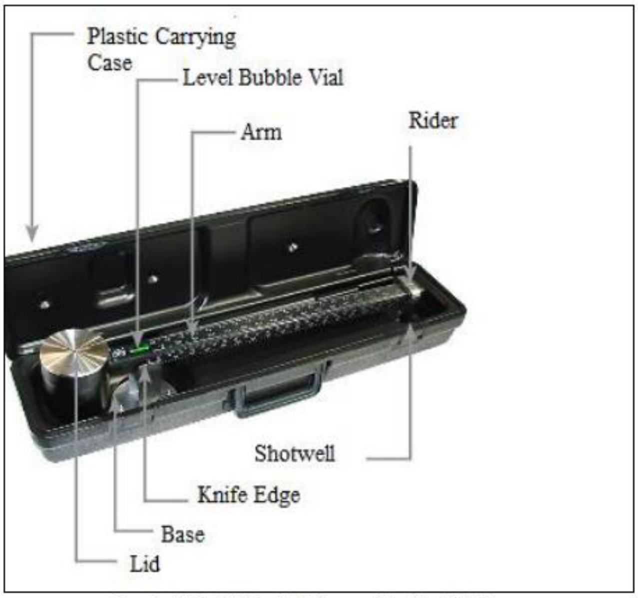

Fig. 1: Mud Weight Taken with Mud Balance.

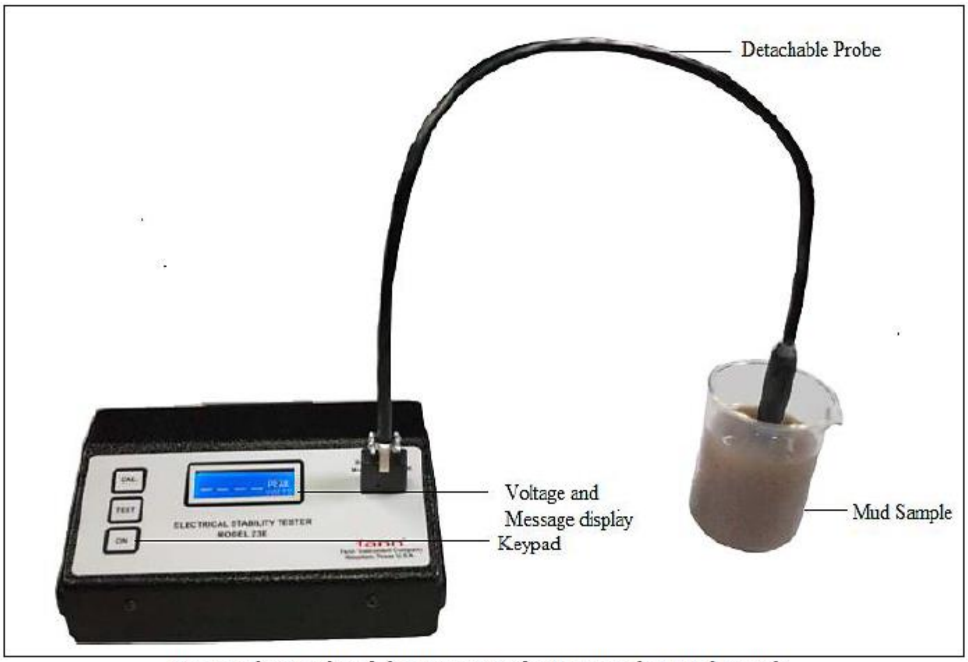

Fig. 2: Electrical Stability Tester Probe Immersed in Mud Sample.

Table 2: *Electrical Stability Values Using Egg Yolk and Versclean VB Emulsifier.*

Temperature (°C)	Mud Electrical Stability (V) with Egg Yolk	Mud Electrical Stability (V) with VersacleanVB
48.9	398	201
120	289	188

Rheology Test

The plastic viscosity, yield point and apparent viscosity of the formulated mud samples were also examined after aging for 16 h. The 8-speed viscometer was used for the measurement at the following dial settings 600, 300, 200, 100, 60, 30, 6 and 3 in rotation per minute (rpm) as shown in Tables 3 and 4. The mud was allowed to gel for 10 sec and 10 min respectively. The plastic viscosity, apparent viscosity and yield point were evaluated using the following relationships:

Plastic viscosity (PV) =dial reading for 600 rpm dial reading for 300 rpm (3)

Yield point (YP)=dial reading for 300 rpm–PV (4)

Apparent viscosity (AV)=600 rpm/2 (5)

The PV and AV were measured in centipoise (cP) while the yield point was measured in (Ib/100 ft^2). The results are shown in Tables 3 and 4 for the two mud samples. The effect of temperature on the viscosity of the mud emulsified with egg yolk was compared at 50 and 120°C and results obtained were illustrated. All tests were conducted according to American petroleum institute [12].

RESULTS AND DISCUSSION

The mud weight measured at 32°C was 10 ppg which is within the range of 8–12 ppg for mud weight of oil-water ratio of 65/35–75/25. Mud weight is an important property as it helps to maintain hydrostatic pressure, suspend cuttings and better cleaning of the bore hole. The electrical stability values using egg yolk and versacleanVB emulsifier are shown in Table 2. At 48.9°C the electrical stability values were 398 and 201 V for the egg yolk and verscleanVB emulsified muds respectively indicating that the mud was more stable with the egg yolk than with the versacleanVB emulsifier. According to Lyons, the estimated electrical stability for oil-based mud weight of 8–10 ppg of oil-water ratio 65/35–75/25 is 200–300 V [13, 14]. The electrical stability of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. The emulsion should be stable enough to incorporate additional water volume if downhole water flow is encountered. The rheology variation with temperature at different viscometer readings are shown in Tables 3 and 4.

Table 3: *Rheology Variation with Temperature at Different Viscometer Readings for Mud with Egg Yolk.*

Dial Setting (RPM)	50°C	70°C	100°C	120°C
600	173	160	64	49
300	121	109	43	32
200	90	75	29	22
100	58	46	23	19
60	37	30	21	15
30	25	22	17	13
6	21	15	14	11
3	14	13	12	10
Gel Strength, 10 sec (Ib/100 ft^2)	14	12	10	11
Gel Strength, 10 min (Ib/100 ft^2)	15	13	11	12
Apparent Viscosity (cP)	86.5	80	32	24.5
Plastic Viscosity (cP)	52	51	21	17
Yield Point (Ib/100 ft^2)	69	58	22	25

***Table 4:** Rheology Variation with Temperature at Different Viscometer Readings for Mud with VersacleanVB.*

Dial Setting (RPM)	50°C	70°C	100°C	120°C
600	168	157	62	47
300	119	107	42	30
200	89	72	26	20
100	53	42	20	16
60	31	27	17	13
30	20	19	13	11
6	15	10	11	9
3	13	9	10	10
Gel Strength, 10 sec (Ib/100 ft^2)	13	11	10	11
Gel Strength, 10 min (Ib/100 ft^2)	14	12	11	12
Apparent Viscosity (cP)	84	78.5	31	23.5
Plastic Viscosity (cP)	49	50	20	17
Yield Point (Ib/100 ft^2)	70	57	22	13

The viscosity values of each of the mud investigated was highest at 50°C than at temperatures of 70, 100 and 120°C. Figure 3 compares the electrical stability of the mud sample using egg yolk and versacleanVB. The mud exhibited higher stability with the egg yolk compared with versacleanVB. The electrical stability of the mud formulated with egg yolk at 48.9°C was 398 V and decreased to 289 V at 120°C. In the same order, the electrical stability of the mud formulated with versacleanVB was 201 V at 48.9°C and decreased to 188 V at 120°C. This decrease in electrical stability at increased temperature is associated with drilling fluids with oleic phase. The electrical stability of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. The emulsion should be stable enough to incorporate additional water volume if downhole water flow is encountered. Figures 4 and 5 illustrate the variation in viscosities with temperatures of the muds formulated using egg yolk and versacleanVB as emulsifiers respectively. From the figures there is not much variation in the viscosity of the mud emulsified by egg yolk and that of versacleanVB.

The viscosity of each of the mud steadily decreased as the temperature increased. The viscosity was highest at 50°C than at 70, 100 and 120°C. At 50°C and viscometer dial speed of 600 rpm, the viscosities were 173 and 168 cP for the mud emulsified with egg yolk and the mud emulsified with versacleanVB respectively. At increased temperature of 120°C, at the same dial speed of 600 rpm, the viscosities of the mud samples reduced to 49 and 47 cP respectively (Figure 6).

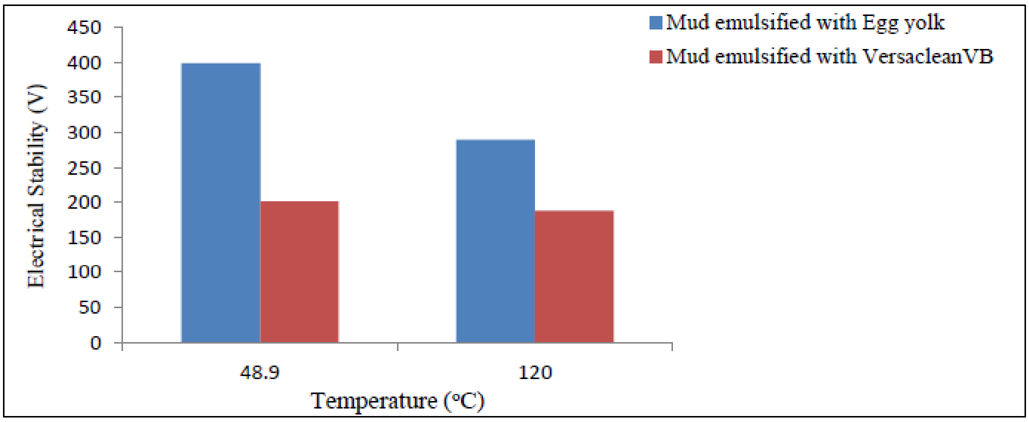

***Fig. 3:** Comparative Analysis of the Electrical Stability using Egg Yolk and VersacleanVB.*

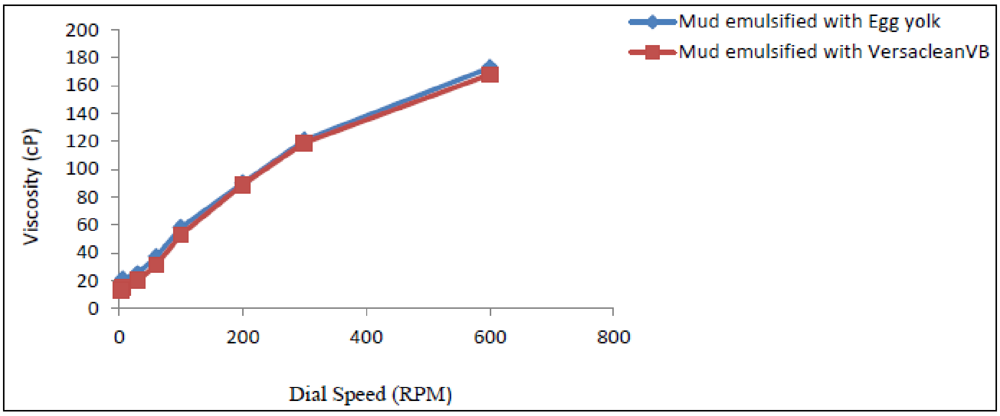

Fig. 4: Comparative Analysis of the Viscosities of the Muds using Egg Yolk and VerscleanVB Emulsifier at 50°C.

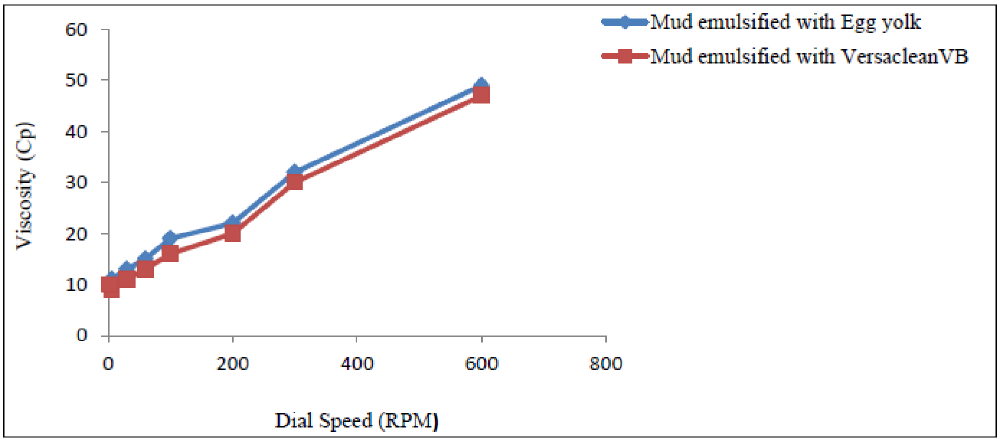

Fig. 5: Comparative Analysis of the Viscosities of the Muds using Egg Yolk and VerscleanVB Emulsifier at 120°C.

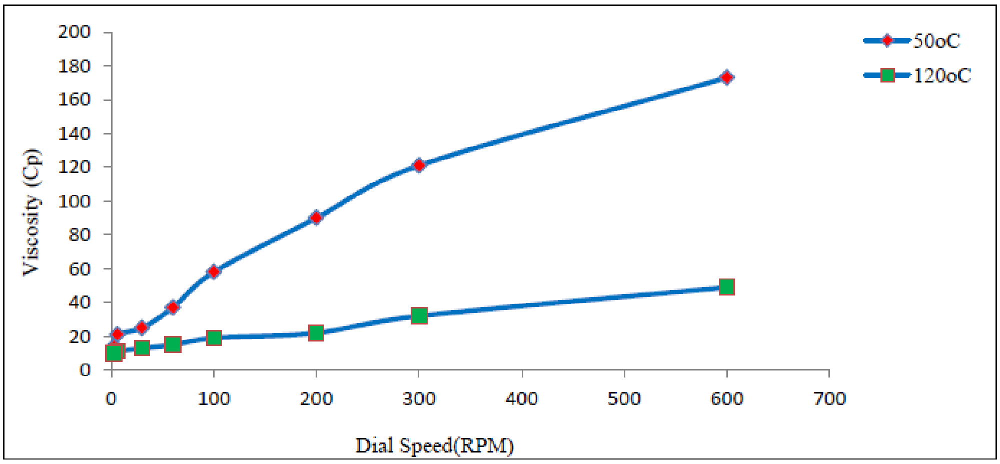

Fig. 6: Effect of Temperature on the Viscosity of the Mud Emulsified with Egg Yolk.

CONCLUSION

Based on the results from this study, the following conclusions are drawn:

The electrical stability value of the mud emulsified with egg yolk was higher than that emulsified with versacleanVB. The electrical stability of the mud formulated with egg yolk at 48.9°C was 398 V and decreased to 289 V at 120°C. In the same order the electrical stability of the mud formulated with versacleanVB was 201 V at 48.9°C and decreased to 188 V at 120°C. This is an indication of compatibility of the egg yolk and the vegetable base oil. The decline in stability at increased temperature is associated with drilling fluids containing oleic phase as the number of collisions between droplets increases. The reduced stability value of 289 V for the mud emulsified by egg yolk was still within the acceptable estimated value according to literature. There was no significant variation in viscosity of the mud samples emulsified with egg yolk and versacleanVB. The plastic viscosity was 52 cP at 50°C and 17 cP at 120°C for the egg yolk emulsified mud while that of versacleanVB was 49 and 17 cP. The invert emulsion system was formulated without a wetting agent and secondary emulsifier. This will reduce cost of mud formulation. The study suggests that egg yolk could serve as an emulsifier for vegetable oil based invert emulsion mud for compatibility and in line with the call for non-toxic additives for environmentally friendly drilling mud. Generally, non-toxic additives and vegetable oils have the capability to replace some of the toxic additives and mineral oils which is beneficial in compatibility with the environment, lower overall cost and less harmful to personnel.

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50/50 Oil-Water Ratio Invert Emulsion Drilling Mud Using Vegetable Oil as Continuous Phase

P. C. Ihenacho, M. Burby, G. G. Nasr, G. C. Enyi

Abstract—Formulation of a low oil-water ratio drilling mud with vegetable oil continuous phase without adversely affecting the mud rheology and stability has been a major challenge. A low oil-water ratio is beneficial in producing low fluid loss which is essential for wellbore stability. This study examined the possibility of 50/50 oil-water ratio invert emulsion drilling mud using a vegetable oil continuous phase. Jatropha oil was used as continuous phase. 12 ml of egg yolk which was separated from the albumen was added as the primary emulsifier additive. The rheological, stability and filtration properties were examined. The plastic viscosity and yield point were found to be 36cp and 17 Ib/100 ft^2 respectively. The electrical stability at 48.9°C was 353v and the 30 minutes fluid loss was 6ml. The results compared favourably with a similar formulation using 70/30 oil - water ratio giving plastic viscosity of 31cp, yield point of 17 Ib/100 ft^2, electrical stability value of 480v and 12ml for the 30 minutes fluid loss.

This study indicates that with a good mud composition using guided empiricism, 50/50 oil-water ratio invert emulsion drilling mud is feasible with a vegetable oil continuous phase. The choice of egg yolk as emulsifier additive is for compatibility with the vegetable oil and environmental concern. The high water content with no fluid loss additive will also minimise the cost of mud formulation.

Keywords—Environmental compatibility, low cost of mud formulation, low fluid loss, wellbore stability.

I. INTRODUCTION

SUCCESSFUL drilling and completion of oil and gas wells to a considerable extent depend on the properties of the drilling fluids. The fluids are supposed to perform certain functions such as maintenance of wellbore stability, minimise fluid loss, lubricate and cool drill bit, clean and transport cuttings out of the wellbore. The choice of fluid depends mainly on the formation lithology, temperature and pressure, cost and logistics, environmental and health considerations. Invert emulsion fluids are usually preferred for drilling highly technical and challenging formations like shale. This is due to the fluids suitable properties such as improved wellbore stability, high lubricity, penetration rate, and greater cleaning abilities with less viscosity. The enhanced shale inhibition ability of invert emulsion fluids is of particular importance in shale formations for multi-stage fracking. Invert emulsion mud was defined by [7] as oil based drilling mud to which water is added to. According to [9] while drilling through shale, the water activity of the invert emulsion fluid is maintained at a lower level than the water activity of the shale thus creating an osmotic pressure that drives the flow of water from the shale to the invert emulsion fluid thereby preventing shale hydration. These properties may help to reduce operation cost; however, the use of potentially hazardous base fluids and chemicals like diesel pose several environmental issues associated with the disposal and hazards to personnel. The uncontrolled release of these fluids can result in the contamination of surrounding areas, including sources of drinking water, and can negatively impact natural habitat. Therefore, the current trend is towards development of environmentally friendly oil-based drilling fluids using alternative base oils to diesel while maintaining high technical performance. Over the years several plant oils have become increasingly popular in the raw materials market as diesel substitutes [6]. The most popular is Rapeseed oil, Mahua oil, Cottonseed oil, Sesame oil, Soya bean oil, palm oil, Canola oil, Moringa seed oil, Soapnut and Jatropha because of their low toxicity. In their study, [6] confirmed that Jatropha oil exhibited lower toxicity compared to canola and diesel oils. However, there have been conflicting issues with the vegetable oils in maintaining the mud technical properties like good rheology, high stability and low fluid loss.

It is always a challenge to reduce the oil-water ratio (OWR) during the formulation of mud system. So far the industry was not able to reduce the OWR in their formulation beyond 85/15. This is a major gap in the previous works in the subject area toward the development of sustainable oil-based mud (OBM) systems using non-toxic, edible vegetable oils [4]. Vegetable oils are highly viscous and a low oil- water ratio will mean a very high viscous mud. Brine has a high density and a mixture of salts. Hence, the more the amount of brine internal phase added to a viscous vegetable oil the more complex the entire nature of the mud system becomes. A low /water ratio (e.g. 60/40) is beneficial in producing low fluid loss although mud rheology will need to be considered [5]. Therefore, this study examined the possibility of a reduced oil-water ratio invert emulsion system with a vegetable oil continuous phase. This will reduce cost of formulation because the more water in the mud system the cheaper the cost of formulation. With a reduced OWR, the fluid loss will be low which is beneficial for wellbore stability and low oil retention on cuttings which makes for easy cuttings treatment and disposal. In a preliminary test result of vegetable oil-based mud, [1] stated that Duratone used as Fluid-loss control additive in mineral oil-based muds is not suitable for vegetable oil-based mud; hence the need for a suitable fluid loss additive for vegetable oil- based muds. While the industry works on a

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suitable fluid loss additive for vegetable oil, a low OWR such as 50/50 will give a low fluid loss. This study has formulated the mud without a fluid loss additive. While considering low OWR with a vegetable oil continuous phase [3], [4] emphasised the need for careful selection of additives for vegetable oil-based mud stating that additives that will function in base-oil from hydrocarbon or synthetic source may not be functioning well in vegetable grade oil-base medium. Therefore, this study has used egg yolk as an emulsifier additive for compatibility and environmental concern.

A low OWR is beneficial for low fluid loss. This study examined the possibility of 50/50 oil-water ratio invert emulsion drilling mud with a vegetable oil continuous phase.

II. Materials and Method

An invert emulsion mud consisting of 350ml of oil-water ratio 50/50 using Jatropha oil as the continuous phase and egg yolk as emulsifier was formulated. Hen egg yolk was carefully separated from the albumen (egg white) by rolling it on a filter paper to remove adhering fragments of the albumen and allowed to flow into a suitable plastic container. The densities of the base oil (Jatropha) and water were measured using the mud balance. Using a beaker, 175 ml of Jatropha oil and 175ml of water were measured. The weighing balance was used to measure the required quantities of calcium chloride and bentonite as shown in the mud composition in Table I. The calcium chloride was added to the beaker of water. Using the Hamilton Beach mixer, the measured materials were thoroughly mixed, adding 12ml of egg yolk dropwise while mixing for 60 minutes.

The weight of the formulated mud was measured using the mud balance. The mud balance was thoroughly cleaned and dried to avoid any irregularities in the reading. While in the upright position; the cup was filled with the mud to the brim and covered with the lid firmly seated while allowing some of the mud to expel through the vent to release any trapped air or gas. The outside of the cup was wiped and dried of any mud. The beam was placed on the base support and balanced by moving the rider along the graduated scale until balance was achieved when the bubble got under the centreline. At this point the mud weight was read in pounds per gallon (ppg). The mud weight was 8.1ppg.

The electrical stability of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. The electrical stability test was carried out using the Fann electrical stability tester shown in Fig. 1. The test was conducted at the recommended test temperature of 48.9°C and later at 120°C to check any variation at elevated temperature. The electrode probe was thoroughly cleaned, wiped and swirled in the base oil used for the mud formulation, cleaned and dried. The mud sample was placed in a glass beaker and maintained at the test temperature. The sample was hand-stirred using the electrode probe for 10 seconds to ensure uniform composition and temperature of the sample. The electrode probe was positioned not to touch the bottom or sides of the container and the electrode surfaces completely covered by the sample. The test button was pushed and released for the automatic voltage ramp. The electrode probe was never moved during the measurement. At the stop of the ramp indicating voltage breakdown, the electrical stability reading was taken in volts. The test was repeated and the average of the readings was recorded as the electrical stability of the Invert emulsion mud as shown in Table II.

The filtration property was checked using the high temperature high pressure (HTHP) filter press shown in Fig. 2 at 120°C and 500psi. The filtrate was collected at the end of 30mins as shown in Table III.

The rheological property (Plastic viscosity, Yield point and apparent viscosities) of the formulated mud sample was also examined after aging for 16 hours. The 8-speed viscometer was used for the measurement at the following dial settings 600, 300, 200, 100, 60, 30, 6, and 3 in rotation per minute (RPM) as shown in Table IV. The mud was allowed to gel for 10 seconds and 10 minutes respectively. The plastic viscosity, apparent viscosity and yield point were evaluated using the following relationships:

$$\text{Plastic viscosity (PV)} = \text{dial reading for 600 rpm} - \text{dial reading for 300 rpm} \quad (1)$$

$$\text{Yield point (YP)} = \text{dial reading for 300rpm} - \text{PV} \quad (2)$$

$$\text{Apparent Viscosity (AV)} = \text{600rpm} / 2 \quad (3)$$

The PV and AV were measured in centipoise (cP) while the yield point was measured in (Ib/100 ft^2).

A repeat mud formulation was done with oil-water ratio of 70/30 increasing the bentonite to 10g and reducing the emulsifier to 8ml while other components remain unchanged. Similar tests were conducted to compare the rheological, emulsion stability and filtration properties of 50/50 oil-water ratio mud.

All tests were conducted according to [2].

III. Results and Discussion

The mud weight measured at 32°C was 8.1ppg for oil-water ratio of 50/50 and 70/30. Mud weight is an important property as it helps to maintain hydrostatic pressure, suspend cuttings and better cleaning of the bore hole. The electrical stability values of the 50/50 and 70/30 OWR muds at various temperatures shown in Table II were within the range of electrical stability for a stable mud. At 48.9°C the electrical stability values were 353v and 480v for the mud of 50/50 and 70/30 OWR muds respectively. According to [8], the estimated electrical stability for oil-based mud of mud weight of 8-10 ppg of OWR 65/35-75/25 is 200-300 volts. The electrical stability of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. The emulsion should be stable enough to incorporate additional water volume if down hole water flow is encountered. The 50/50 OWR mud had a lower fluid loss of 6ml compared to12ml of the 70/30 OWR mud as shown in Table III. The rheological behaviour of the 50/50 and 70/30 OWR muds at different viscometer readings are shown in Table IV. Higher water content clearly improves the fluid loss, though the viscosity of the system may be high, hence the quantity of the viscosifier used for the mud of 50/50 oil-water ratio was 3g of bentonite.

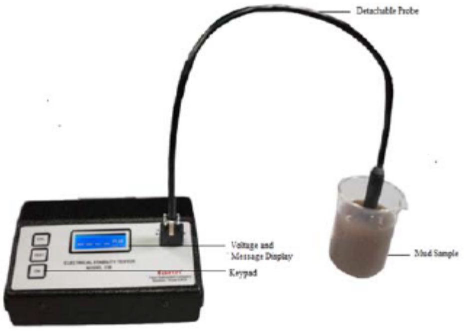

Fig. 1 Electrical Stability Tester

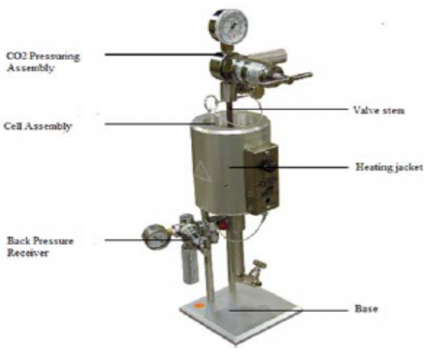

Fig. 2 High Temperature-High Pressure (HTHP) Filter Press

Fig. 3 compares the electrical stability of the 50/50 and 70/30 OWR muds. The electrical stability of the 50/50 OWR mud compared favourably with 70/30 OWR mud without much variation. At 48.9°C the electrical stability values were 353v and 480v for the 50/50 and 70/30 oil-water ratio muds respectively. However, the electrical stability of the 50/50 OWR mud decreased to 258V at 120°C. In the same order the electrical stability of the 70/30 OWR mud decreased to 393V. This decrease in electrical stability at increased temperature is associated with drilling fluids with oleic phase. The electrical stability of an oil-based drilling mud is a property related to its emulsion stability and oil-wetting capability. Fig. 4 compares the viscosities of the 50/50 and 70/30 OWR mud samples. There is no significant variation in the viscosities of the 50/50 and 70/30 OWR muds. From Table IV, the plastic viscosity of the 50/50 OWR mud is 36cp and a yield point of 17 Ib/100 ft^2 while the 70/30 OWR mud had plastic viscosity of 31cp and yield point of 17 Ib/100 ft^2. The 10 seconds and 10 minutes gel strength of the 50/50 OWR mud was 3 and 4 Ib/100 ft^2 while that of 70/30 OWR mud was 6 and 7 Ib/100 ft^2 respectively. The 30 minutes fluid loss for the 50/50OWR mud was 6ml and that of 70/30 was 12ml as shown in Fig. 5. There was no fluid loss additive in the mud formulation. These results have shown that with a good composition, a 50/50 OWR invert emulsion mud with vegetable oil continuous phase is feasible. Despite the higher water content, the electrical stability, rheological behaviour of the 50/50 OWR mud compared favourably with usual 70/30 OWR mud while achieving a low fluid loss which is essential for wellbore stability.

TABLE I
MUD COMPOSITION

Component	Quantity	
	50/50 OWR Mud	70/30 OWR Mud
Base oil (Jatropha oil)	175ml	245ml
Water	175ml	105ml
Bentonite	3g	10g
Egg yolk emulsifier	12ml	8ml
Calcium Chloride	4g	4g
Lime (Calcium Oxide)	3g	3g

TABLE II
ELECTRICAL STABILITY VALUES OF 50/50 AND 70/30 OWR MUDS

Temperature (°C)	Electrical Stability(V) 50/50 OWR Mud	Electrical Stability(V) 70/30 OWR Mud
48.9	353	480
120	258	393

TABLE III
FLUID LOSS OF 50/50 AND 70/30 OWR MUDS AT 120°C AND 500 PSI

Fluid loss (ml)	50/50 OWR Mud	70/30 OWR Mud
30mins	6	12

IV. CONCLUSION

Based on the results from this study, the following conclusions are drawn:

The variation in the electrical stability values of the 50/50 and 70/30 OWR muds was minimal. There was also no significant viscosity variation of the two samples despite the higher water content of the 50/50 OWR mud. The higher water content in the 50/50 OWR means less retention of oil on the cuttings which means less treatment of the cuttings before disposal, also reducing cost of disposal and less impact on the environment. The mud system was formulated without a wetting agent and fluid loss additive; this reduces cost of formulation. The use of egg yolk as emulsifier is for compatibility with the vegetable oil continuous phase and the call for non-toxic additives. The fluid loss of 6ml of the 50/50 OWR ratio is beneficial for well bore stability.

Generally, with a good composition using guided empiricism, 50/50 OWR invert emulsion mud is feasible with a vegetable oil continuous phase. This is technically viable in reduction of fluid loss for enhanced wellbore stability. Less oil retention on cuttings will reduce cost of disposal and environmental impact. The high water content with no fluid loss additive also means less cost of mud formulation.

TABLE IV
RHEOLOGICAL BEHAVIOUR AT DIFFERENT VISCOMETER READINGS FOR 50/50 AND 70/30 OWR MUDS

Dial Setting (RPM)	Mud Type	
	50/50 OWR Mud	70/30 OWR Mud
600	89	79
300	53	48
200	38	40
100	23	26
60	15	16
30	9	11
6	3	9
3	2	6
Gel Strength,10seconds (Ib/100 ft²)	3	6
Gel Strength,10 minutes (Ib/100 ft²	4	7
Apparent Viscosity (cP)	44.5	39.5
Plastic Viscosity(cP)	36	31
Yield Point (Ib/100 ft²)	17	17

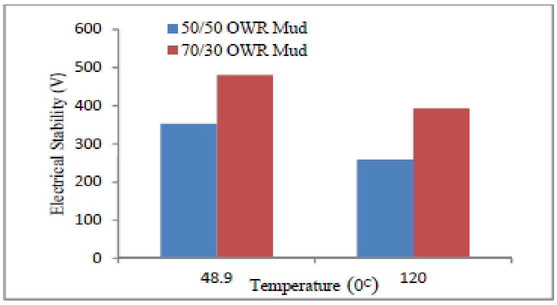

Fig. 3 Comparative Analysis of the Electrical Stability of 50/50 and 70/30 OWR Muds

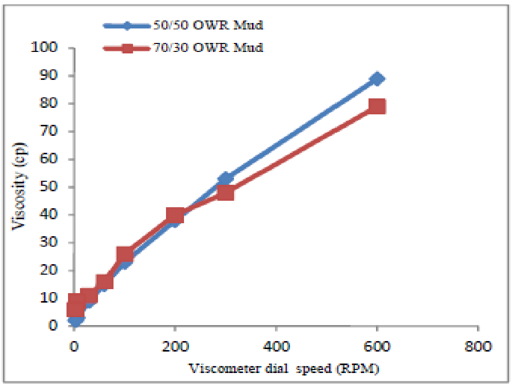

Fig. 4 Comparative Analysis of the Viscosities 50/50 and 70/30 OWR Muds

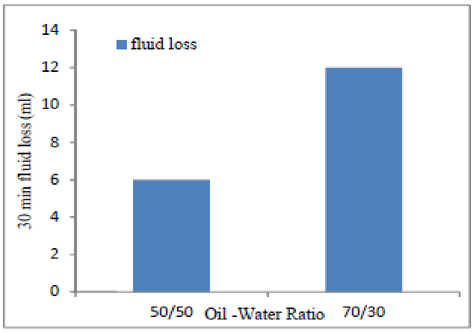

Fig. 5 Comparative Analysis of the Fluid loss of 50/50 and 70/30 OWR Muds

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Economic evaluation of environmentally friendly vegetable oil-based invert emulsion

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Abstract: Stringent environmental regulations and technical requirements of difficult formations such as shale demand the use of functional mud system to complete a well safely and economically. The economic viability of 50/50 oil-water ratio invert emulsion which uses vegetable oil and egg yolk as a non-toxic emulsifier was evaluated. The evaluation showed less cost of mud formulation by 67% and disposal by 47.5%. This equate to saving of $55.82 per barrel of invert emulsion formulated and $28.50 per barrel disposed. The low oil-water ratio mud is viable for low fluid loss for enhanced wellbore stability and less oil retained on drilled cuttings

Keywords: Environmental compatibility, non-toxic additives, vegetable oil base fluid, low cost Wellbore stability.

1.Introduction

Drilling fluid or mud is an essential element that drives every drilling operation. It solely represents one fifth (15 -20%) of the total cost of well drilling [1]. Since no two drilling operations are the same it is difficult to get a standard drilling fluid. The difference in environmental regulations throughout the world contributed to the difficulty in finding an effective, high performance drilling fluid with low cost. A cost effective, customised fluid solution is necessary to ensure maximum drilling performance and reduce the risk of major down hole issues. According to [2], the economic losses caused by wellbore instability account for more than one billion dollars every year and the lost time accounts for over 40% of all drilling related non-productive time.

The choice of fluid depends mainly on the formation lithology, cost and environmental concerns. Invert emulsion mud has been extensively used in the oil and gas industry especially for difficult and challenging formations such as shale due to the fluids sterling properties such as improved wellbore stability, penetration rate, and greater cleaning abilities with less viscosity. The enhanced shale inhibition ability of invert emulsion fluids is of particular importance in shale formations for multi-stage fracking. [1] defined invert emulsion mud as oil based drilling mud to which water is added. According to [2] while drilling through shale formation, the water activity of the invert emulsion fluid is maintained at lower level than the water activity of the shale. This creates an osmotic pressure that drives the flow of water from the shale to the invert emulsion fluid thereby preventing shale hydration. These properties may help to reduce operation cost. However, the use of potentially hazardous base fluids and chemicals like diesel pose environmental issues associated with the disposal and hazards to personnel. This has called for alternative base oils to diesel and non-toxic additives that will be environmentally friendly while maintaining high technical performance. Over the years several plant oils such as Rapeseed oil, Mahua oil, Cottonseed oil, Sesame oil, Soya bean oil, palm oil, Canola oil, Moringa seed oil, Soapnut and Jatropha have become popular as substitute for diesel because of their low toxicity. Some of these vegetable oils are relatively more expensive than diesel which could ultimately increase the cost of drilling fluid however, considering the harmful effect of diesel oil-based mud and high cost of disposal, the use of non-toxic vegetable oils becomes necessary.

[3] investigated the rheological, filtration and toxicity properties of palm oil-based mud. He observed that palm oil-based mud was non-toxic, cheap and had high flash point and good emulsion stability. However, the mud had undesirable properties such as high plastic viscosity, high pour point, low aniline point and high filter loss. [4] encouraged the use of oil based muds developed with palm oil and groundnut oil due to their high level of biodegradability and better eco-toxicological properties, however they exhibited adverse effects such as high viscosity and progressive gel. In a similar study, [5] investigated the physical and chemical properties of castor oil as vegetable oil-based mud and also observed relatively very high viscosity. [6][7] investigated the properties of Jatropha oil as vegetable oil-based mud, they observed that Jatropha oil exhibited better adaptability, higher carrying capacity and less pressure loss in pipe than diesel oil-based mud.

Above reviews on work with vegetable oils suggests that Jatropha oil is the most relatively technically, environmentally and economically viable alternative to diesel oil. However, the formulation of low oil-water ratio mud with vegetable oil has a major challenge. It adversely affects the mud rheology and stability. According to [8], low oil-water ratio of 60/40 is beneficial in producing low fluid loss although mud rheology will need to be considered. It is always a challenge to reduce oil-water ratio during the formulation of a mud system. So far the industry was not able to reduce the oil-water ratio in their mud formulation beyond 85/15. This is a major gap in the previous work towards the development of sustainable oil based mud systems using vegetable oils [9]. In a preliminary test result of vegetable oil-based mud, [10] noted that duratone used as fluid-loss control additive in mineral oil-based muds is not suitable for vegetable oil-based muds. [11] emphasized the need for careful selection of additives for vegetable oil-based mud. He noted that additives that will function in base-oil from hydrocarbon or synthetic source may not be functioning well in vegetable grade oil-base medium. In line with compatible and non – toxic additives, [12] formulated 50/50 oil-water ratio invert emulsion using Jatropha oil and egg yolk as a non-toxic emulsifier. The electrical stability value of the mud was 353volts at 48. 9°C. The high water content clearly improves the filtration property with a 30-minute fluid loss of 6ml which is essential for wellbore stability. This will also reduce the oil retained on drilled cuttings thereby reducing the cost of disposal.

This study therefore evaluates the economic viability of 50/50 oil-water ratio invert emulsion using Jatropha oil and egg yolk as a non-toxic emulsifier.

2. Physico-chemical Properties of Prospective Vegetable Oil Base Fluids

The knowledge of the physico-chemical properties of prospective vegetable oils base fluids is very essential as it helps in early estimation of the mud composition and behaviour. These properties include the following: *specific gravity flash point fire point pour point, kinematic viscosity, aniline point and cloud point.* According to [11], vegetable oil-based mud will have several advantages over the mineral oil-based mud due to the possession of the following superior properties such as high flash point, high fire point, and high biodegradability. He further stated that higher flash and fire points indicate better fire resistant capacity and minimum chances of causing operational problems associated with low flash and low fire points. The high flash and fire point also ensure enhanced safety in handling, storage and transportation. Vegetable oils are usually 95-100% biodegradable, non-toxic and pose little or no danger to aquatic or terrestrial, offshore or onshore environment compared to mineral oils of only 30% biodegradability. Table 1 compares the properties of Jatropha and Diesel oils as base oils in oil-based mud.

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Table 1 Comparison of Properties of Jatropha and Diesel oils as Base oils used in Oil-Based Muds

Property	Aniline Point (^{O}C)	Pour Point (^{O}C)	Flash Point (^{O}C)	Fire Point (^{O}C)	Kinematic Viscosity @40^{O}C, cST	Aromatic Content (%)
Required Properties of Base Oil	> 65	<Ambient temperature	> 66	> 80	2.3 -3.5	4-8
Diesel Oil		- 6	65	78	2.86	
Jatropha Oil		6	214	256	36.92	

Source of Required Properties of Base oil- **Yassin et al 1991**

Key: **Green**- Compatible, **Red**- Not compatible, **White** –Not determined

Diesel and Jatropha oils were evaluated in relative to some characteristics such as availability, cost effectiveness, environmental compatibility, rheological and filtration properties. These characteristics were ranked in low, medium and high as shown in Table 2

Table 2 Evaluation Matrix of Diesel and Jatropha Oil

Base Oil	**Rheological Properties**	**Filtration Properties**	**Cost Effectiveness**	**Availability**	**Environmental Compatibility**
Diesel	High	High	Low	High	Low
Jatropha	Medium	High	High	High	High

2.1 Cost Comparison for formulation of 50/50 OWR Diesel and Jatropha Oil Based Muds

One barrel of 50/50 OWR invert emulsion mud was formulated using diesel and jatropha oils respectively. The conventional additives were used for diesel based mud while egg yolk was used as non-toxic emulsifier in the case of the Jatropha oil-based mud. The cost comparison of these muds was based on the base oil and the emulsifier. It is worth stating here, that not all the benefits of vegetable oil-based mud are measurable in financial terms considering all the environmental benefits. It is also difficult to find accurate comparative data for total cost of formulation of diesel oil-based and vegetable oil-based muds. With the available data, comparison of the formulation cost of one barrel of diesel oil-based mud and vegetable oil-based mud is shown in Table 3.

Table 3 Cost Comparison for formulation of 1bbl of 50/50 OWR Diesel and Jatropha Oil Based Muds

Cost Parameter	**Diesel Oil-Based Mud**	**Jatropha Oil-Based Mud**
Cost of base oil (US$/bbl)	130.62	54.8
Required volume of base oil (bbl)	0.50	0.50
Cost of base oil per required volume (US$/bbl)	65.31	27.4

Cost of Mud Additive - emulsifier (US$/bbl)	1.5	0.007
Required volume of Mud Additive(emulsifier) (ml)	12	12
Cost of Mud Additive per required volume (US$/bbl)	18	0.09
Cost of base oil + Additives (US$/bbl)	**83.31**	**27.49**
Total cost of formulation of 1barrel (US$/bbl)	**83.31**	**27.49**

2.2. Cost Comparison of Management and Disposal of Diesel oil-based and Vegetable oil-based muds

Throughout the drilling process, drilling mud is recirculated, which helps to decrease waste by reusing as much mud as possible. However, when the drilling process is completed, the drilling waste must be disposed of. Method of disposal of the used mud and drilled cuttings vary depending on the choice of the operator. The choice to a large extent depend on the type of generated cuttings and cost of treatment and disposal. Disposal method could be onsite, offsite, using farmlands, landfills, and thermal technologies. Salt water muds and oily cuttings are not suitable for onsite management. In some cases environmental sensitivity precludes onsite waste management. Cost effectiveness is also another reason for commercial waste management facilities because rather than constructing, operating and closing an onsite facility for a relatively small volume of waste [13]. Oil-based mud cuttings from diesel and mineral oils pose a complex and costly waste management challenge and cannot be discharged on-site. Waste streams high in hydrocarbons ranging from 10-40 % like oil-based mud are candidates for thermal treatment technology. Cost of thermal treatment of oily waste ranges from $75 to $150 per ton with labour being a large component [14]. Diesel oil-based muds pose greater risk than vegetable oil based mud through skin irritation and effects of inhalation. The added transport and disposal costs as well as potential liability issues associated with diesel oil-based mud have restricted the widespread usage while putting vegetable oil-based mud as a preferred alternative. In 1997 disposal costs reported by [15] offsite commercial disposal facilities for oil-based drilling wastes ranged from $0 to $57/bbl and for water based drilling waste ranged from $0.20 to $14.70. Most operators charge transportation cost by hour typically $55.00/hr to $175.00/hr. Others use per-load or per container basis for instance in one case $1.00/bbl to $3.00bbl [16]. Not all the benefits of management and disposal of vegetable oil based mud are measurable in financial terms considering all the environmental benefits. It is also difficult to find accurate comparative data for total drilling waste management costs for diesel oil-based and vegetable oil-based muds. With the available data, comparison of the disposal cost of one barrel of diesel oil-based mud and vegetable oil-based mud is shown in Table 4.

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Table 4 Cost Comparison of Management and Disposal of Diesel oil-based and Vegetable oil-based muds

Cost Parameter	**Diesel Oil-Based Mud**	**Vegetable Oil-Based Mud**
Cost of transportation of drilling wastes to disposal site (US$/bbl)	3.0	3.0
Cost of Commercial disposal of drilling wastes (US$/bbl)	57.0	28.50
Cost of transportation + Disposal (US$ /bbl)	**60.0**	**31.50**
Total cost of disposal of 1barrel (US$/bbl)	**60.0**	**31.50**

3. Result and Discussion

Comparison of the cost of formulation of one barrel of mud evaluated on base oil and emulsifier showed that Jatopha oil-based mud at the cost of $27.49/bbl is cheaper than diesel oil-based mud at $83.31/bbl as illustrated in Figure 1. Similarly, the cost of disposal of drilling wastes of jatropha oil-based mud at $31.50 is lower than that of diesel oil-based mud at $60.00 as shown in Figure 2. These evaluations as illustrated in Figure 1 and Figure 2 respectively have shown less cost of mud formulation by 67% and disposal by 47.5%. This equates to saving of $55.82 per barrel of invert emulsion formulated and $28.50 per barrel disposed as shown in Figure 3. The cuttings generated using diesel oil-based muds need special treatment before discharging them to prevent contamination of water with free oil. The use of more water in the formulation of 50/50 OWR invert emulsion mud means less cost of formulation. This is technically viable in reduction of fluid loss for enhanced wellbore stability. Less oil retention on cuttings will reduce cost of disposal and environmental impact. The high water content with no fluid loss additive also means less cost of mud formulation. [12] While drilling through shale, the water activity of the invert emulsion fluid is maintained at a lower level than the water activity of the shale creating an osmotic pressure that drives the flow of water from the shale to the invert emulsion fluid thereby preventing shale hydration and less fluid loss which are essential for wellbore stability. High wellbore stability will ensure maximum drilling performance thereby reducing non-productive down-time and economic losses. The comparison of the cost of formulation and disposal of diesel oil-based mud and Jatropha oil-based mud has shown that vegetable oil-based mud is relatively more economically viable than diesel oil-based mud.

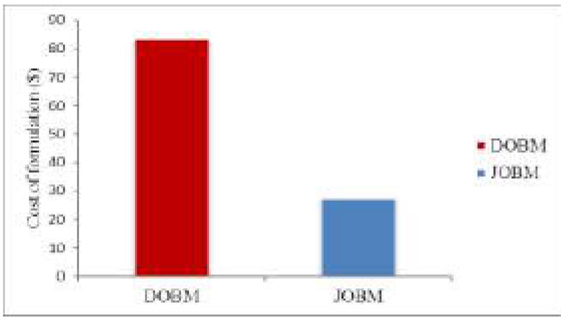

Figure1 Cost Comparison for formulation of 1bbl of 50/50 OWR Diesel and Jatropha Oil Based Muds

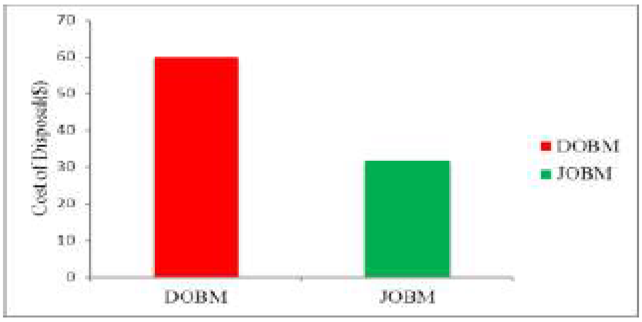

Figure 2 Cost Comparison of Management and Disposal of Diesel oil-based and Jatropha oil-based muds

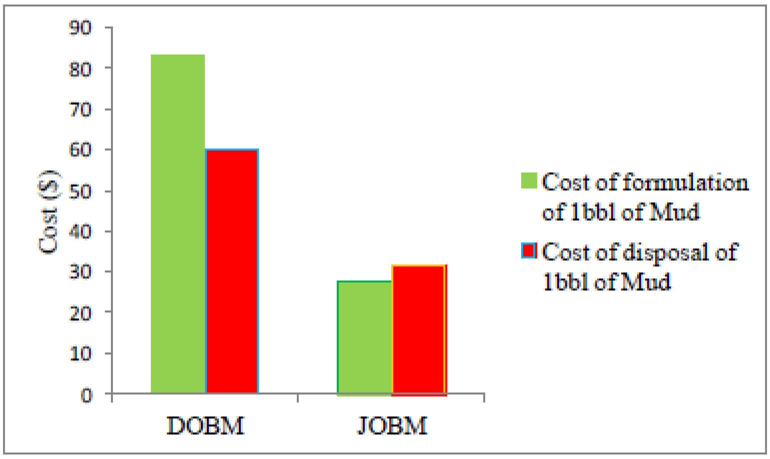

Figure 3 Cost Comparison of Formulation and Disposal of Diesel oil-based and Jatropha oil-based muds

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Conclusion

The economic evaluation of 50/50 oil-water ratio invert emulsion using vegetable oil and egg yolk as a non-toxic emulsifier has shown less cost of mud formulation by 67% and disposal by 47.5%. This equates to saving of $55.82 per barrel of invert emulsion formulated and $28.50 per barrel disposed. The low oil-water ratio mud is essential for low fluid loss for enhanced wellbore stability and less oil retained on drilled cuttings. It is difficult to find accurate comparative data for total drilling formulation, waste management costs for diesel oil-based and vegetable oil-based muds, however from available data, diesel oil-based mud exhibited higher cost compared to jatropha oil-based mud. Again, it is not all the benefits of formulation, management and disposal of vegetable oil based mud are measurable in financial terms considering all the environmental benefits. The use of Jatropha oil and egg yolk as emulsifier means more safety to personnel, no detrimental effect on the environment and overall reduction on cost of mud formulation, treatment and disposal.

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Shale-fluid Interaction in Environmentally Friendly Invert Emulsion Using an X-Ray Computed Tomography (CT) Scan

Peace C Ihenacho[1] Martin Burby[2] Ghasem Nasr[3]

Abstract

Incompatibility of drilling fluids with shale formations is one of the root causes resulting in instability and in major cases collapse of the wellbore. Shale-fluid interaction has been investigated in Jatropha oil-based invert emulsion and is recognised as one of the alternatives to diesel mud for environmental concerns. Immersion test was conducted using dried Marcellus shale samples with exposure to jatropha oil-based mud, diesel oil-based mud and water based mud. Test samples were analysed using an X-ray Computed Tomography (CT) Scan. Significant volume increase by 370.14mm^3 (11%) and 803.98mm^3 (23%) was observed for the 1 and 7-day tests respectively in the shale sample exposed to water based mud with significant development of fractures, which is not conducive for wellbore stability. Comparatively, there was no development of fractures on the samples exposed to diesel and jatropha oil based muds with slight volume increase by 30.95mm^3 (0.88%) and 88.54mm^3 (2.53%) for the 1 and 7-day tests with no variation in the structures. This indicates that Jatropha oil-based mud could be categorised as a relatively environmentally friendly substitute to diesel mud for drilling shale formations while maintaining wellbore stability.

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Key words: Shale Stability, Wellbore Stability, Shale-Fluid interaction, Invert emulsion mud, Environmental Compatibility, Economic viability.

1 Introduction

Shale instability is a major cause of wellbore problems in drilling operations. According to Lal (1999), shale instability makes up over 70% of borehole problems. Shale instability to a considerable extent depends on the chemistry of the drilling fluids. Among other functions, drilling fluids are supposed to maintain wellbore stability, clean and transport cuttings out of the wellbore. Incompatibilities of drilling fluids and shale formations are often the root cause of shale instability. These incompatibilities can result in washouts, poor penetration rates, increased drilling cost, shale sloughing, borehole encroachment and other wellbore instability events. Although shale has many common properties, each shale formation has specific features in mineral composition, rock structures and deformation properties. The shale behaviour of fluid in one area or formation cannot always be extrapolated to another area or formation. Drilling fluid chemistry and exposure time have direct bearing on shale strength (Hemphil et.al. 2008)

When shale is exposed to fluids, it exhibits different behaviours such as fracturing, swelling and dispersion. Drilling fluids can cause shale instability by altering pore pressure or effective stress-state and shale strength through shale –fluid interaction (LaI 1999). To prevent wellbore instability drilling mud must be designed which can inhibit reactive shales, improper selection of either the weight or selection of the drilling fluid can cause wellbore instability, stuck pipe, low rate of penetration, difficulties in casing, logging and formation damage (Patil et.al.2016). The difference in shale fluid interaction is strongly related to its clay minerals, structure, bedding, thin laminae and pre-existing fractures (He et al.2014).

According to Likrama and Diaz (2015) causes of wellbore instability in shale may be separated into mechanical and chemical categories stating that mechanical instability results from mechanical failure of weak rock formations owing to the stresses in the vicinity of the wellbore. On the other hand, chemical instability results from the chemical reaction between water-based drilling fluids and clays present in the shale rocks which in turn cause swelling, weakening and destabilisation of the wellbore. Chemical-induced shale instability due to the drilling fluid-shale interaction alters shale strength as well as the pore pressure in the vicinity of the borehole walls (Azar and Samuel 2007). Wellbore stability is not an issue for most shale formations with oil-based and synthetic-based drilling fluids. As high cost and environmental restrictions have largely limited the use of oil-based mud (He.et al. 2014), shale-fluid interaction is investigated in Jatropha oil-based invert emulsion as one of the alternatives to diesel mud.

2 Materials and Method

The traditional laboratory methods such as dispersion test, bulk hardness test and swelling test cannot fully reflect the impacts of rock structure on fracture development and rock failing. Immersion test is one of the most efficient methods of evaluating fluid-rock interaction and fracture development (Gomez and He 2012). This study was conducted by immersion test. The shale samples used are the Marcellus shale which is black in colour. In order to replicate the real shale sample in a 3-D format, the computed tomography (CT) Scan was used to capture the real images of the samples before and after the immersion test. The schematic set-up of the CT scan is shown in Figure 1. CT Scan is a non-destructive technique using X-rays for visualising interior features within solid objects, and for obtaining digital information on their 3-D geometries and properties. A CT image is typically called a *slice*, as it corresponds to what the object being scanned would look like if it were sliced open along a plane. A CT slice corresponds to a certain thickness of the object being scanned. While a typical digital image is composed of pixels (picture elements), a CT slice image is composed of *voxels* (volume elements).

2.1 Choice of Shale Test Sample

Selection of a test sample depends on the availability and purpose of the tests. It is expected that under ideal conditions, correctly preserved shale core samples would give accurate results though such samples may not be readily available due to high cost of acquiring them. Shale cuttings taken from drilled formations can also be used for some tests. (Friedheim et al 2011). The core samples used in this study is the Marcellus shale which can be found in the Pennsylvania, New York, Ohio and West of Virginia. Marcellus extends more than 34,000,000 acres of real estate with at least 50 feet formation thickness which could contain 500 Tcf gas in place with estimates of recoverable gas at 50Tcf. (Belvalkar and Oyewole 2010). Like any other shale, Marcellus have low porosity and permeability. From Marcellus drilling news, drillers pointed out that Marcellus is advantageous over other shales stating that it is shallower thereby taking less time and money to drill. The news further stated that its location in the Pennsylvania is favourable as a state without a severance tax and with reasonable regulations.

2.2 Preparation of Test Sample

The test samples were cut into sizes of 37.5 mm diameter with a thickness of 3.1 mm. The samples were weighed and conditioned in the oven at 50°C for 24 hours. The initial weight was 14.83g. At the end of 24hours in the oven, the samples were re-weighed three times giving an average weight of 14.28kg. The same sizes of samples were also conditioned in the same manner for the 7 days test. There were six test samples for in all for the 1-day and 7-day immersion tests in the three mud samples- water-based, diesel and jatropha oil-based muds. This was to avoid immersion of a particular sample more than once into a mud sample. The test sample replicated in a 3-D using the CT scan volume analysis was 3502.21 mm^3.

2.3 Immersion Test.

Three mud samples: Water–based mud, 50/50 oil-water ratio diesel oil and jatropha oil invert emulsion muds were formulated to evaluate the interaction between the shale samples and the muds. Following the drying of the samples, they were scanned using the computed tomography (CT) Scan shown in Figure 1 to capture the real images of the samples before the immersion test. The shale samples were immersed into the formulated muds and were observed for 1- day and 7- days respectively. At the end of each observation period, each shale sample was re-scanned to check for any fractures, enlargement and extension of any existing fractures. Figure 2 represents the original test sample before the 3D representation with the CT scan.

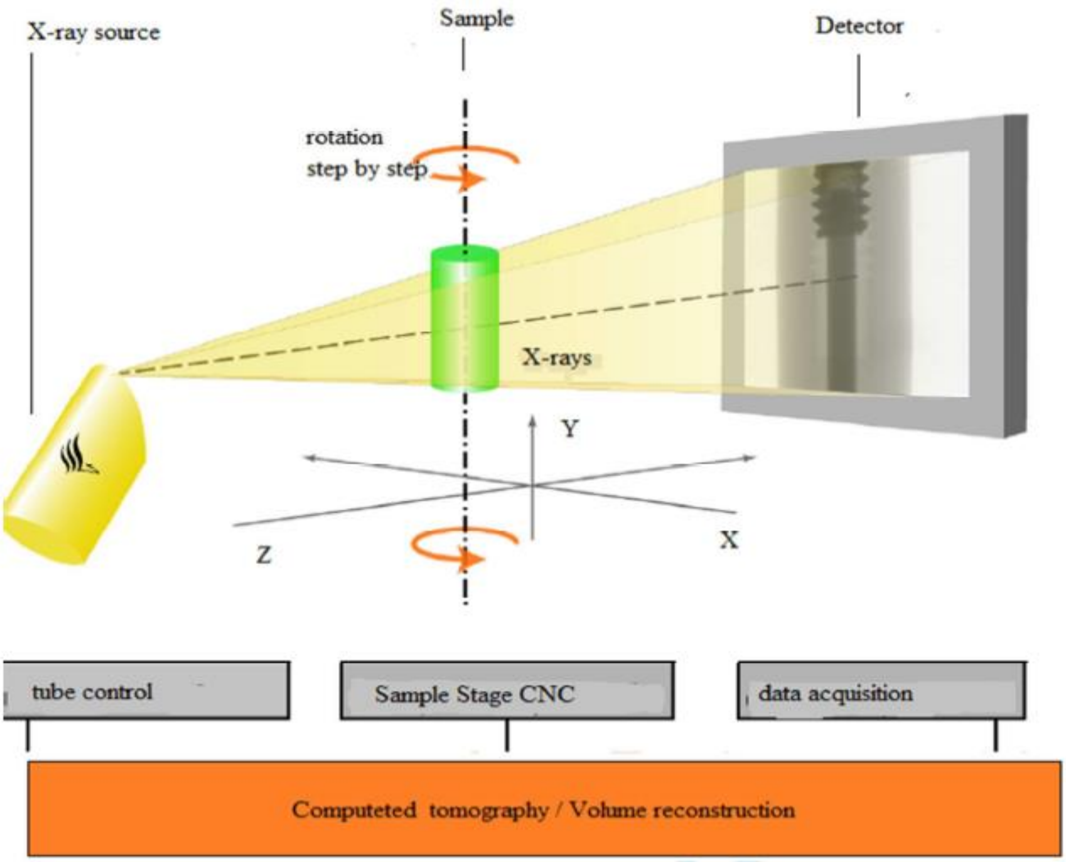

Fig. 1 Schematic Set-up of CT Scan Data Acquisition Process

Fig. 2 Original shale test sample before 3D representation on CT scan.

The images captured on the CT scan were analysed using Volume Graphics (VG) Studio Max 2.2 software. VG Studio Max is a software package for the visualisation and analysis of voxel data. It is used in a variety of application areas such as industrial CT, medical research, life sciences, animation and many others. VG Studio Max 2.2 has many functions including volume analysis, wall thickness analysis, porosity/inclusion analysis, coordinate measurement and CT reconstruction. In this study, the volume analysis was carried out. The shale samples were scanned before the immersion test as shown in Figure 3. The volume analysis of the sample size before the immersion in the mud was 3502.21mm^3.

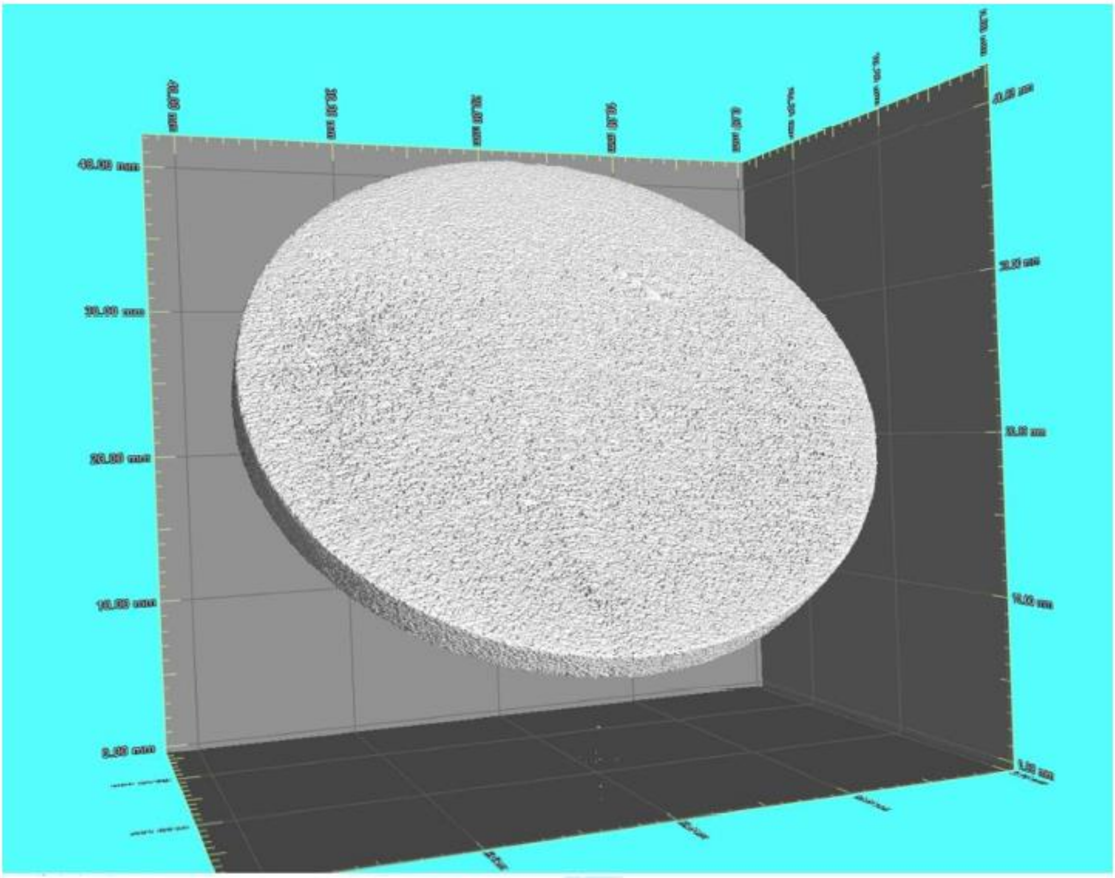

Fig. 3 Shale core sample before immersion in mud

3 Results and Discussions

Shale-fluid interaction using the immersion test remains one of the most efficient methods of evaluation of fluid-rock interaction and fracture development. Interactions between shale and drilling mud and their consequences on wellbore stability were evaluated. This was undertaken over a day and 7-days period of immersion in water based mud, 50/50 oil-water ratio diesel and jatropha oil- based muds.

Figure 4 represents the image from the 1-day immersion test in water-based mud. As shale is heterogeneous in nature, the colour patches indicate the different interaction of the core sample on immersion in the mud. However, the focus of this study is on the level of shale interaction with the mud. The volume analysis after 1-day immersion was 3872.35mm^3

compared to the initial volume of 3502.21 mm^3 indicating an increase in volume by 370.14mm^3. Figure 5 represents the 7-day immersion showing significant interaction with the mud as reflected in the increase in volume by 803.98mm^3. The volume of the core sample was 4306.19mm^3 compared to the initial volume of 3502.21 mm^3. Significant development of fractures and increase in volume is not conducive for wellbore stability.

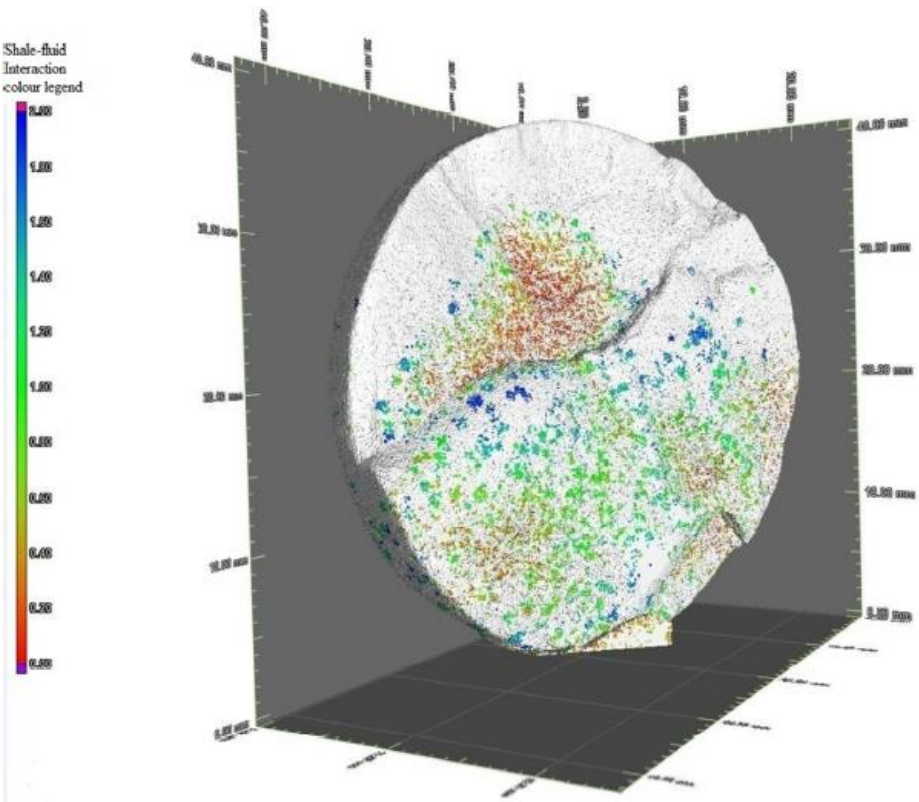

Fig. 4 1-day immersion in Water- Based Mud

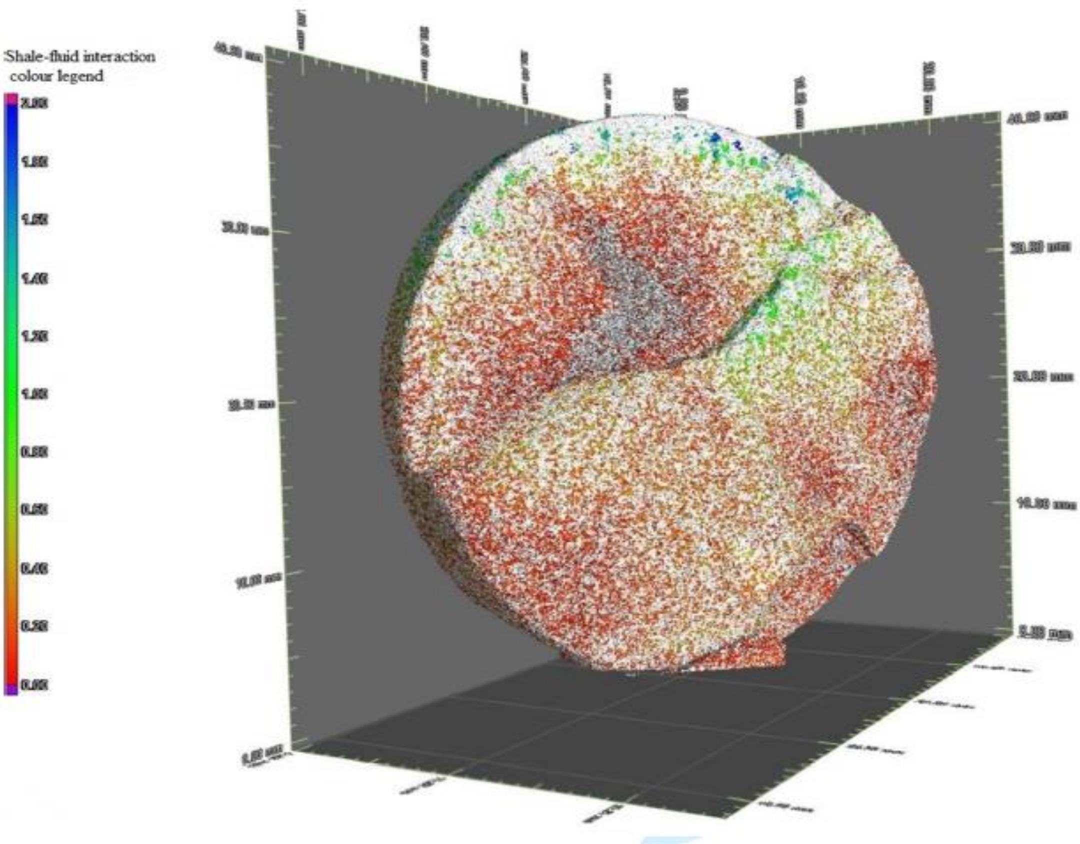

Fig. 5 7- day Immersion in Water Based-Mud

Comparatively, the 1-day and 7days immersion in diesel and Jatropha oil-based muds exhibited no enlargement or development of new fractures in the core sample as shown in Figure 6. The level of interaction with the mud was low with a volume of 3533.16mm^3 and 3590.75mm^3 at the end of 1-day and 7-day immersion respectively compared to the initial volume of 3502.21mm^3 before the immersion as shown in the percentage variation in volume in Table 1. The volume increase was 0.88% (30.95mm^3) and 2.53% (88.54mm^3) compared to 11% (370.14mm^3) and 23% (803.98mm^3) obtained in the water-based mud graphically represented in Figure 7. There was no variation in appearance of shale exposed to diesel oil based mud and jatropha oil based mud. This indicates that Jatropha oil-based mud could serve as a substitute to diesel oil based mud while drilling shale formations as it is relatively friendly to the environment and will not hydrate shale.

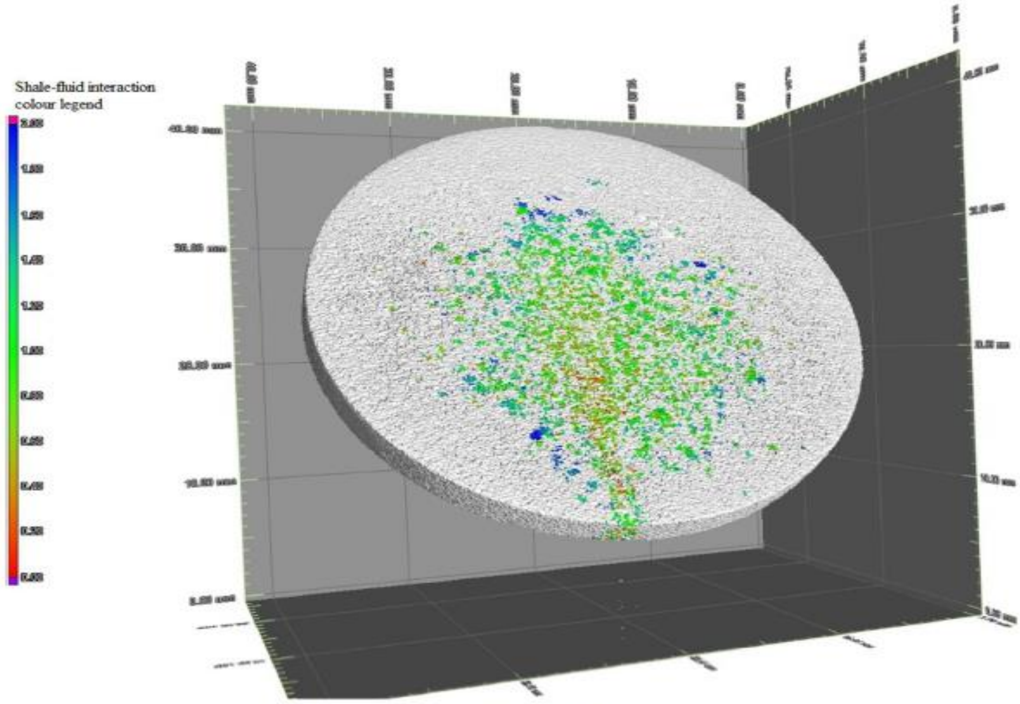

Fig. 6 1day and 7 days immersion in Diesel and Jatropha oil-based Muds.

Table 1 Percentage Variation in Volume Distribution of Test Core Samples

Mud Samples	Test Core Sample (mm^3)	1-Day Immersion (mm^3)	Percentage change in Volume (%)	7-Day Immersion (mm^3)	Percentage change in Volume (%)
Water-Based Mud	3502.21	3872.35	11.0	4306.19	23.0
Jatropha Oil-Based Mud	3502.21	3533.16	0.88	3590.75	2.53
Diesel Oil-Based Mud	3502.21	3533.16	0.88	3590.75	2.53

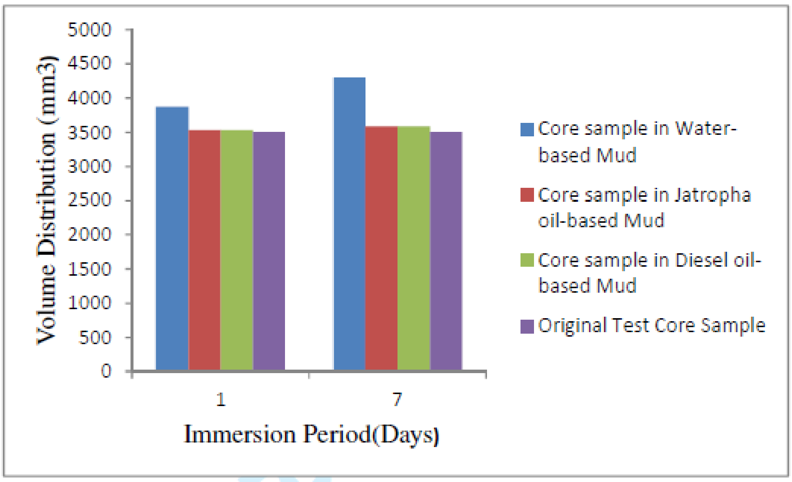

Fig. 7 Comparison of Volume distribution **of** the Core sample in the mud samples

4 Conclusions

Wellbore instability is a prevalent problem in shale formation. Incompatibilities with drilling fluids and shale formations are often the root cause of shale instability. Observation of the behaviour of the samples in the fluids and the post analyses suggest areas where the samples tend to fail. These areas could be related with the presence of clay-rich beddings, thin laminated structures, pre-existing fractures and other natural features in the formation. Fracturing along the bedding planes and laminae was dominant when the shale was exposed to water based mud. There was significant fracture development after 7 days in the shale sample exposed to the water based mud indicating high interaction with the shale sample which is not good for wellbore stability. However, this may be minimised with appropriate chemical inhibitors. There was no enlargement or development of new fractures in diesel and jatropha oil based muds diesel. There was no variation in appearance of shale exposed to diesel oil based mud and jatropha oil based mud. Considering the economic viability of Jaropha oil over diesel oil, this further suggests that Jatropha oil can be used as diesel alternative for a relatively environmentally friendly invert emulsion mud suitable for drilling shale formations while maintaining shale and wellbore stability

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Appendix B

The Table generated for the variation of rheological properties with electrical stability at various temperatures

B1: Plastic Viscosity variation with Electrical Stability at Various Temperatures

B2: Yield Point variation with Electrical Stability at Various Temperatures

B3: Apparent Viscosity variation with Electrical Stability at Various Temperatures**.**

B1: Effect of Electrical Stability on Plastic Viscosity

Temperature (ºC)	Electrical Stability **(V)** Mud Emulsified with Egg yolk	Plastic Viscosity (cP)	Electrical Stability (V) Mud Emulsified with VersacleanVB	Plastic Viscosity (cP)
50	353	52	368	52
60	328	51	348	50
100	278	21	305	20
120	258	12	273	14

B2: Effect of Electrical Stability on Yield Point

Temperature (ºC)	Electrical Stability **(V)** Mud Emulsified with Egg yolk	Yield Point (Ib/100 ft^2)	Electrical Stability (V) Mud Emulsified with VersacleanVB	Yield Point (Ib/100 ft^2)
50	353	69	368	52
60	328	58	348	50
100	278	25	305	20
120	258	22	273	14

B3: Effect of Electrical Stability on Apparent Viscosity

Temperature (ºC)	Electrical Stability **(V)** Mud Emulsified with Egg yolk	Apparent Viscosity (cP)	Electrical Stability (V) Mud Emulsified with VersacleanVB	Apparent Viscosity (cP)
50	353	86.5	368	84
60	328	80	348	76.5
100	278	32	305	31
120	258	24.5	273	23.5

Appendix C

Composition of Water-Based Mud used for Shale-fluid Interaction

C1: Composition of Water Based Mud

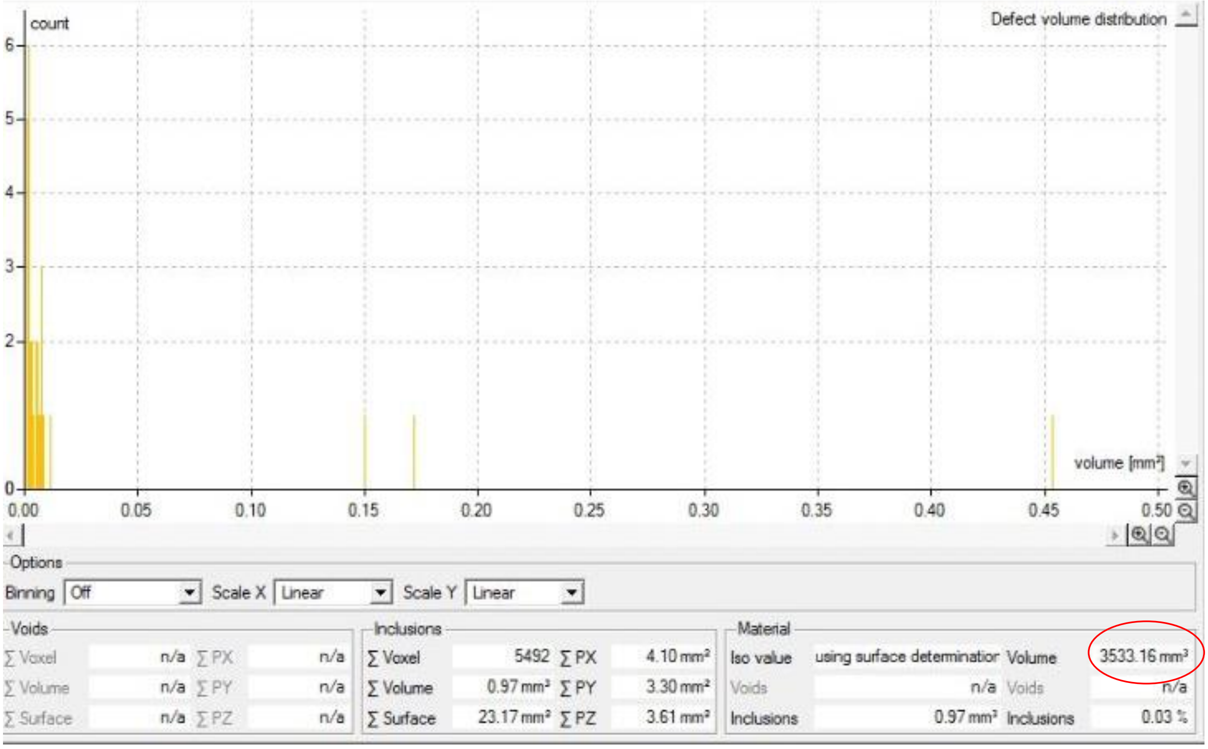
Component	Quantity
Water	350 ml
Bentonite	22.5g
NaOH	5 ml

Appendix D

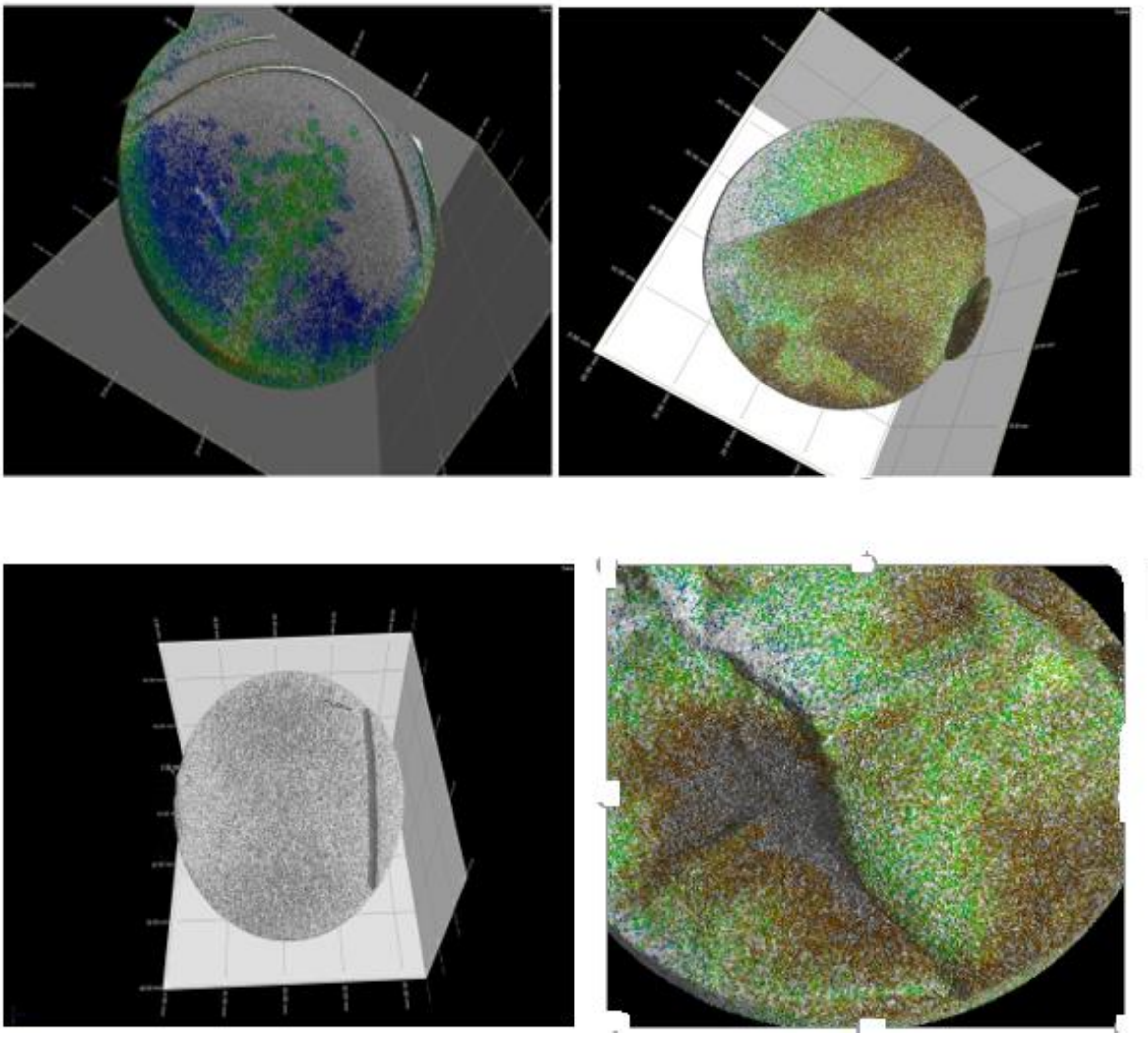
Sample of images with defects on shale-fluid interaction

D1: Defect Volume Distribution for One-Day Immersion in Jatropha and Diesel Oil-Based Muds

D2: Sample of Defective Images of One to Seven Days Immersion



D1: Defect Volume Distribution For One-Day Immersion in Jatropha and Diesel Oil-Based Muds



D2: Sample of Defective Images of One to Seven Days Immersion