The Effect of Lime and Primary Emulsifier on Rheological Behaviour of Palm Fatty Acid Distillate (PFAD)-based Drilling Fluid

by

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CERTIFICATION OF APPROVAL

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(PETROLEUM ENGINEERING)

Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the originality work is my own except as specified in the references and acknowledgements, and the original work contained herein have not been undertaken by unspecified sources or persons.

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(RAIHANA BT. RADZLAN)

ABSTRACT

This project in its present form is the proposal to scrutinize on the development of eco-friendly drilling fluid by using Palm Fatty Acid Distillate (PFAD) as a based in oil-based mud. The initial idea was to analyse the characterization of biodiesel (PFAD) as continuous phase in drilling fluid. Rheology test and mud tests were then conducted for PFAD-based drilling fluid and conventional oil-based mud (mineral diesel), which will lead to the justification of the PFAD-based drilling fluid adaptability level in replacing the conventional oil-based mud. The PFAD-based drilling fluid is then will be tested with different weight percentage of drilling fluid additive in order to study the effect of each additive on its behaviour.

The contributions of this project are twofold. This project is not only proposed potential alternative that preserves oil-based mud advantages, but also promoting eco-friendly project by using biodiesel as a based.

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CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

Oil based-drilling fluids are widely used in drilling, especially in highly technical and challenging wells because it performs better than water-based mud. Oil-based drilling mud provides good wellbore stability, good lubrication that leads to faster rate of penetration, temperature stability, reduced risk of differential sticking and low formation damage. However, the disposal of oil-contaminated drill cuttings causes environmental hazard. The industry has been replacing highly aromatic oils (e.g. diesel) with low aromatic mineral oils. Nevertheless, as environmental legislation and controls become more stringent, even the newer and less polluting mineral and synthetic oils in vogue now may be adjudged unsuitable because of their non-biodegradability. Indeed, today, in many parts of the world like the USA, United Kingdom, Holland, Norway, Nigeria and Australia, the use of diesel and mineral oil-based drilling fluids in offshore operations is already either severely restricted or banned because of their toxicity, persistency and bioaccumulation. (Dosunmu, 2010)

It is undeniable environmental protection is very important worldwide. Hence, many operators around the globe are becoming more conscious of the impact that their exploration and production activities have on the environment. In Asia, this trend is catching on. Many Asian governments are also beginning to impose tighter environmental regulations for operating companies to comply with both on- and offshore. With the establishment of these corporate and legislative standpoints, Drilling and HSE engineers and advisors in Asia are under greater pressure. Efficient and environmentally friendly ways to use non-damaging drilling fluids as well as to reduce cuttings and dispose of the waste need to be find. (Global, 2013)

1.2 PROBLEM STATEMENT

A typical well may generate between 1000 and 1500 tonnes of cuttings. With average oil retention of 15%, around 150-225 tonnes of oil from the drilling fluid is discharged into the sea for each well that is drilled, thereby causing a large area around the drilling site being affected. The disposal of the oil-contaminated drill cuttings raises a concern over the ecological impact on marine life (Dosunmu, 2010). It is undeniable environmental protection is very important worldwide. Therefore, research has been conducted to find alternatives that can replace the conventional oil-based mud while preserving it advantages. Hence, to satisfy both the environmental and technical criteria, the industry is recognized the potential of biodiesel-based mud. In this paper, PFAD-based drilling fluids are developed and the effect of lime and primary emulsifier on it will be focused.

1.3 OBJECTIVES & SCOPE OF STUDY

The objective of this project is to analyse the rheological behaviour of biodiesel-based drilling fluid. Hence, a new eco-friendly drilling fluid can be proposed. The objective can be subdivided as below;

Table 1: Objective and Scope of study

Objective	Scope of study
Characterization of biodiesel as continuous phase in drilling fluid	• Identify the physical characteristic of biodiesel and bio-crude oil. • Examine the characteristic of both types of oil and their blending.
Rheological behaviour of biodiesel-based drilling fluid	• Examine and comparing the rheological properties of biodiesel drilling fluids with conventional oil-based drilling fluid.
The effect of lime and primary emulsifier on the biodiesel-based drilling fluid	• Examine the behaviour of biodiesel-based drilling fluid with different amount of lime and primary emulsifier.

To achieve this, the formulated of bio-crude and biodiesel-based drilling fluids is compared with the conventional oil-based mud from Scomi Oiltools Bhd.

1.3.1 HYPOTHESIS

- The API filtration of the biodiesel-based mud meets the requirements of field application.
- The rheological parameters of biodiesel-based mud are feasible to replace the conventional oil-based mud.
- Less amount of lime and primary emulsifier will yield a better result for biodiesel-based mud.

1.4 RELEVANCY OF THE STUDY

This project is relevant to the author's field of study and also majoring since study on drilling fluids and its characteristics are one of the vital areas in drilling engineering course. Replacing the conventional oil-based mud continuous phase with more eco-friendly product, either palm oil or rubber seed oil which may satisfy both the environmental and technical criteria. Environmentally as it is biodegradable and technically as it has potential in replacing conventional continuous phase performance (mineral diesel). In addition, rheological studies are the key element in preparing circulating system especially for a new proposed element. Plus, palm oil and rubber seed oil which are from a renewable source also meet the requirement of strategy of sustainable product.

1.5 FEASIBILITY OF THE PROJECT WITHIN THE SCOPE AND TIME FRAME

In order to complete this project, the project task has been divided into three phase within the time frame. The first phase is to characterize the bio-crude, biodiesel as well as the blending of both oil. Secondly, the rheological test and mud tests were conducted to examine the rheological behaviour of biodiesel-based drilling fluid. Thirdly, the effect of lime and primary emulsifier on the biodiesel-based drilling fluid will be tested. Those aspects will be compared with the commercial conventional oil-based mud. The first and second phase is expected to complete before the third week of FYP II and the third phase followed weeks after that.

CHAPTER 2: LITERATURE REVIEW

This chapter will be focusing on all the elements that are going to be taking considered in order to ensure the efficiency and effectiveness of the project flow.

2.1 DRILLING FLUID HISTORY DEVELOPMENT

Drilling fluids or mud is any fluid that is used in a drilling operation in which that fluid is circulated or pumped from the surface, down the drill string, through the bit and back to the surface via the annulus. Drilling fluids also represent till one fifth (15 to 18%) of the total cost of well petroleum drilling, must generally comply with three important requirements (Mohamed Khodja, 2010);

i) Easy to be used,

ii) Not too expensive and

iii) Eco-friendly.

The complex drilling fluids play several functions simultaneously. They are intended to clean the well, hold the cuttings in suspension, prevent caving, ensure the tightness of the well wall, flood diesel oil or water and form an impermeable cake near the wellbore area. Moreover, they also have to cool and lubricate the tool, transfer the hydraulic power and carry information about the nature of the drilled formation by raising the cuttings from the bottom to the surface.

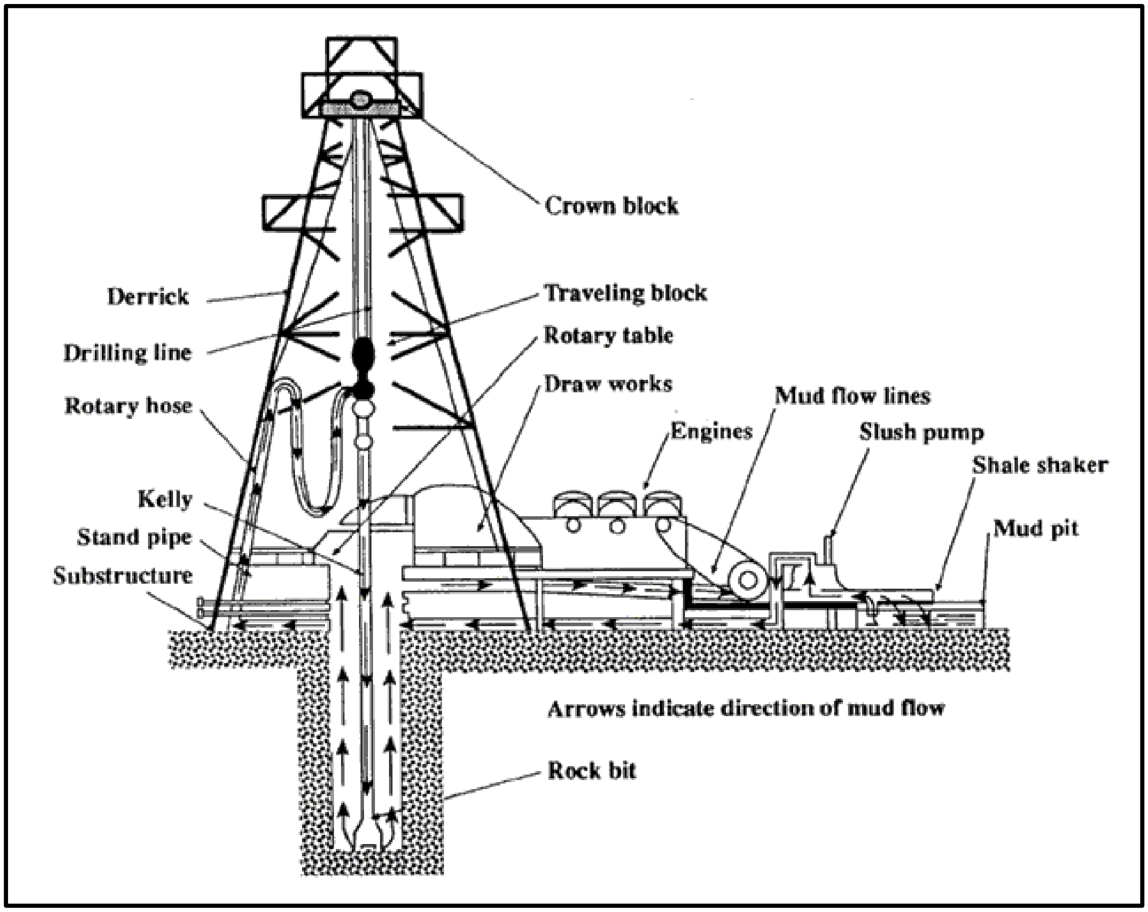


Figure 1: Simple diagram of rotary rig (Drilling Rig, 2004)

Drilling fluids went through major technological evolution, since the first operations performed in the US, using a simple mixture of water and clays, to complex mixtures of various specific organic and inorganic products used nowadays. These products improve fluid rheological properties and filtration capability, allowing penetrating heterogeneous geological formations under the best conditions (Mohamed Khodja, 2010).

2.2 DRILLING FLUID CLASSIFICATION

Drilling fluid can be classified into two categories which include;

- The water-based mud
- The oil-based mud

2.2.1WATER-BASED MUD

Water-based drilling fluids or mud (WBMs) use water or brine as the continuous or external phase with the critical functions (density, viscosity, filtration, lubricity, etc.) achieved by addition of various materials. Water based fluids are the most extensively used drilling fluids. They are easy to build an inexpensive to maintain. Three major sub-classifications of water-based drilling fluid include;

Table 2: Type of WBM

Type of WBM	Description
Non-Inhibitive fluids	These types of fluids do not suppress clay swelling
Inhibitive fluids	These fluid types appreciably retard clay swelling and achieve inhibition in the presence of cations typically, Sodium (Na^+), Calcium (Ca^{2+}) and Potassium (K^+).
Polymer fluids	Fluids that rely on macromolecules, either with or without clay interactions to provide mud properties and are applied in diverse forms. These fluids can be inhibitive or non-inhibitive depending on the type of cation used. (Amoco Production Company Driiling Fluid Manual, 1994)

However, there are some of the problems created by water-based mud which include hole enlargement, bit balling, accretion, low rates of penetration and insufficient hole cleaning. (Billy, et al., 2007).

2.2.2 OIL-BASED MUD

The solids in an oil base fluid are oil wet, all additives are oil dispersible and the filtrate of the mud is oil. The water, if present, is emulsified in the oil phase. There are two basic classifications of oil-based fluids;

1) All-oil mud
2) Invert emulsions

The amount of water present will describe the type of oil base fluid. The oil used in these types of oil base fluids can range from crude oil, refined oils such as diesel or mineral oils, or the non-petroleum organic fluids that are currently available. The latter type fluids variously called inert fluids, pseudo oils, oil-based fluids and synthetic fluids are now considered more environmentally acceptable than diesel or mineral oils. Invert emulsions are oil mud that is formulated to contain moderate to high concentrations of water (Amoco Production Company Driiling Fluid Manual, 1994). Oil-based drilling mud and synthetic-based drilling mud have many inherit advantages over water-based drilling fluids including temperature stability, tolerance contamination and corrosion protection (Dye, et al., 2006) and according to the Norwegian Oil Industry Association Working Group (1996).

Oil mud offers many advantages over water-based mud. The high initial cost of the oil-based mud can be a factor in not selecting this type of mud system. However, if the overall drilling costs are considered, the costs accompanying the use of an oil mud are usually less than that for water.

2.3 FUNCTIONS OF DRILLING FLUID

The primary functions of drilling fluid can be subdivided into several functions, which are;

2.3.1 REMOVE CUTTINGS FROM WELLBORE

Cuttings from drill bit must be transported to the surface. Failing in transportation will causes the drilling efficiency decreases. Therefore, mud must be designed such that it can;

- Carry cuttings to the surface while circulating
- Suspend the cuttings while no circulating

2.3.2 COOL AND LUBRICATE THE BIT

The rock cutting process will generate a great deal of heat at the bit. The overheat condition will then lead to quickly wear out. Nevertheless, this problem can be avoided by cooling the bit. This circulation of mud will help to cool the bit down and lubricate the cutting process.

2.3.3 PROTECT THE WALL OF THE WELLBORE

The mud has to seal off the permeable formations to avoid damages. It will form a thin impermeable mud cake (or known as filter cake) at the borehole wall. The cake should not be too thick, otherwise it may cause stuck pipe. In addition, the mud cake also protects the borehole from caving-in.

2.3.4 PREVENT FORMATION FLUID FLOWING INTO THE WELLBORE

The mud is designed to create an overbalanced drilling condition. Hydrostatic pressure exerted by the mud column should be slightly higher than the formation pressure. If not, an influx of formation fluids into the wellbore will occur. However, if the hydrostatic pressure is too high, it will fracture the formation and causes loss of circulation. The mud can sometimes seep through the filter cake and into the formation (filtrate). The lost mud and filtrate can cause solid deposition and clay hydration in the pore space which then lead to reducing permeability.

2.3.5 DATA LOGGING

The drilling fluid characteristics need to be controlled, which requires accurate information about the well and formations being drilled. The information obtained from logs and cores depends mainly on the filtration properties of the mud. Distortion of formation due to thick filter cake can make difficulties in logging operation.

2.4 MUD PROPERTIES

It is compulsory to specify not only the type of drilling mud to be used for each hole interval to be drilled but also the properties of such mud. These are the density, flow properties or rheology, filtration and solid contents as well as chemical properties. To avoid costly drilling problem, these properties must be field controlled and properly maintained at their preselected values. For this reasons, it is essential to monitor any changes by conducting field tests and thereby determining the cause of any problem and finding solution. Here, emphasis is placed on the definition and functions of mud properties.

2.4.1 DENSITY (MUD WEIGHT)

The term weight is used in connection with mud more often than density, even though density is the more correct right term. Ideally, a mud weight as low as the weight of water is desired, for optimum drilling rates and for maximizing the chances of fracturing the formation. However, in practice, mud weights in excess of two times the weight of water may be necessary to contain abnormal pressures or to mechanically stabilize unstable formations. (Jamal J. Azar, 2007). To summarize, mud weight depends on the type of the formation to be drilled.

2.4.2 VISCOSITY

Viscosity is a measured of liquid's resistance to flow. For drilling fluids, there are 3 parameters measured;

- Funnel viscosity (sec/qt)
- Yield Point (lbs/100 ft^2)
- Plastic Viscosity (cp)

Those above properties are measured by using Marsh funnel and as well as Multi-rate rotational viscometer. The measurement of the marsh funnel is used for comparison purposes. It only indicates if the viscosity has changed. Meanwhile, the yield point and plastic viscosity significantly can be summarized as table below;

Table 3: Significant comparison between Plastic Viscosity and Yield Point (Nasir, 2010)

Plastic Viscosity (cp)	Yield Point (lbs/100 ft^2)
▪ It depends on the friction between solids and liquid ▪ It represent the shear rate viscosities encountered at the drill bit ▪ A low PV indicates that the mud is capable of drilling rapidly because of the low viscosity of mud exiting at the bit. ▪ High PV is caused by a viscous base fluid and by excess colloidal solids. To lower PV, a reduction in solids content can be achieved by dilution.	▪ It is a measure of the attractive forces between active clay particles in the mud under flowing conditions ▪ It is used to evaluate the ability of a mud to lift cuttings out of annulus ▪ A higher YP implies that drilling fluid has ability to carry cuttings better than a fluid of similar density but lower YP. ▪ YP can be lowered by adding deflocculant and increased by adding flocculant

2.4.3 GEL STRENGTH

Gel strength denotes the thixotropic properties of the mud. It indicates:

- the pressure required to initiate flow after the mud has been static for sometime
- The suspension properties of the mud and hence its ability to suspend cuttings when the mud is stationary

Gels are described as strong or fragile. For a drilling fluid, the fragile gel is more desirable as the pressure required to initiate flow is smaller.

2.4.4 pH

Mud must always be treated to be alkaline (pH 7 – 9.5). If mud pH is above 9.5 (too alkaline) it will causes mud viscosities increases and shale instability occurs. In other hand, if mud pH is below 7 (acidic), corrosion problem will occur (which can be caused by CO_2 and H_2S) (Nasir, 2010).

2.5RHEOLOGY

Rheology refers to the deformation and flow behavior of all forms of matter. Certain rheological measurements made on fluids, such as viscosity, gel strength, yield point and etc. help determine how this fluid will flow under a variety of different conditions. This information is important in the design of circulating systems required to accomplish certain desired objectives in drilling operations.

2.5.1 VISCOSITY

THEORY

Viscosity is defined as the resistance of a fluid to flow and is measured as the ratio of the shearing stress to the rate of shearing strain. Two types of fluid characterizations are;

a) Newtonian (true fluids) where the ratio of shear stress to shear rate or viscosity is constant, e.g. water, light oils and etc.
b) Non-Newtonian (plastic fluids) where the viscosity is not constant, e.g. drilling mud, colloids etc.

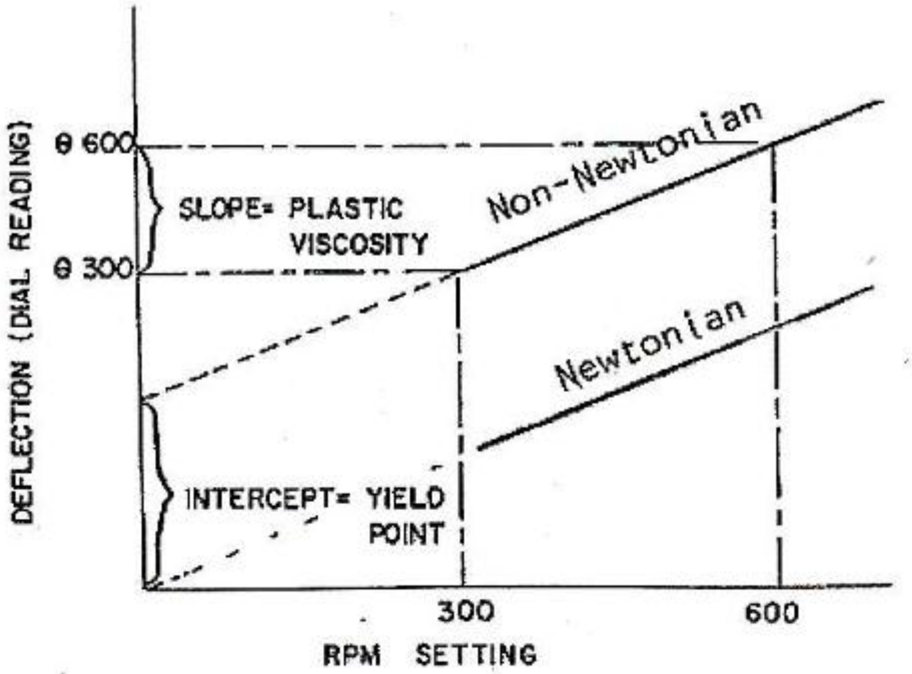


Figure 2: Flow Curves of Newtonian and Non-Newtonian fluids

2.5.2 GEL STRENGTH

THEORY

The Gel strength is a function of the inter-particle forces. An initial 10-second gel and a 10-minutes gel strength measurement give an indication of the amount of gelation that will occur after circulation ceased and the mud remains static. The more the mud gels during shutdown periods, the more pump pressure will be required to initiate circulation again.

Most drilling mud is either colloids or emulsions which behave as plastic or non-Newtonian fluids. The flow characteristics of these differ from those of Newtonian fluids (i.e. water, light oils, etc.) in that their viscosity is not constant but varied with the rate of shear. Therefore, the viscosity of plastic fluid will depend on the rate of shear at which the measurements were taken.

2.5.3 YIELD POINT

THEORY

This is the measure of the electro-chemical or attractive forces in the mud under flow (dynamic) conditions. These forces depend on;

a) Surface properties of the mud solids
b) Volume concentrations of the solids
c) Electrical environment of the solids

The yield point of the mud reflects its ability to carry drilled cuttings out of the hole.

Measurement

YP = 300 RPM – Plastic Viscosity

2.5.4 FILTRATION

THEORY

The loss of liquid from a mud due to filtration is controlled by the filter cake formed of the solid constituents in the drilling fluid. The test in the laboratory consists of measuring the volume of liquid forced through the mud cake into the formation drilled in a 30 minute period under given pressure and temperature using a standard size cell. It has been found in early work that the volume of fluid lost is roughly proportional to the square root of the time for filtration.

$$V \propto \sqrt{t}$$

The two commonly determined filtration rates are the low pressure, low temperature and the high pressure high temperature.

2.3 BIODIESEL

Mineral oil-based drilling mud is toxic, not readily biodegradable and thus has cumulative impact on the terrestrial, coastal and marine habitats. The base fluids for mineral oil-based mud development (usually diesel) have limited source of supply. In addition, their use is subjected to more and more constrains due to increasing evolution of environmental legislations.

One of the ways to avoid these problems while keeping the advantages of oil-based mud is to substitute diesel in mud with vegetable or animal oils. In Nigeria today, the environmental acceptance of a non-water soluble drilling mud base fluid depends not only on its toxicity as measured from traditional bio-assays, but also on its biodegradability under aerobic and anaerobic conditions. (Fadairo, Tozunku, Kadiri, & Falode O.A, 2012)

2.3.1 DEFINITION

Biodiesel refers to a vegetable oil- or animal fat-based diesel fuel consisting of long-chain alkyl (methyl, propyl or ethyl) esters. Biodiesel is typically made by chemically reacting lipids (e.g., vegetable oil, animal fat) with an alcohol producing fatty acid esters. (Biodiesel, 2013).

Biodiesel is synthesized by interesterification. Oil crops, wild-oil bearing crops, engineering micro algae, animal fats and hogwash oil all can be the raw material of the interestification. (Wang, et al., 2012)

2.3.2 PROPERTIES

Biodiesel is renewable and can replace mineral diesel. The main component is fatty acid methyl-ester (FAME). The characteristics of biodiesel are as following (Wang, et al., 2012);

- Environmentally friendly: low sulfur content, without aromatic alkane, easily biodegradable.
- Good safety performance because flash point is high, biodiesel is not hazardous article
- Biodiesel can renewable which meet the requirements of the strategy of sustainable development.

2.3.3 CURRENT RESEARCH

Vegetable oils are becoming a promising alternative to diesel fuel because they are renewable in nature and can be produced locally and environmental friendly as well. They have practically no sulphur content, offer no storage difficulty, and they have excellent lubrication properties. Moreover, vegetable oils yielding trees absorb more carbon dioxide from the atmosphere during their photosynthesis than they add to the atmosphere on burning. Hence, they essentially help to alleviate the increasing carbon dioxide content in the atmosphere. The substitution of diesel oil by renewable fuels produced within the country generates higher foreign exchange savings, even for the major oil exporting countries. Therefore, developing countries can use this kind of project not only to solve their ecological problems but also to improve their economy. In view of the several advantages vegetable oils has potential to replace petroleum-based fuels in the long run (Ramadhas, Jayaraj, & Muraleedharan, 2005).

Some of the on-going research into finding more suitable crops and improving oil yield especially in replacing the conventional oil based mud with biodiesel are;

JATROPHA

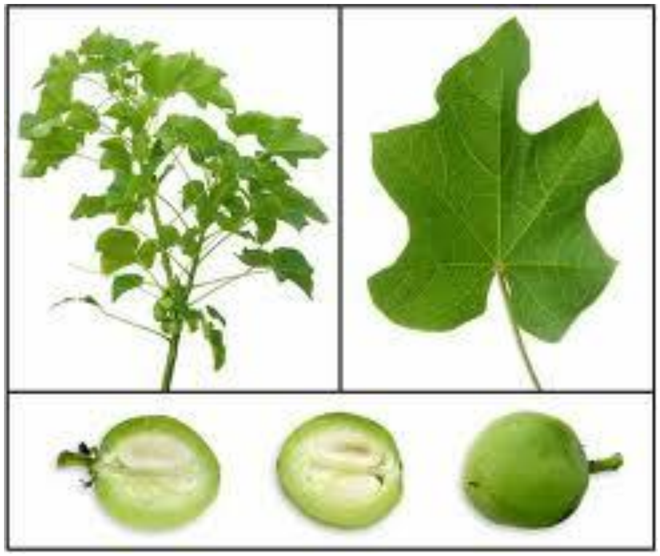


Figure 3: Jatropha plant and seed (Intelligentsia International, 2008)

Jatropha is a type of plant that comes from the family Euphorbiacea. The name Jatropha comes from Greek which is “Jatras” that mean Doctor and “Thrope” that means Nutrition. First is native plant in Central America and now it has been produced in other subtropical areas such as India, Africa and North America. Originally the Jatropha comes from Caribbean; however Portuguese traders have brought the Jatropha out to Africa and Asia. The Jatropha has a really advantageous point where it is resistant to drought and pests and has a seeds that contain up to 30-40% of oil content. The seed is usually crushed in order to extract the oil to be used as biodiesel and the remaining will be used as biomass for powering electricity. (Jatropha Cultivation, Production, Properties and Uses, 2010)

There is extensive research on oil from Jatropha seed in India. The seed contains high number of oil which is about 33-60%. The oil extracted from mechanical ways is processed and can be used as biodiesel for running diesel engine. The government of India has fixed the biodiesel price as Rs. 25 per liter. (Abdulla, 2008).

There is a study from Covenant University, Nigeria which focused on environmental safe drilling mud using this plant seed called jatropha. The oil was extracted from the jatropha seed and added to mud samples to study its stability for drilling operation as well as its toxicity, filtration, pH, viscosity, density and degree of safety to the environment. (Adesina, Anthony , Gbadesign , Eseoghene, & Oyakhire, 2012)

Based on the latest research that has been conducted, it has been found out that jatropha oil-based mud (JOBM) has an undesirably high apparent viscosity at ambient temperature. This is as a result of the inherently high viscosity of the base fluid-jatropha oil. In addition, temperature and salinity give a negative impact on the rheological properties of oil-based drilling fluids. However, JOBM showing better adaptability under these condition. Plus, JOBM also exhibit better results for pH and density variation with temperature (Fadairo, Tozunku, Kadiri, & Falode O.A, 2012).

PALM OIL AND GROUNDNUT-OIL

Figure 4: Left side shows the palm oil seed and the right side shows the groundnut (Palm Oil, 2005) (Groundnut Oil, 2006)

The capability for both vegetable oils derived from palm oil and groundnut oil in developing environmentally friendly oil based mud is examined. Rheological test is conducted between these biodiesel-based muds with the conventional oil-based mud. The comparisons established are then lead into several conclusions, which are;

- Palm oil and experimental oil based mud are very viscous, with the palm oil based mud demonstrating strong progressive gel characteristics before hot rolling (virgin mud).
- It presented rheological readings acceptable for a virgin mud before hot rolling.
- After hot rolling for 16 hours at 250oF (aging mud), the experimental oil based mud become highly viscous and failed to give any reading on the rheometer. It exhibited significant thermal degradation. It also shows that the fatty acids components of the oil broken down.
- Toxicity of diesel, palm oil and groundnut oil were compared by exposing corns planted on humus soil beds prepared with palm. It has been found that as the corns is exposed to diesel, it lost its greenness and died. Meanwhile, as they are exposed to the palm oil and groundnut oil, their greenness retain. This can be concluded that palm oil and groundnut oil have better eco-toxicological properties.

However, the preliminary tests indicate that additive chemistry must be employed in the formulation of the vegetable oil-based mud in order to make them very functional in a drilling operation (Dosunmu, 2010).

2.4 PROPOSED BIODIESEL

In this project, rubber seed oil, palm oil and PFAD are proposed as the biodiesel to be examined in order to replace the conventional oil-based mud.

2.4.1 RUBBER SEED

Figure 5: Rubber seed (Rubber Seed Oil, 2006)

Christopher Columbus is believed to have first found rubber in tropical South America around 1500. Hevea brasiliensis, the common variety of rubber tree produces 99% of world's natural rubber. The seed contains an oily endosperm. Generally 37% by weight of the seed is shell and the rest is kernel. The oil content of air-dried kernel is 47% (A Study on the Production of Biodiesel from Rubber Seed Oil (Hevea Brasiliensis), 2013).

In Malaysia, the flowering seasons of rubber are in March and August. The peak seed falls follows approximately six month later (Manual Teknologi Penanaman Getah , 2004). Rubber seed oil is a non-edible vegetable oil. The increase in the price of non-edible oil in recent years generated interest in the collection and processing of rubber seeds. According to a study conducted by the rubber board, on an average, a healthy tree can give about 500 g of useful seeds during a normal year and this works out to an estimated availability of 150 kg of seeds per hectare. The price of rubber seeds is around RM 3 per kg (Sue-Chern, 2012). Rubber seeds are produced mostly in North of Peninsular Malaysia (Perak, Perlis, and Kedah).

2.4.2 PHYSICO-CHEMICAL PROPERTIES OF RUBBER SEED OIL

Table 4: Physico-chemical Properties of Rubber Seed Oil (Srivastava, 2008)

Fuel property	Diesel Oil	Rubber seed oil (bio-crude)	ROME (biodiesel)
Density (gm/cc^3)	830	930	860
Specific gravity	0.830	0.930	0.860
Viscosity (cp)	3.55	66	6
Flash Point (^0C)	55	198	72
Calorific Value (MJ/Kg)	43	37.5	35

Based on the properties above has led to the justification of choosing rubber seed oil as the main base for replacing the conventional oil-based mud. The properties of both bio-crude and biodiesel rubber seed oil which are almost similar to diesel oil lead to the several phase of the testing for replacing the conventional oil-based mud. The test will begin by analyse the capability of the bio-crude oil in replacing the conventional as the alternative based for the oil-based mud. If the properties are not satisfying, it will later upgrade with the biodiesel. However, some additives need to be adding in order to offset the higher viscosity and also to ensure other properties while formulated the alternative oil-based drilling fluids meet the API Recommended Practice 13B-2.

2.4.3 PALM OIL

Palm oil also is used in this project. Based on the previous study, it has been known that palm oil will contribute to the high viscosity of mud. Therefore, in this research project, the palm oil used, PFAD (palm fatty acid distilled), will be blended with PO (Palm Olein) and also RSB (rubber seed biodiesel).

PFAD (palm fatty acid distillate) is a by-product of physical refining of crude palm oil products and is composed of free fatty acids (81.7%), glycerides (14.4%), squalene (0.8%), vitamin E (0.5%), sterols (0.4%) and other substances (2.2%). PFAD is used in the animal feed and laundry soap industries as well as a raw material for the oleo chemicals industry. Vitamin E, squalene and phytosterols are value-added products which could be extracted from PFAD and are of potential value for the nutraceutical and cosmetic industries (Ab Gapor Md Top, 2010).

2.4.2 PROPERTIES OF PALM OIL

Table 5: Properties of Palm Oil (Chempro, 2008)

Temperature (^oC)	Viscosity (cSt)	Heat Capacity (KJ/kg.^oC)	Conductivity (W/m.^oC)	Density (kg/m^3)
40	40.24	1.902	0.1708	880

2.4.3 FUEL PROPERTIES OF PFAD BIODIESEL

Table 6: Fuel Properties of PFAD Biodiesel (choo, 2007), (S.Chongkong, C.Tongurai, P.Chetpattananondh, & C.Bunyakan, 2007)

Properties	Unit	EN 14214:2003[1]		ASTM D6751:06[2]	
		Min	Max	Min	Max
Ester Content	% mass	96.5	-	-	-
Density at 15 ^OC	kg/m^3	860	900	870	900
Viscosity at 40 ^OC	cSt	3.5	5.0	1.9	6.0
Flash Point	^{o}C	120	-	130	-
Cloud Point	^{o}C	-	-	-3	12
Pour Point	^{o}C	-	-	-15	10

The requirements as stated in those standard (or/and) must be comply by prepared sample in order to determine the prepared sample is pass or fail as optimum or standardize biodiesel.

[1] European Standard For Biodiesel

[2] Standard Specification For Biodiesel Fuel (B100) Blend Stock For Distillate Fuels

2.5 MUD COMPOSITION (ADDITIVE) THAT WILL BE VARIED

In this project, there are two composition that will varied in the mud formulation. The effect of their changes on the trend behaviour of the drilling fluid/mud will be observed and analyse for optimization purpose.

2.5.1 PRIMARY EMULSIFIER

It has been known there are two main categories of mud which are water-based mud and oil-based mud. In oil-based mud, one of the most important chemical used is emulsifier. Emulsifier consists of two types which are primary and secondary emulsifier. The classification of both emulsifier can be summarize as below;

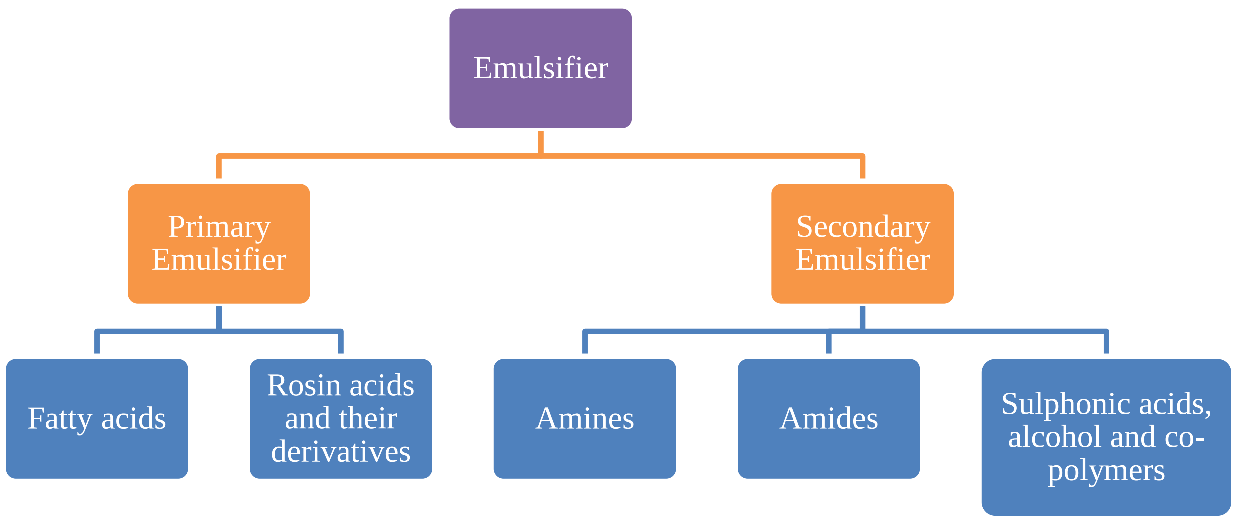


Figure 6: Classification of emulsifier

The function of primary emulsifier is to emulsify the water inside oil so that there is no free water in filtrate and the secondary emulsifier is the wetting agent. The efficiency of the emulsifier can be identify from the emulsion stability test using the electrical stability meter and from the filtration of the mud using the HTHP filter pressure (Muhammad, 2012).

2.5.2 LIME

Lime is one of the chemical additives used in the mud composition to control the mud properties. The function of lime in the drilling fluid/mud are (Energy, 2013);

- Alkalinity and pH Control

  Designed to control the degree of acidity or alkalinity of the drilling fluid.

- Bactericides

  Used to reduce the bacteria count

- Corrosion inhibitors

  Used to control the effects of oxygen and hydrogen sulphide corrosion.

- Flocculants

  There are used to cause the colloidal particles in suspension to form into bunches, causing solids to settle out. In addition, it contribute for mud thickening by stimulate the primary emulsifier.

CHAPTER 3: METHODOLOGY

This research is an experimental research. Various experiments to study the properties of biodiesel need to be conducted. In order to gain more understanding before conducting the experiments, the author did a case studies research by reading any related reading materials related to biodiesel. However, the information of using rubber seed oil and PFAD in oil and gas industry is limited.

The experiments in this research can be divided into three phases. The first phase of the experiments is to characterize the biodiesel as continuous phase in drilling fluid. The biodiesel used were Palm Fatty Acid Distillate (PFAD), Palm Olein (PO) and the blending of both oils (PFAD+PO). These experiment were conducted in the Bio-hydrogen Laboratory Research Centre, Block P, at Universiti Teknologi PETRONAS. The properties of each sample of oil was also examined in order to determine which mixing ratio is suitable to be proceed for the next phase. For instance, a stringent experiment in determining the oil sample's viscosity is conducted to obtain a reliable and accurate value. The viscosity experiment is repeated several time and conducted by using different equipment. The pour point, cloud point, flash point as well as density are also determined.

In the second section, the best sample which have meet the requirement and standard in first section, will be used in the mud preparation process as a base. This section will be conducted in the Scomi Oiltools Laboratory in Shah Alam. Details rheology properties such as low end rheology, High Temperature High Pressure fluid loss, Emulsion Stability reading, gel strength, plastic viscosity and yield point will be examined and tested.

The third section of the experiment is to study the properties of the previous biodiesel-based drilling fluid (2nd section) by changing the formulation of the drilling fluid itself (e.g.: lime, primary emulsifier). This experiment will be conducted completely in Scomi Oiltools Laboratory, Shah Alam.

3.1 PROJECT WORK

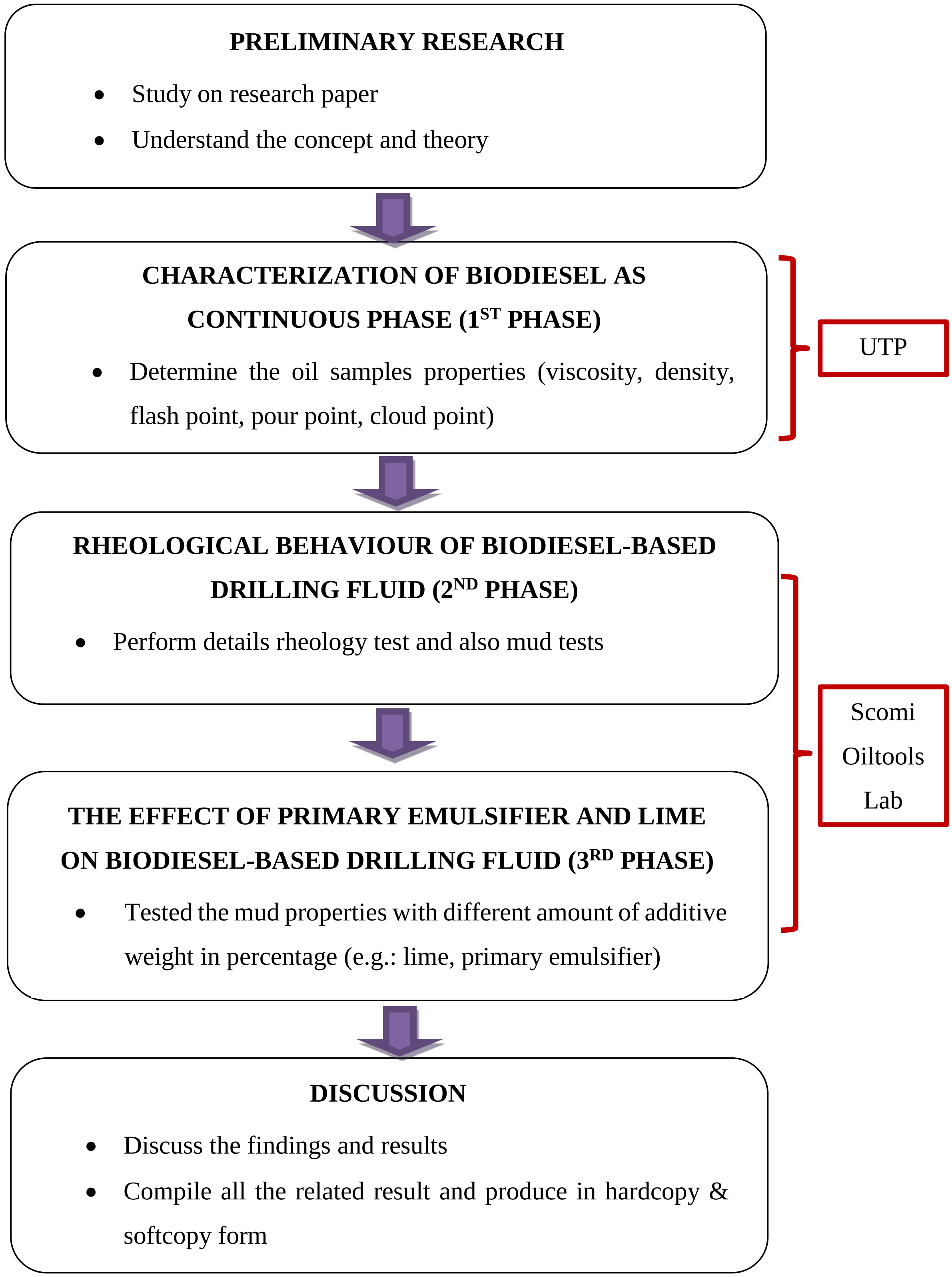


Figure 7: Project Workflow

3.2 TEST EQUIPMENT USED

3.2.1 CHARACTERIZATION OF BIODIESEL AS CONTINUOUS PHASE (1^{ST} PHASE)

EQUIPMENT: ROTARY EVAPORATION R-215

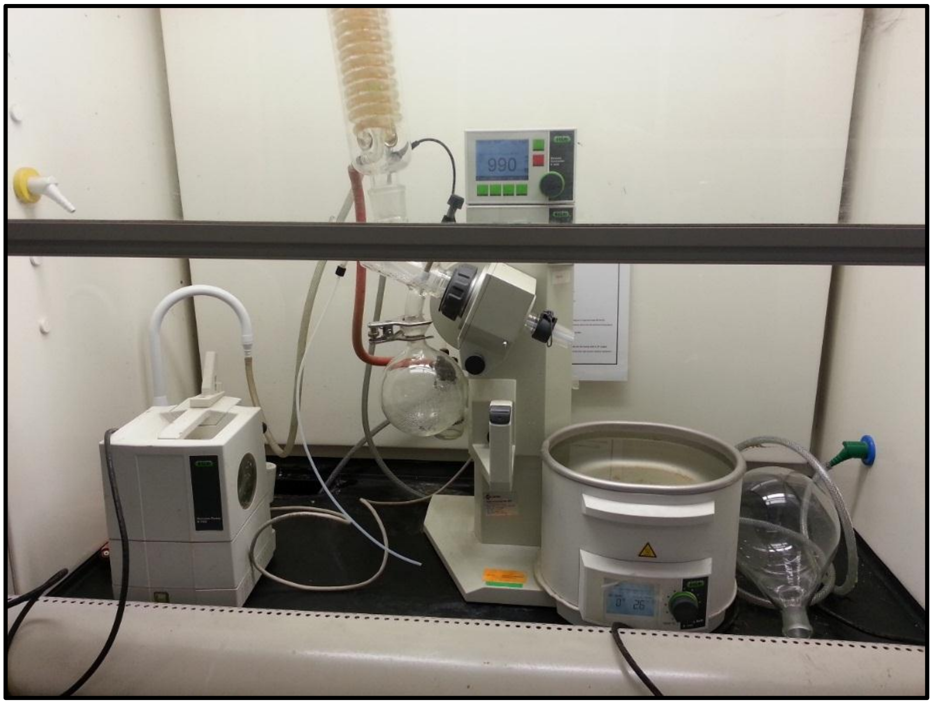


Figure 8: Rotary Evaporation R-215

Parameter: Removing Methanol from Oil sample

Procedure

1. Sample is taken about 1 litre from the biodiesel machine
2. Then, the sample is putted inside the conical flask provided at the equipment of rotary evaporation
3. The conical flask is soak in water for the purpose of heat transferring (T = 70^{O}C)
4. The sample is leaving for about 30-45 minutes in a vacuum space
5. Sample is taken out and the methanol presence is tested
6. Steps 1-5 is repeated, until 5 litre of sample is obtained

If the oil sample condition is not satisfying (methanol presence is still or probably available in the sample), the time allocation at step 4 is increased.

EQUIPMENT: BOHLIN RHEOMETER

Figure 9: Bohlin Rheometer

Parameter: Viscosity (Pa.s)

Procedure

1. Small amount of sample is put on the plate
2. Temperature is set at 40^{o}C and the Gap time is set for 60 seconds.
3. After each sample is tested, the plate is cleaned carefully to prevent any scratch on the plate and contamination from occurred which may affect the reliability of results.
4. The sample which has been used for this experiment is discharged as it can't be reused because its properties had already changed.
5. Steps 1-3 is done with water first (act as calibration) and then repeated with 11 samples of different blending amount of PFAD+PO.
6. The results obtained is recorded.
7. This experiment is repeated three time and the average value is taken.

EQUIPMENT: BROOKFIELD CAP 2000+ VISCOMETER

Figure 10: Brookfield Cap 2000+ Viscometer

Parameter: Viscosity (cP)

Procedure

1. Small amount of sample is put on the plate
2. Temperature is set at 40^{o}C and the time is set for 60 seconds at 500 rpm.
3. After each sample is tested, the plate is cleaned carefully to prevent any scratch on the plate and contamination from occurred which may affect the reliability of results.
4. The sample which has been used for this experiment is discharged as it can't be reused because its properties had already changed.
5. Steps 1-3 is repeated with different spindle size (1/2/3/4/5/6). It is done with water first (act as calibration) and then repeated with 11 samples of different blending amount of PFAD+PO.
6. The results obtained is recorded.
7. This experiment is repeated three time and the average value is taken.

EQUIPMENT: KINEMATIC VISCOSITY BATH

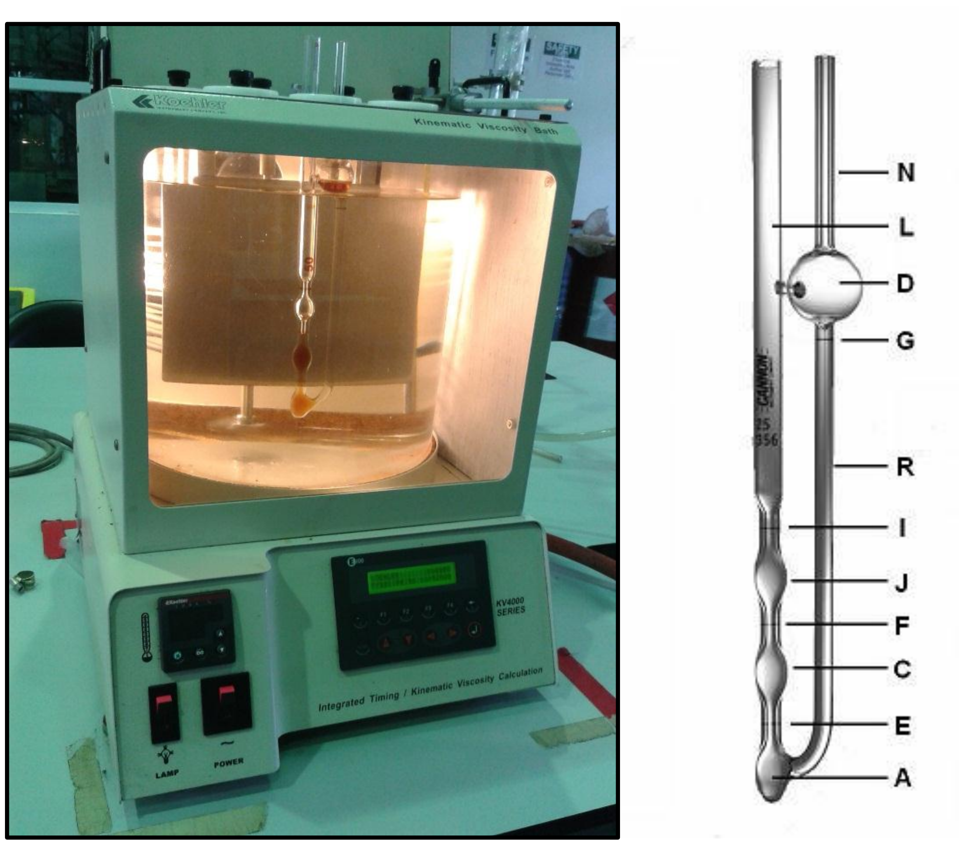


Figure 11: Kinematic Viscosity Bath (Right), Cannon-Fenske Opaque Viscometer (Left)

Parameter: Viscosity (cSt)

Procedure

1. The temperature of kinematic viscosity bath is set to be constant at 40^{o}C
2. To charge the sample into the viscometer (Figure 10-left side), invert the instrument and apply suction to tube arm L, immersing tube N in the liquid sample, and draw liquid to mark G. Wipe clean arm N, and turn the instrument to its normal vertical position.

Figure 12: Rocker 300 is used as the source in supplying the suction force to the viscometer for the sample charging process

3. Place the viscometer into the holder and insert it into the constant temperature bath. Align the viscometer vertically in the bath by means of a small plumb bob in tube L, if a self-aligning holder has not been used.
4. Allow sample to flow through capillary tube R and approximately half-fill bulb A, stopping the meniscus in bulb A by placing a rubber stopper in tube N.
5. Allow approximately 10 minutes for the sample to come bath temperature at 40^{o}c. Make sure the meniscus in bulb A does not reach line E.
6. Remove the rubber stopper and allow the meniscus to travel upwards into bulb C, measuring the efflux time for the meniscus to pass from mark E to mark F.
7. The kinematic viscosity of the sample is calculated by multiplying the efflux time in seconds by the viscometer constant for bulb C.
8. Steps 1 thru 8 is done with water first (act as calibration) and then repeated with 11 samples of different blending amount of PFAD+PO.
9. This experiment is repeated three time and the average value is taken.

3.2.2 RHEOLOGICAL BEHAVIOUR OF BIODIESEL-BASED DRILLING FLUID (2nd PHASE)

The base fluids used is the oil which passes the standard and requirement from 1st Phase of project. Each sample is then will be compared with commercially available mineral base oil from Scomi Oiltools Bhd. The base oil/water ratio (OWR) was kept constant 75/25 in all formulations and the drilling fluid density is set to about 12.0 lb/gal in all samples (Burrows, E.Joannah, J.Hall, & J.Krishner, February 2001), (M.Nasiri, Ashrafizadeh, & A., March 2009). After some calculation is done, the formulation of preparing PFAD-based drilling fluid with the steps are obtain as below;

12.0 lb/gal of CONFI-DRILL (Specific Gravity PFAD = 0.910)

Table 7: Formulation for PFAD-based drilling fluid

Mud Materials	Trade Name	Mixing Order	Time (Min)	Concentration
				PFAD
Base Oil	Sarapar 147	-	-	0.585 bbl.
Primary Emulsifier	CONFI MUL P	1	2	3 ppb
Secondary Emulsifier	CONFI MUL S	2	2	6 ppb
Viscosifier (Premium Organophilic Clay)	CONFI GEL HT	3	5	3.75 ppb
Fluid Loss Agent	CONFI TROL	4	2	4 ppb
Lime	Lime	5	2	10 ppb
Drill Water		6	15	0.205 ppb
Calcium Chloride, 94% Powder	Calcium Chloride			26.14 ppb
Barite, 4.2 SG	DRILL BAR	7	5	193.18 ppb
Additional Time			27	
Total Time			60	

In addition, the approach used in the 2nd phase can be summarize as follow;

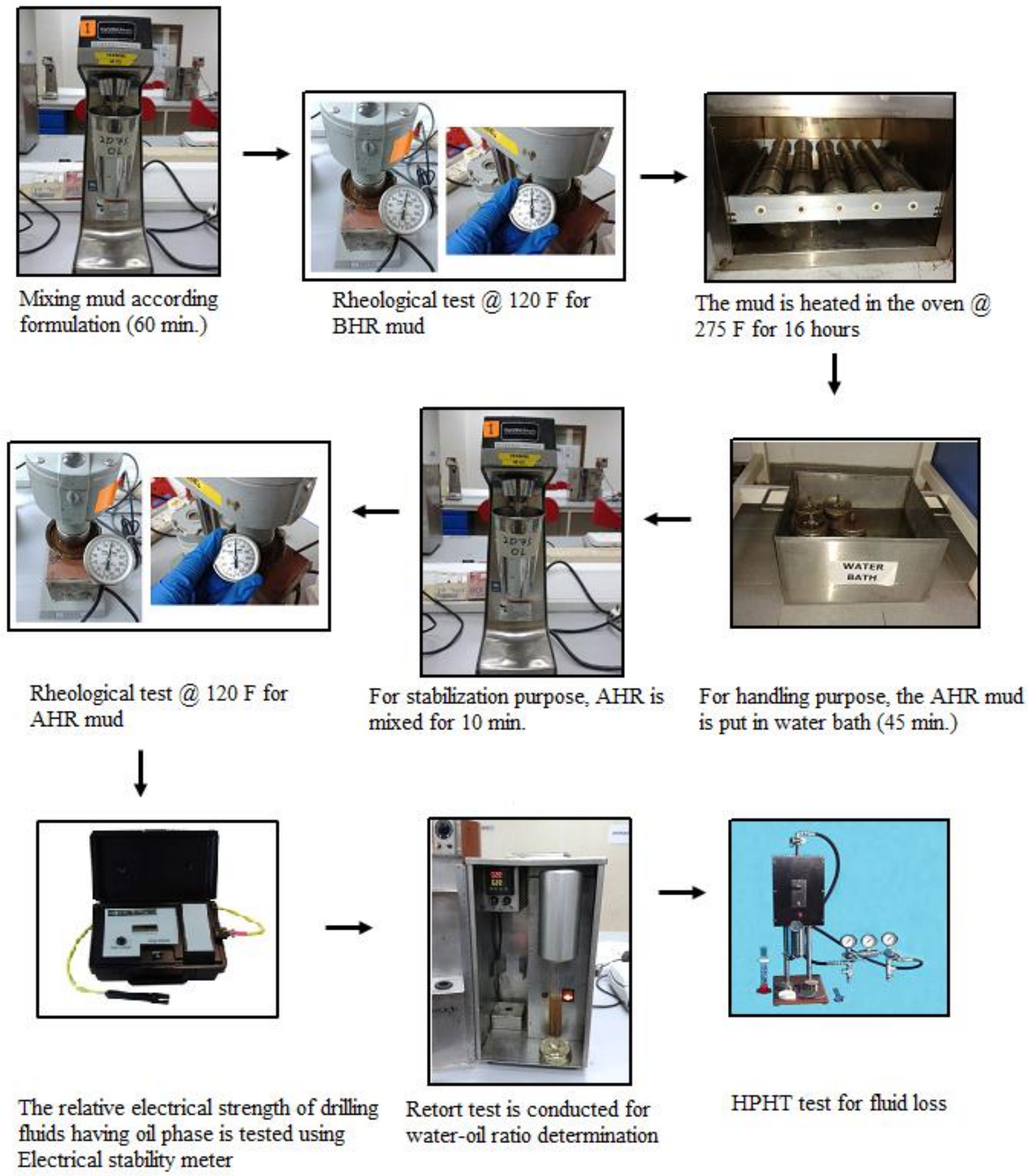


Figure 13: Methodology used in the 2nd Phase

3.2.3 THE EFFECT OF PRIMARY EMULSIFIER AND LIME ON BIODIESEL-BASED DRILLING FLUID (3^{rd} PHASE)

In this phase, the mud properties is observed by changing the mud formulation itself in order to have biodiesel-based drilling fluid that meet the standards in which optimize the findings from 2^{nd} phase. Lime and Primary Emulsifier will be the manipulated variables in this experiment. The methodology used in this phase is same as in the 2^{nd} phase, the different is the weight percentage of lime and primary emulsifier that will be used.

3.5 GANTT CHART

	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
1	Topic Selection							M																					
2	Preliminary Research Work							I D																					
3	Submission of Extended Proposal							S																					
4	Proposal Defence							E																					
5	Mud Preparation							M																					
6	Mud Rheology Experiment							B R																					
7	Submission of Interim Draft Report							E A																					
8	Submission of Interim Report							K																					
9	FYP II																												

Figure 14: Project Gantt chart

	Detail / Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1.	Lab work	■	■	■	■	■	■	■	MID SEMESTER BREAK								
2.	Submission of Progress Report							●									
3.	Lab work continues									■	■	■	■	■			
4.	Pre-Sedex											●					
5.	Submission of Draft Report												●				
6.	Submission of Dissertation													●			
	Submission of Technical Paper													●			
7.	Oral Presentation														●		
8.	Submission of Project Dissertation																●

Figure 15: Project Gant Chart for FYP II

CHAPTER 4: RESULT & DISCUSSION

4.1 CHARACTERIZATION OF BIODIESEL AS CONTINUOUS PHASE (1^{ST} PHASE)

The first step need to be done is to examine the oil samples properties. Either the properties of biodiesel samples have meet the EN 14214:2003[3] and ASTM D6751:06[4] standard or not. Below are the properties of PFAD biodiesel prepared in this project;

Table 8: PFAD Biodiesel in this project

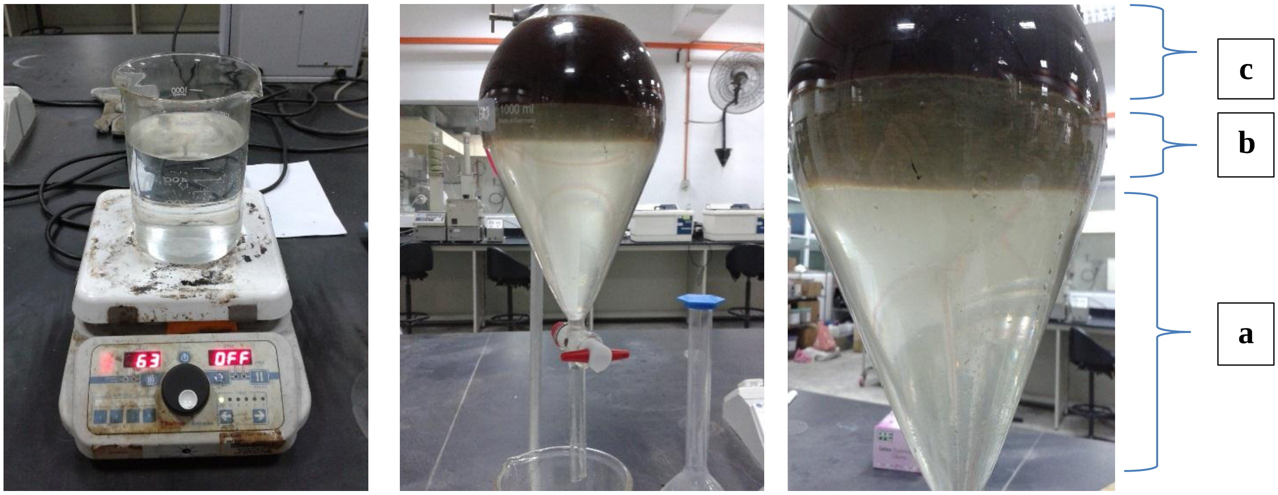
Properties	Unit	PFAD Biodiesel in this project	EN 14214:2003		ASTM D6751:06	
			Min	Max	Min	Max
Ester Content	% mass	97	96.5	-	-	-
Density at 15 ^{O}C	kg/m^3	890	860	900	870	900
Viscosity at 40 ^{O}C	cSt	4.8	3.5	5.0	1.9	6.0
Flash Point	^{o}C	110	120	-	130	-
Cloud Point	^{o}C	16	-	-	-3	12
Pour Point	^{o}C	-	-	-	-15	10

Based on the properties obtained and listed above, it can be seen clearly that the flash point (below) and cloud point (exceed) of the PFAD Biodiesel in this project does not meet the standard. This error is probably caused by the presence of methanol in the prepared PFAD Biodiesel which was not completely removed during the process of removing methanol. The excessive presence of methanol can greatly affect the quality of prepared PFAD Biodiesel. Therefore, it is very important to recheck and make sure that approximately all the methanol has been removed from the sample prepared. In other hand, there is no data for pour point due to the device failure.

[3] European Standard For Biodiesel

[4] Standard Specification For Biodiesel Fuel (B100) Blend Stock For Distillate Fuels

In order to remove the methanol, several techniques can be applied. There are two technique which normally used, which are using automatic equipment namely Rotary Evaporation R-215 or by conducting washing process several time by manually.



Water is heated at < 100°C in order to keep it at warm temperature	The warm water is then poured into the container consist of PFAD. The mixture is leaved for 20-30 min.	The 'a' & 'b' are discharged. Process is repeated until no more 'a' & 'b' are formed.

Figure 16: Washing Process of PFAD

Whereby 'a' = water, 'b' = glycerol, 'c' = pure oil

Thus, it can be concluded that, after undergone the process of removing the methanol, the prepared PFAD biodiesel is ready to be the feedstock for this project.

For the 1st phase of this project, the parameter emphasized in examine the oil samples properties is **viscosity**. In order for the oil samples to be passed to be used in the 2nd phase of this project, their viscosity (kinematic viscosity) should not exceed **10.00 cSt at** temperature of **40°C** (acceptable range in oil and gas industry). Based on the experiments conducted, the data obtain have been quality check (QC) and summarize as below;

Table 9: The Kinematic Viscosity of Palm Fatty Acid Distillate (PFAD) with Palm Olein (PO) at 40°C

Mixing Ratio of PFAD-PO	Time (min)	Time (s)	Constant (cSt/s)	Viscosity (cSt)
Distilled water	3.56	236	0.004	0.94
100-0	20.01	1201	0.004	4.80
90-10	26.45	1605	0.004	6.42
80-20	33.36	2016	0.004	8.06
70-30	**47.15**	**2835**	**0.004**	**11.34**
60-40	**73.07**	**4387**	**0.004**	**17.55**

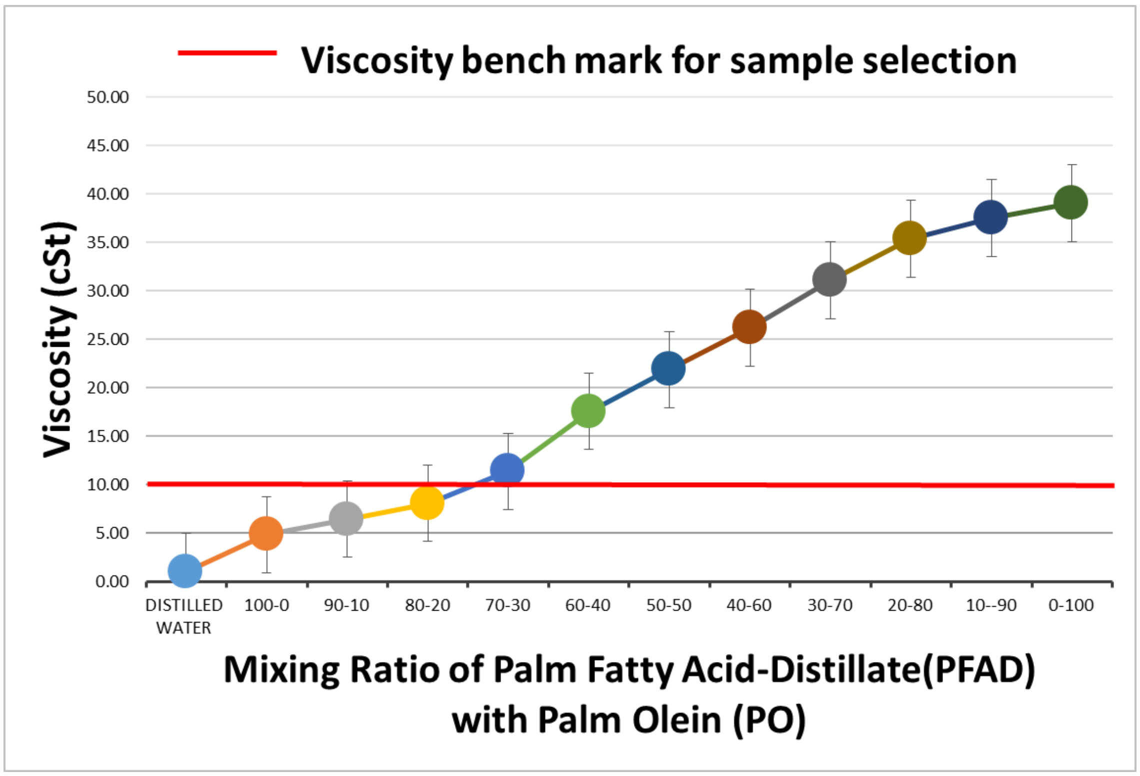


Figure 17: The viscosity trend of the prepared oil sample at 40°C

Based on the result above, the suitable oil samples to be used for the 2nd Phase of this project are pure PFAD and also oil sample with mixing ratio of PFAD-PO 90:10 so as

80:20. This is because, only this 3 oil samples that gives viscosity reading less than 10 cSt and meet the API Recommended Practice 13B-2 to be used as the base for drilling fluid.

However, after considering the external factor such as materials that will be used in the drilling fluid formulation itself, which will increase the viscosity, only pure PFAD (100:0) will be used in the next phase.

4.2 RHEOLOGICAL BEHAVIOUR OF BIODIESEL-BASED DRILLING FLUID (2^{nd} PHASE)

In this stage, a total of 13 samples by using PFAD as based for drilling fluid are created. Each sample created is tested for their rheology properties before and after it is hot rolled for 16 hours at temperature of 275^{0}F. The normal formulation of drilling fluid by using PFAD as based is tested first and compared with the available drilling fluid in the market.

Table 10: Comparison of normal formulation of PFAD-base drilling fluid with common drilling fluids in market.

Products	mixing time, min	Sarapar 147	PFAD Sample	Diesel Fuel	Saraline 185
Base Oil		152.25	186.32	172.06	150.48
CONFI-MUL P	2	3.00	3.00	3.00	3.00
CONFI-MUL S	2	6.00	6.00	6.00	6.00
CONFI-GEL HT	5	3.75	3.75	3.75	3.75
CONFI-TROL	2	4.00	4.00	4.00	4.00
LIME	2	10.00	10.00	10.00	10.00
fresh water	15	69.30	71.75	69.30	69.30
CaCl2		25.06	26.14	25.80	25.06
DRILL BAR	5	229.00	193.18	208.55	229.00
Properties Initial:	**Spec Base**	**Sarapar 147**	**PFAD Sample**	**Diesel Fuel**	**Saraline 185**
Mud density, lb/gal (formulated)	**12.0**	**12.0**	**12.0**	**12.0**	**12.0**
Rheological properties		120 °F	120 °F	120 °F	120 °F
600 RPM		51	-	96	55
300 RPM		30	-	67	34
200 RPM		21	-	55	25
100 RPM		14	291	43	17
6 RPM	**8-12**	7	196	27	9
3 RPM		6	177	25	8
PV, cP	**<35**	21	-	29	21
YP, lb/100 ft^2	**15-25**	9	-	38	13
Gel 10 sec, lb/100 ft^2	**6-10**	8	150	27	11
Gel 10 min, lb/100 ft^2		12	158	33	18
ES, volts at 120 °F	**>500**	421	-	1267	777
Properties AHR, BHST 16 hr, (275°F):	**Spec**	**Sarapar 147**	**PFAD Sample**	**Diesel Fuel**	**Saraline 185**
Mud density, lb/gal	**12.0**	**12.0**	**12.0**	**12.0**	**12.0**
Rheological properties		120 °F	120 °F	120 °F	120 °F
600 RPM		52	-	63	57
300 RPM		27	-	37	32
200 RPM		19	-	29	22
100 RPM		12	-	20	14
6 RPM		4	-	10	5
3 RPM		3	-	9	4
PV, cP		25	-	26	25
YP, lb/100 ft^2		2	-	11	7
Gel 10 sec, lb/100 ft^2		4	-	10	5
Gel 10 min, lb/100 ft^2		6	-	15	7
ES, volts at 120 °F		279	-	847	530
OWR					
oil, ml		30	25.5	31	31
water, ml		10	7.5	8	9
solids, ml		10	17	11	10
HTHP (500 psi, 275 °F), ml/30 minute		2	1.2	0.8	2.8
water, ml		-	-	-	-
oil, ml		1	0.6	0.4	1.4
total, ml		1	0.6	0.4	1.4
filter cake, mm					

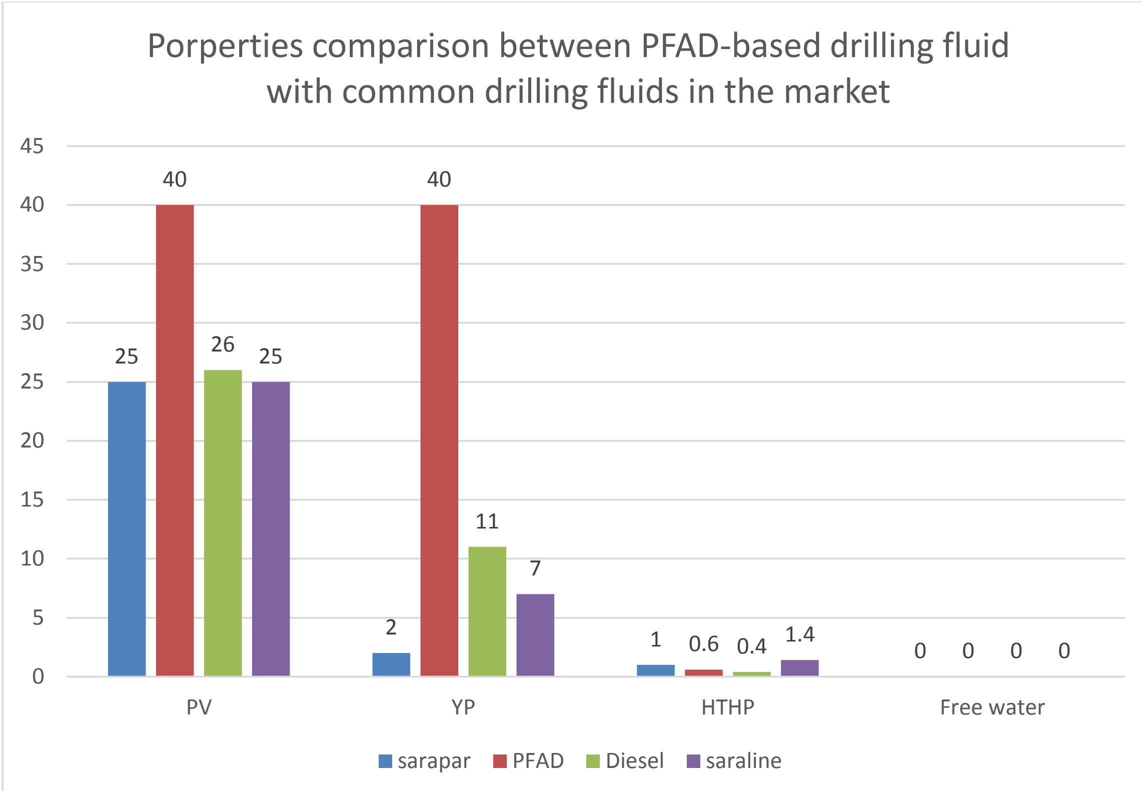


Figure 18: Properties comparison of PFAD-based drilling fluid with common drilling fluids in the market (*40 reading indicates off scale value)

From the experiment conducted, it has been found that the PFAD-based drilling fluid is very viscous as compared to other, either before or after it is hot rolled. This has led to the higher value (off scale) of plastic viscosity and yield point. However, the performance of PFAD-based drilling fluid in handling fluid loss is quite good as it gives value within the acceptable range in oil and gas industry.

Table 11: The range value of the rheological parameter that must be meet in preparing drilling fluid (according to acceptable range in oil and gas industry) after hot rolling.

Parameter	Value / Range
Density	12
Plastic viscosity (PV)	< 35
Yield point (YP)	15-25
6 rpm	8-12
Initial Gel strength (10 sec.)	6-10
Fluid loss (HTHP)	< 5
Free water	0

In drilling fluid field, the properties after it has been hot rolled is given more emphasized. This is because, the behavior of the drilling fluid after being hot rolled is considered same as its behavior while it is used down deep. Based on the result above, after hot rolled, the PFAD-based drilling fluid has a higher reading of rheology until it is off scale (>300 in value for 600 rpm and below so as >1999 for Electrical stability meter reading, ES). As a result the machine can't read its plastic viscosity and yield point.

The formation of viscous drilling fluid by using PFAD as based in drilling fluid is probably due to the reaction of the materials used in preparing the mud itself. For instance, logically when acid react with base it will produce soap or precipitation. This idea is used in the reaction between PFAD oil which consists mainly fatty acid (act as acid) react with lime (act as base) which eventually create precipitation, increase the amount of suspension and causes the drilling fluid become very thick or highly viscous. This properties might not favorable to be used in drilling fluid as it will causes drilling problems such as pipe sticking and wellbore instability. Hence, modification need to be done in the formulation to make sure the PFAD-based drilling fluid meet the standard rheological parameter.

4.3 THE EFFECT OF PRIMARY EMULSIFIER & LIME ON THE PFAD-BASED DRILLING FLUID (3rd PHASE)

Since, the PFAD itself consist of fatty acid and already viscous in its form, the ideas come to reduce the amount of fatty acid and reaction between acid with base which perhaps will reduce the viscosity of the PFAD-based drilling fluid.

Table 12: Summary of alternatives to be used to modified the PFAD-based drilling fluid

Possible Causes	Description	Alternative	Reliability
Primary emulsifier	Primary emulsifier supply fatty acid to the drilling fluid in order to maintain the emulsifying of water inside oil so that there is no free water in filtrate.	reduce amount used	• The PFAD itself consists of fatty acid. • High amount of fatty acid will increase the viscosity of the drilling fluid especially after hot rolling process (Yield point) as the fatty acid chain will break-down and dissolves as particles in the drilling fluid.
Lime	Lime is used to stimulate the primary emulsifier to activate the flocculants process to take place especially after hot rolled.	reduce amount used	• Excess lime will react with the PFAD and also with the primary emulsifier which eventually will plug the mud flow. • This is because fatty acid will act as acid and lime as base, which consequently cause the colloidal particles in suspension to form into bunches, causing solids to settle out which contribute to the increasing in viscosity of the drilling fluid.

Due to the analysis above, several sample has been created with different amount of primary emulsifier and also lime in order to study their effect on the PFAD-based drilling fluid. Detail observation is also given to see the comparison between those sample especially before and after hot rolling process as well as their rheological and mud tests.

Below is the observation of some of the samples;

THE OBSERVATION OF SAMPLES BEFORE & AFTER HOT ROLL

Table 13: Comparison of mud features before and after hot rolled [P=Primary emulsifier, S=Secondary emulsifier, L=Lime]

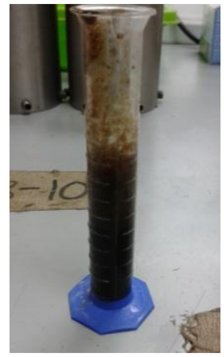
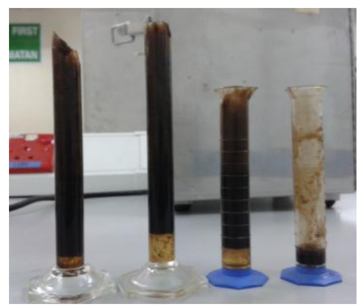
Sample	Before Hot Roll (BHR)	After Hot Roll (AHR)
2 (0P 9S 5L)		
4 (1P 8S 0L)		
5 (1P 8S 0.5 L)		

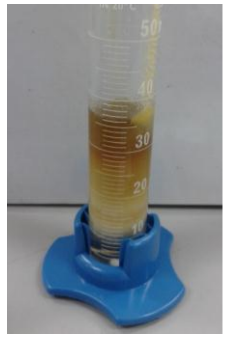
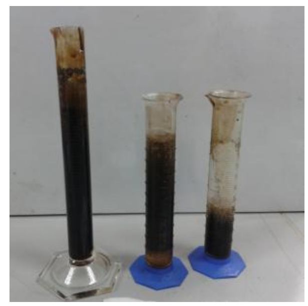
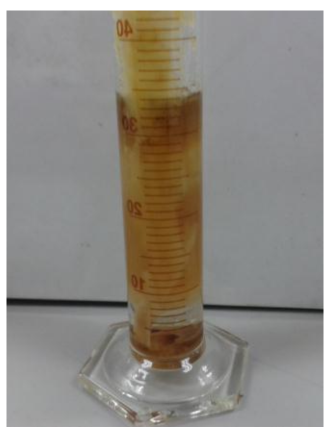
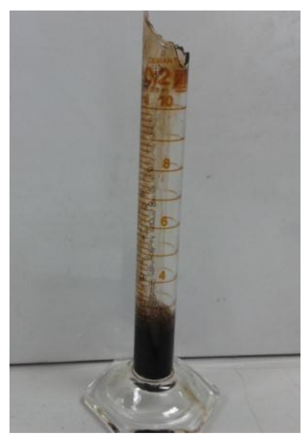
6 (1P 8S 1 L)		

Based on the picture above, it can be seen the difference texture of the mud before and after it undergoes hot rolled process. The features and textures of the mud after going hot rolling is smoother. This probably due to the dissolving of particles inside the mud itself as a result of being placed at high temperature (275^0F) for 16 hours.

OBSERVATION ON THE RESULTS OF RETORT & HPHT FILTER PRESSURE TEST

Table 14: Observation on results of rheological and mud tests conducted to sample after it is hot rolled [P=Primary emulsifier, S=Secondary emulsifier, L=Lime]

Sample	Retort Test	HPHT (Filtration)
1 (0P 9S 0L)	 Consists of water at the bottom, dense emulsion at the middle & oil on top.	 Bubbles are form in the filtration
2 (0P 9S 5L)	 Consists of water at the bottom, dense emulsion at the middle (less than **0P 9S 0L**) & oil on top	 The amount of filtrate is larger and consists free water
3 (0P 9S 10L)	 Consists of water at the bottom, dense emulsion at the middle (less than **0P 9S 5L**) & oil on top	 The filtration is quite thick.

4 (1P 8S 0L)	 Consists of water at the bottom, light emulsion at the middle & oil on top	 The amount of filtration is larger and quite thick.
5 (1P 8S 0.5L)	 Consists of water at the bottom, light emulsion at the middle & oil on top	 The amount of filtrate is larger and consists free water
6 (1P 8S 1L)	 Consists of less water at the bottom, light emulsion at the middle & oil on top	 The amount of filtration is smaller and no free water is exist

Those rheological and mud tests above are conducted on the sample after it has been hot rolled. This is because, the mud is in the ideal condition especially when it will be using under high pressure and temperature. Plus, the mud composition also had changed as a result of high pressure and temperature. Each of the observation is then recorded in a matrix form for standard viewing.

Table 15: Results of PFAD-based drilling fluid with no primary emulsifier and different amount of lime

Products	mixing time, min	1		2		3	
PFAD Sample		186.32		186.32		186.32	
CONFI-MUL P	2						
CONFI-MUL S	2	9.00		9.00		9.00	
CONFI-GEL HT	5	3.75		3.75		3.75	
CONFI-TROL	2	4.00		4.00		4.00	
LIME	2			5.00		10.00	
fresh water	15	71.75		71.75		71.75	
CaCl2		26.14		26.14		26.14	
DRILL BAR	5	193.18		193.18		193.18	
Properties Initial:	**Spec Base**	**1**		**2**		**3**	
Mud density, lb/gal (formulated)		**12.0**		**12.0**		**12.0**	
Rheological properties		120 °F		120 °F		120 °F	
600 RPM		115		-		-	
300 RPM		111		282		267	
200 RPM		81		257		243	
100 RPM		56		224		210	
6 RPM	**8-12**	18		153		144	
3 RPM		17		146		136	
PV, cP	**<35**	4		-		-	
YP, lb/100 ft^2	**15-25**	107		-		-	
Gel 10 sec, lb/100 ft^2	**6-10**	31		135		130	
Gel 10 min, lb/100 ft^2		32		139		132	
ES, volts at 120 °F	**>500**	-		1402		881	
Properties AHR, BHST 16 hr, (275°F):	**Spec**	**1**		**2**		**3**	
Mud density, lb/gal	**12.0**	**12.0**		**12.0**		**12.0**	
Rheological properties		120 °F		120 °F		120 °F	
600 RPM		158				-	
300 RPM		98				-	
200 RPM		81		287		-	
100 RPM		62		244		-	
6 RPM		41		172		-	
3 RPM		39		167		-	
PV, cP		60		-		-	
YP, lb/100 ft^2		38		-		-	
Gel 10 sec, lb/100 ft^2		40		195		-	
Gel 10 min, lb/100 ft^2		39		198		-	
ES, volts at 120 °F		1690		1256		-	
OWR							
oil, ml		28.5		20		19.5	
water, ml		6.5		10		12	
solids, ml		15		20		18.5	
HTHP (500 psi, 350 °F), ml/30 minute		16.2		22.8		20	
water, ml		0.2		0.6		1	
oil, ml		7.9		10.8		9	
total, ml		8.1		11.4		10	
filter cake, mm							

Table 16: Results of PFAD-based drilling fluid with low amount of primary emulsifier and increasing amount of lime

Products	mixing time, min	4		5		6	
PFAD Sample		186.32		186.32		186.32	
CONFI-MUL P	2	1.00		1.00		1.00	
CONFI-MUL S	2	8.00		8.00		8.00	
CONFI-GEL HT	5	3.75		3.75		3.75	
CONFI-TROL	2	4.00		4.00		4.00	
LIME	2			0.50		1.00	
fresh water	15	71.75		71.75		71.75	
CaCl2		26.14		26.14		26.14	
DRILL BAR	5	193.18		193.18		193.18	
Properties Initial:	**Spec Base**	**4**		**5**		**6**	
Mud density, lb/gal (formulated)		**12.0**		**12.0**		**12.0**	
Rheological properties		120 °F		120 °F		120 °F	
600 RPM		171		200		196	
300 RPM		133		158		174	
200 RPM		114		141		156	
100 RPM		92		116		131	
6 RPM	**8-12**	52		69		76	
3 RPM		47		64		71	
PV, cP	**<35**	68		42		22	
YP, lb/100 ft^2	**15-25**	65		116		152	
Gel 10 sec, lb/100 ft^2	**6-10**	44		59		64	
Gel 10 min, lb/100 ft^2		46		62		67	
ES, volts at 120 °F	**>500**	676		692			
Properties AHR, BHST 16 hr, (275°F):	**Spec**	**4**		**5**		**6**	
Mud density, lb/gal	**12.0**	**12.0**		**12.0**		**12.0**	
Rheological properties		120 °F		120 °F		120 °F	
600 RPM		104		170		196	
300 RPM		72		136		159	
200 RPM		60		117		137	
100 RPM		46		95		112	
6 RPM		27		60		70	
3 RPM		25		57		63	
PV, cP		32		34		37	
YP, lb/100 ft^2		40		102		122	
Gel 10 sec, lb/100 ft^2		23		44		51	
Gel 10 min, lb/100 ft^2		26		46		61	
ES, volts at 120 °F		531		1166		1689	
OWR							
oil, ml		27		26		26	
water, ml		10		10		8	
solids, ml		13		14		16	
HTHP (500 psi, 350 °F), ml/30 minute		41		39.6		4.4	
water, ml				1			
oil, ml		20.5		18.8		2.2	
total, ml		20.5		19.8		2.2	
filter cake, mm							

In order to analyse the effect of primary emulsifier and lime on the PFAD-based drilling fluid, a graph has been built to give a better picture on the rheological parameter of each sample and easier for comparison to be made.

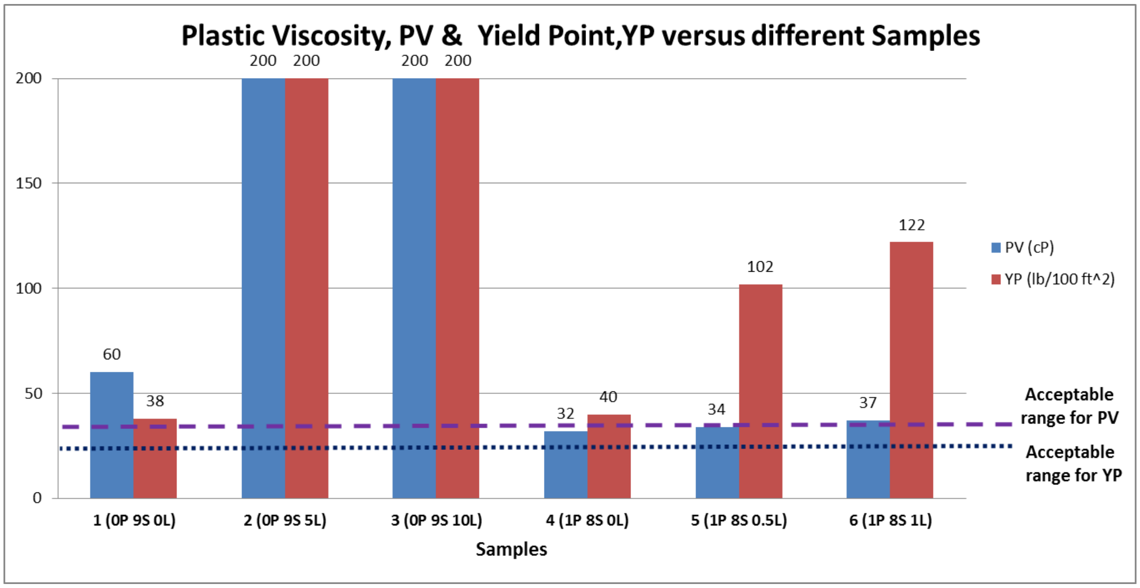


Figure 19: PV&YP versus different samples (*200 indicates off scale)

From figure above, there are two samples which the readings are off scale which are sample no.2 and no. 3. Another four [sample no. 1, 4, 5 and 6] samples are compared with the acceptable range of rheological parameter in oil and gas industry. In this case, the YP for all four samples are more than the acceptable range. Meanwhile, for the PV, sample no. 1 yields a higher value and the other three samples value are within the tolerable range of ideal PV. The higher value of PV and YP is most probably contributed by the higher viscosity (PV) and also excessive material suspension in the drilling fluid (YP).

Best Sample: Sample no. 4 [1P 8S 0L], Sample no. 5 [1P 8S 0.5L], Sample no.6 [IP 8S 1L]

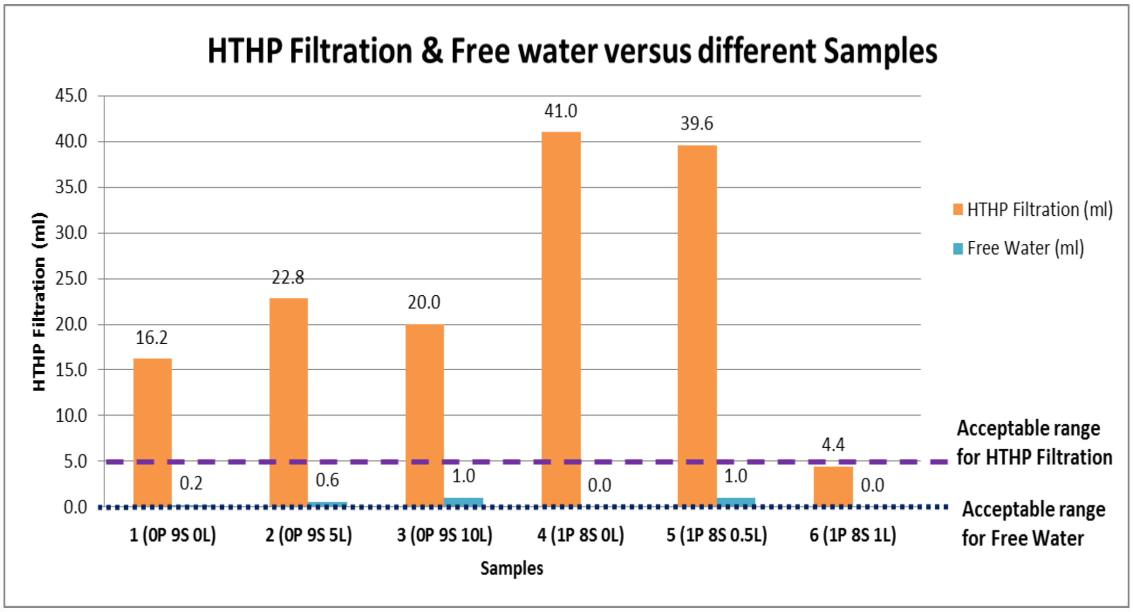


Figure 20: HTHP & Free water versus different formulation of sample

The acceptable range for HTHP filtration is less than 0.5 ml. However, in this project, all the sample's HTHP filtration results are more than 0.5. To narrow the best sample to be chosen, the results which nearest to ideal value will be selected. Nevertheless, for free water, there are two samples (sample no. 4 and 6) which meet the ideal standard. No free water means that the emulsifying of water in oil is in good performance.

For the sample with no primary emulsifier (first 3 samples), the HTHP filtration results are relatively lower than those samples with primary emulsifier (sample no. 4, 5 and 6). This is probably due to the presence of solely secondary emulsifier in the mud composition which causes the mud become thick. In addition, this samples also came with free water which indicates that the water in oil is not well emulsify as a result of the absentee of primary emulsifier.

In another hand, for sample with the presence of primary emulsifier, the HTHP filtration results and amount of free water are decreasing with the increasing amount of lime. This behaviour indicates that the performance of emulsifying water in oil is improving with the presence of primary emulsifier and increasing number of lime. However, the amount of lime should not be more than 10 in weight percentage as a very viscous mud will be formed (original PFAD-based mud in 2nd phase).

Best Sample: Sample no. 1 [0P 9S 0L], Sample no. 6 [IP 8S 1L]

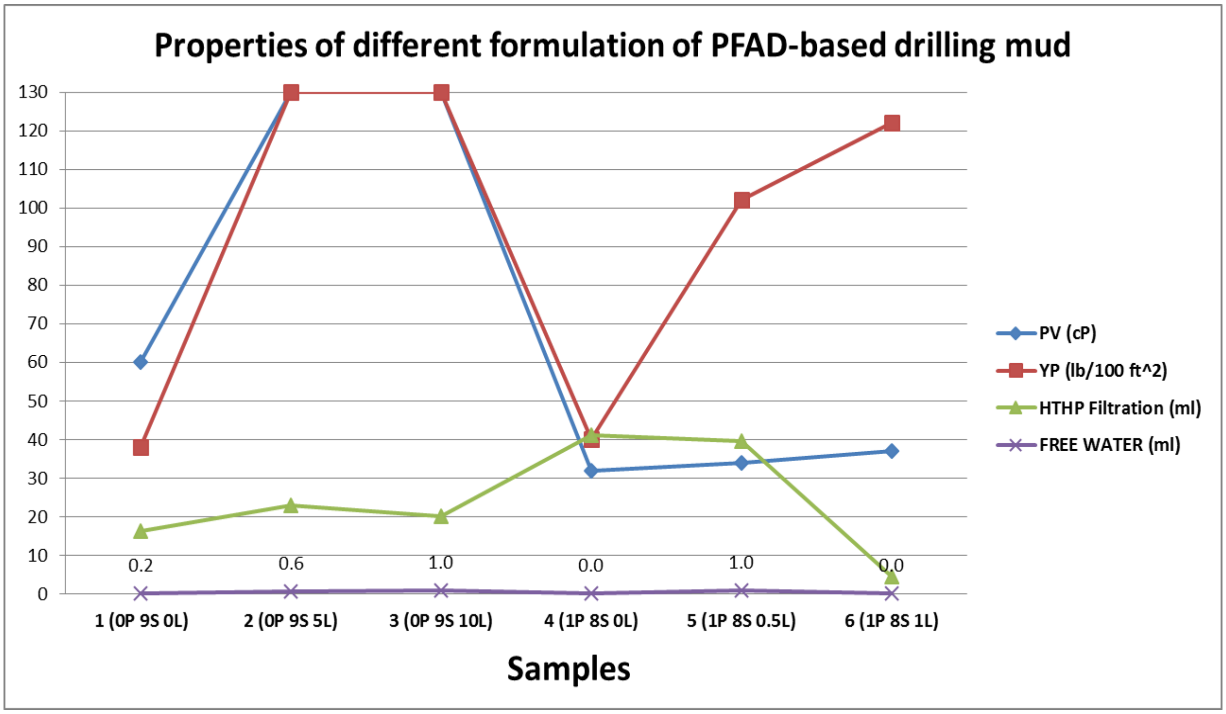


Figure 21: Properties of different formulation of PFAD-based drilling mud

Figure above is the summary of the overall trend of the effect of changing the amount (by weight percentage) of primary emulsifier and lime in the formulation of the drilling mud. This properties is also used in determining the best formulation of the PFAD-based drilling fluid as a results of the changes. Tabular form as below is created to make the comparison in choosing the best formulation of PFAD-based drilling fluid is easier after the analysis is conducted on the trend and performance of each samples.

Table 17: Analysis of the best formulation of PFAD-based drilling fluid as compared to ideal standard [Best = 1, Poor = 0]

Samples	**1**	**2**	**3**	**4**	**5**	**6**
PV & YP	0	0	0	1	1	1
HTHP Filtration & Free water	1	0	0	0	0	1
Total	1	0	0	1	1	2

From the analysis above, the sample which is the most optimum sample as compared among them is **sample no. 6, (1P 8S 1L)**. This sample have a great performance in PV, YP, HTHP filtration and free water as compare to the ideal standard. To summarize, samples with no primary emulsifier and increasing amount of lime yield a higher rheology reading but have a lower value in filtration and come with the presence of free water. In other hand, samples with low amount of primary emulsifier and increasing amount of lime gives a low rheology reading with decreasing amount of filtration as well as free water.

CHAPTER 5: CONCLUSION & RECOMMENDATION

5.1 CHARACTERIZATION OF BIODIESEL AS CONTINUOUS PHASE (1^{ST} PHASE)

5.1.1 CONCLUSION

- The first objective of the project which is to characterize the oil samples is achieved. In which, it has been found that PFAD biodiesel and also PFAD+PO with mixing ratio of 90:10 and 80:20, are highly potential in replacing the conventional mineral diesel as based in drilling fluid.
- However, this justification wasn't strong enough until the 2^{nd} and the 3^{rd} phase of this project is completed. For the time being, based on the data, results as well as the interpretation obtain from the 1^{st} phase, the properties of oil samples (pure PFAD biodiesel and also PFAD+PO with mixing ratio of 90:10 and 80:20) have passed the test.
- In the next phase, the 100 % of PFAD oil will be tested first and continued with the other mixing ratio if it is not success.
- The first objective of this project which is to characterize biodiesel as continuous phase is achieved.

5.1.2 RECOMMENDATION

- Increase the variation type of biodiesel to be tested such as using pure rubber seed oil, mixing of rubber seed oil and PFAD, and also blending of PFAD with common based used in the market (diesel, sarapar 147, saraline 185)

5.2 RHEOLOGICAL BEHAVIOUR OF BIODIESEL-BASED DRILLING FLUID (2nd PHASE)

5.2.1 CONCLUSION

- Based on the properties of the oil samples in the 1st phase, only pure PFAD will be tested in this project. This is because, even though pure PFAD oil which has the lowest viscosity had formed into a viscous drilling fluid after it has been mixed.
- The normal formulation to prepare oil-based drilling fluid cannot be applied to PFAD-based drilling fluid as it will lead to unfavorable behavior of drilling fluid (doesn't meet the acceptable range in oil and gas industry) especially after hot rolling process.
- The second objective of this project which is to study the rheological behaviour of biodiesel-based drilling fluid is achieved.

5.2.2 RECOMMENDATION

- Modification need to be done in order to make sure that the behavior of PFAD-based drilling fluid meet the acceptable range in oil and gas industry. Such as;
  i. Different amount of weight percentage for additives such as lime, primary emulsifier as well as secondary emulsifier.

5.3 THE EFFECT OF PRIMARY EMULSIFIER & LIME ON THE PFAD-BASED DRILLING FLUID (3rd PHASE)

5.3.1 CONCLUSION

- Based on the result, the trend of the rheological parameter is in good quality with the lower amount of lime and primary emulsifier.
- Samples with no primary emulsifier and increasing amount of lime yield a higher rheology reading but have a lower value in filtration and come with the presence of free water.
- This indicates the emulsifying of water inside oil is lower.
- Secondary emulsifiers generally are not used alone to make a stable oil mud
- Samples with low amount of primary emulsifier and increasing amount of lime gives a low rheology reading with decreasing amount of filtration as well as free water.
- Hence, emulsifier set must be able to meet specifications despite being diluted. The reason behind this is different amount of emulsifier react differently to maintain a stable water-in-oil emulsion
- The 3rd objective of this project which is to study the effect of lime and primary emulsifier on the biodiesel-based mud formulation is achieved.

5.3.2 RECOMMENDATION

- Further study need to be done by decreasing the scale of changes in the weight percentage of primary emulsifier and lime for accuracy and details purpose. The effect on different secondary emulsifier can also be tested as PFAD-based drilling fluid is quite a unique based.
- Additional study should be proceed with the best PFAD-based drilling fluid, **sample no. 6, [1P 8S 1L]** with respect to;
  i. Ability in handling shale instability
  ii. Against contamination (e.g.; sea water, cement, calcium carbonate and etc.)
- Same study including all 3 phases should be done for pure rubber seed oil, mixture of PFAD and Rubber Seed oil.

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APPENDIX

Table 18: The kinematic viscosity of the 11 samples of prepared oil at 40ºC

Mixing Ratio of PFAD-PO	Viscosity (cSt)
Distilled water	0.94
100-0	4.80
90-10	6.42
80-20	8.06
70-30	11.34
60-40	17.55
50-50	21.85
40-60	26.15
30-70	31.08
20-80	35.38
10--90	37.49
0-100	39.04

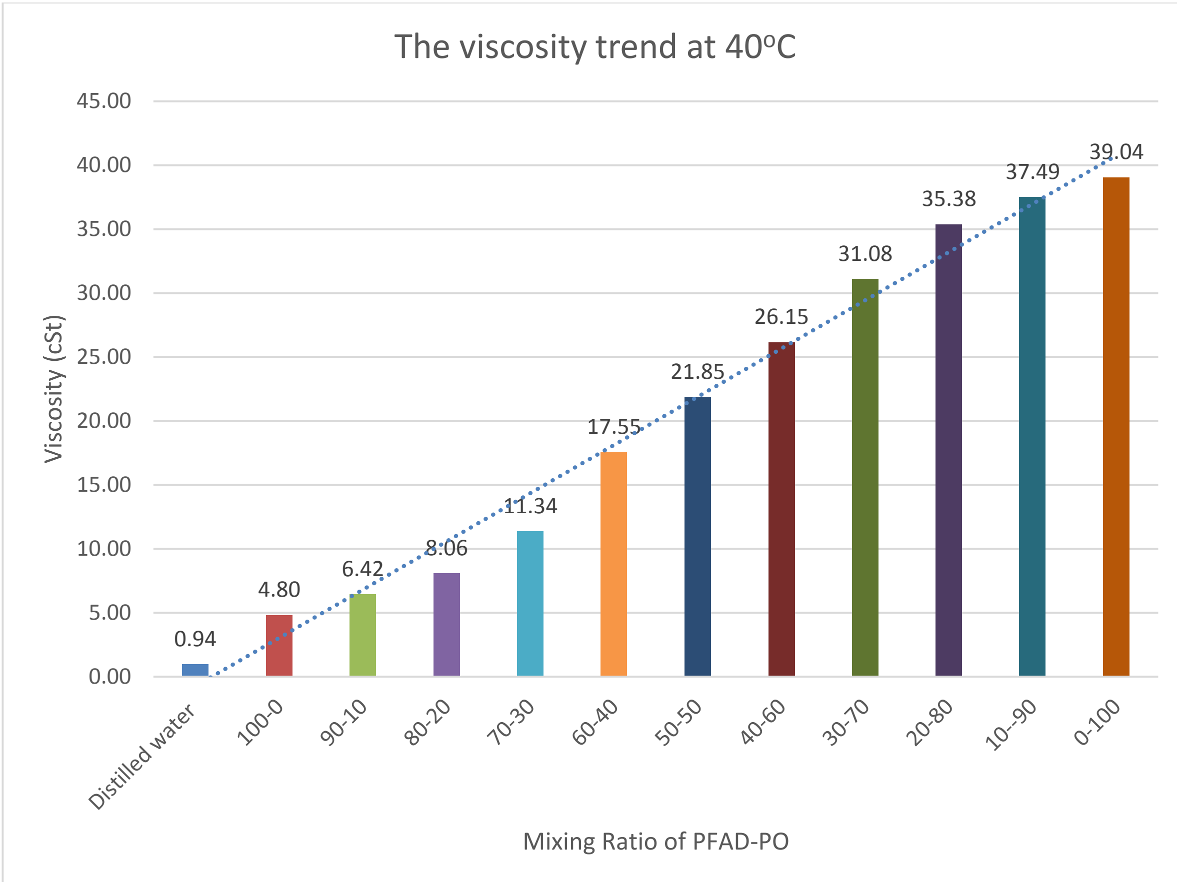

Figure 22: The viscosity trend of 11 samples of prepared oil at 40°C