Study on the Effect of Lime on Biofuel-based (Palm Fatty Acid Distillate) Drilling Fluid

by

Arunan Isvaran

Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Petroleum Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfillment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(PETROLEUM ENGINEERING)

Approved by,

____

(AP DR SUZANA BT YUSUP)

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 original work is my own except as specified in the references and acknowledgements, and the original work contained herein have not been undertaken or done by unspecified sources or persons.

(ARUNAN ISVARAN)

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ABSTRACT

The rising world energy demand has led to exploration for oil and gas in increasingly difficult environments. The exploration has now gone into sensitive regions combining with deviated wells and horizontal well developments under challenging geological formations. Oil-based drilling fluids are widely used in drilling in all parts of the world for highly technical wells and cases. This is because oil-based drilling fluids perform much better than water-based fluids in borehole stabilization and providing lubricity and subsequently faster rates of penetration and performance. However, they are a cause for environmental concern and with the potential of causing bad damage to the environment through mud spills and improper disposal of oil-contaminated drill cuttings and etc.

The industry therefore needed an urge to find a fluid that satisfies both the environmental and technical criteria by showing excellent technical performance and it would have to behave like a traditional OBM with all the technical advantages obtaining regulatory approval and being cost-effective. Thus, this project focuses on this concern of developing an environmentally safe biofuel-based drilling fluid with use of Palm fatty acid distillate oil as the base fluid. The drilling fluid produced should satisfy all the needed criteria with required rheological readings and mud performance as compared to conventional diesel-based drilling fluid.

Firstly, the main idea behind this project is to study the suitability of palm fatty acid distillate (PFAD) to be used as a continuous fluid in drilling fluids replacing the diesel and mineral oils. Next phase is focused on producing a new drilling fluid system using the PFAD as the base oil. Finally, the effect of different concentrations of lime, which is used as fatty acid activator and alkalinity control agent in drilling fluids is analyzed and studied through the regular mud tests for further optimization.

CHAPTER 1: INTRODUCTION

1.1 Background of Study

In oil and gas industry, drilling fluid is a fluid that used to assist the drilling process. Drilling fluids, also referred to as "drilling muds", which include water-based and organic-based drilling fluids, such as oil-based and so-called synthetic-based drilling fluids. For the production of wells for the purpose of recovering hydrocarbons and other fluid and/or gaseous materials, boreholes are drilled into subterranean formations. Typically, a fluid is circulated during drilling from the surface through the interior of the drill string and the annulus between the drill string and the formation and back (as shown on Figure 1 below).

Drilling fluids have a number of important functions which are lubricating the drilling tool, suspend the drilling cuttings in the event of a shutdown during the drilling process, removing the formation cuttings from the wellbore and providing enough hydrostatic pressure to prevent formation fluids from entering into wellbore. Having regard to these functions generally the drilling fluid used for a particular drilling operation is typically designed or otherwise selected to have particular rheological and density properties.

Oil-based drilling fluids are widely used because of its efficiency in drilling deep wells at higher temperature and as well as to encounter clay or shale swelling and to avoid water contamination at basement. However, the conventional oil based drilling fluids are highly environmentally unfriendly, not readily biodegradable, and toxic in nature and thus have cumulative impact on terrestrial coastal and marine habitats.

One of the ways to encounter these problems while keeping the advantages of oil-based muds is to substitute base oil in conventional muds with biodiesel or biofuel, which is a environmentally friendly (with low sulfur content and without aromatic alkane) and easily biodegradable. In this study, biofuel drilling fluids are developed and the properties are compared with conventional oil-based drilling fluid.

Subsequently, a more specific study is done on the biofuel based drilling fluid by focusing on the effect of lime and the behaviour of the drilling fluid.

Lime is the common name for calcium hydroxide, Ca (OH) 2. It is used as a source of calcium and alkalinity in both water- and oil-based drilling fluids. Lime is used to increase pH, to provide excess Lime as an alkalinity buffer, to flocculate bentonite slurries (spud mud) for improved hole cleaning, for removing soluble carbonate ions, for controlling corrosion; and for activating fatty-acid, oil-based drilling fluids additives. The amount of lime needed to raise pH will depend on the type and concentration of solids present as well as the desired pH increase. Lime is also used for pH control in gypsum muds. In this study, the author focuses on development of a biofuel-based drilling fluid and study the effect of lime on it.

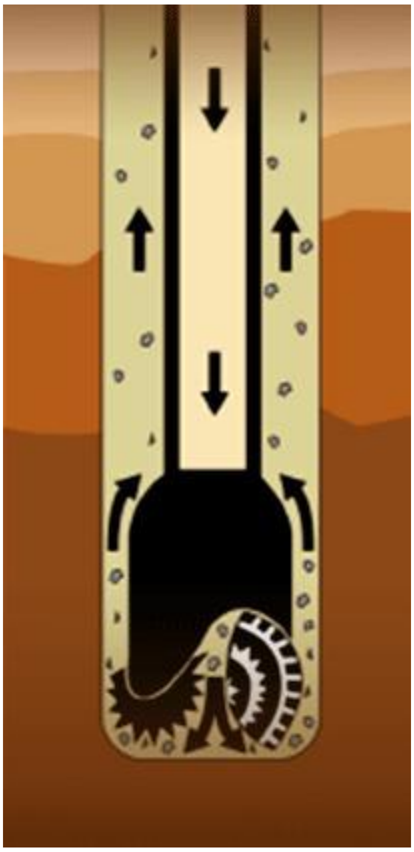


Figure 1: Cuttings in Circulation Drilling Fluids. Source: Oil and Gas UK

1.2 Problem Statement

The project consist of two main parts, first part is the preparation of the biofuel or biodiesel in the laboratory while determining its essential physical properties. Next, is the development of a suitable of bio-fuel based drilling fluid and study the effect of lime on the newly developed drilling fluid as conventional oil-based drilling fluids are effective but polluting and environmentally unfriendly. Oil based muds are considered as toxic waste therefore it cannot be disposed directly into land, river, or ocean. Thus, special care must be taken in disposal and usage of it. Many governments do not allow oil companies which don't have good waste management while drilling with oil base mud. Thus, alternative base fluids or substitute is required to produce an environmentally friendly drilling fluid which has the similar performance and compatibility as the conventionally used oil-based fluids.

The next part consists of study on the effect of lime on the biofuel-based drilling fluid in specific. Drilling fluid has many additives with specific functions and purposes. Typically, one has to introduce lime or hydroxide, $Ca(OH)_2$ as they are considered as one of the essential component in oil based drilling fluids. The lime is used to neutralize the fatty acids in the fluid, activate the emulsifiers or fatty acids, stabilizes the emulsion when present in excess, and controls alkalinity. In the field, it also neutralizes acid gases such as (H2S and/or CO_2). The biofuel or biodiesels usually contains a significant amount of fatty acids in them naturally. Thus, the reaction of lime with different concentrations would have different impact on the drilling fluid. Thus, in general the second phases of this project focuses on the study of effect of lime with different concentrations on the acquired biofuel-based drilling fluid and do comparative study for further optimisation.

Problem Identification

There is a need of an environmentally friendly and biodegradable drilling fluid in this current situation where conventional oil-based drilling fluids (OBM) are very effective but polluting, and environmental regulations continue to restrict the use of oil based muds in many areas of the world. However, these problems can be solved when biodiesel or biofuel is used to replace crude oil or other base oils in oil-based drilling fluids. A biofuel based drilling fluid is developed and the effect of lime on the biofuel drilling fluid is analyzed.

1.2.1 Significance of Project

The aim of this research is to study and determine the effect of different lime concentrations on biofuel-based drilling fluid for further optimisation.

1.3 Objectives

There are several objectives that need to be achieved when completing this project. The objectives are:

1. Preparation and characterization of the biofuel or biodiesel and its blending .
2. Formulate a biofuel-based drilling fluid which is compatible with the required specifications and test it with various drilling fluid tests
3. Study and evaluate the effect lime with different concentrations on the newly developed biofuel-based drilling fluid.
4. The results to be compared and the formulation's performances to be studied and assessed for further optimization recommendations.

1.4 Scope of Study

The research will involve in the understanding of the environmentally safe, newly developed biofuel-based drilling fluid and the effect of different concentrations of lime on it. The study of this project can be broken down to the preparation or characterization of biofuel or biodiesel and the design of biofuel-based drilling fluid and evaluating the effect of lime. The base oil used in this study will be acquired from UTP Research and Development laboratory. The concentration of lime(calcium hydroxide) will be varied and the effect on drilling fluid is studied. The properties of drilling fluid which are included when performing data analysis was based on the low end rheology reading, HTHP fluid loss, gel strength, emulsion stability, plastic viscosity, oil water ratio and yield point.

1.5 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. Moreover, study on effect of lime in particular on a newly developed biofuel-based drilling fluid is quite new to the industry and it has not yet been widely studied and discovered in depth. In this project, the author has applied fluid mechanics and drilling process theory to develop a biodiesel-based drilling fluid which is considered to be biodegradable and environmentally friendly.

1.6 Feasibility of the project within the scope and time frame

The first step in this project will be getting an introduction to the related topics by reading books, journals and research papers. Research will be done in order to better understand the properties of the biofuel or biodiesel and the design and approach for producing the biodiesel-based drilling fluid. Then, further study will be done on effect of lime on the drilling mud for optimization. The study and research will be completed by end of first semester while preparation for the materials will begin after the mid-semester break. Author plans to dedicate the first nine weeks of final year project II (FYP II) to prepare the biodiesel in the laboratory and determine the necessary properties, design the biofuel-based drilling fluid.. Then, the next three weeks will be totally focused on the study of effect of lime and optimisation of the newly developed drilling fluid

CHAPTER 2: THEORY & LITERATURE REVIEW

THEORY

2.1 Drilling Fluid

A drilling fluid or well known as mud, is a fluid that is used in a drilling operation in which that fluid is pumped from the surface (mud pit), down the drill pipe, through the bit, and back to the surface (mud screen) via the annulus. The drilling fluid must perform numerous essential functions that enhance penetration rates, reduce borehole problems and minimize formation damage. The primary functions of drilling fluid are to carry the drill cuttings loosened by the drill bit from the parent formation to the surface through the annulus and also to suspend the cuttings during a 'shutdown'. Other functions include cooling and cleaning the drill bit, reducing friction between the drill string and the borehole wall, maintaining stability for the uncased section of the borehole, preventing inflow of fluids from permeable rocks and forming a thin, low permeability filter cake which seals pores in formations penetrated by the drill bit (Gray and Darley, 1980). For different well, different drilling fluids have been developed and formulated in the oil industry to meet these functions and requirements. Drilling fluids are discussed in detail in Gray and Darley (1980). They state that there are three types of drilling fluids which are (will be explained in detail in 2.1.1.):

- **Water Based Mud (WBM)** is drilling fluid that uses water as a continuous phase.
- **Oil Based Mud (OBM)** is a drilling fluid where the continuous phase is composed of liquid hydrocarbon.
- **Synthetic Based Mud** where the base fluid is synthetic oil. This is most often used on offshore rigs because it has the properties of an oil based mud, but the toxicity of the fluid fumes are much less than oil based fluid.

The equipment in the circulating system consists of a large number of items (shown on the Figure 2, below). The mud pump takes in mud from the mud pits and sends it out a discharge line to a standpipe. The standpipe is a steel pipe mounted vertically on one leg of the mast or derrick. The mud is pumped up the standpipe and into a flexible, very strong, reinforced rubber hose called the rotary hose or kelly hose. The rotary hose is connected to the swivel. The mud enters the swivel the swivel: goes down the kelly, drill pipe and drill collars and exist at the bit. It then does a sharp U-turn and heads back up the hole in the annulus. The annulus is the space between the outside of the drill string and wall of the hole. Finally the mud leaves the hole through a steel pipe called the mud return line and falls over a vibrating, screen like device called the shale shaker. Agitators installed on the mud pits help maintain a uniform mixture of liquids and solids in the mud. If any fine silt or sand is being drilled, then devices called desilters or desanders may be added. Another auxiliary in the mud system is a device called degasser.

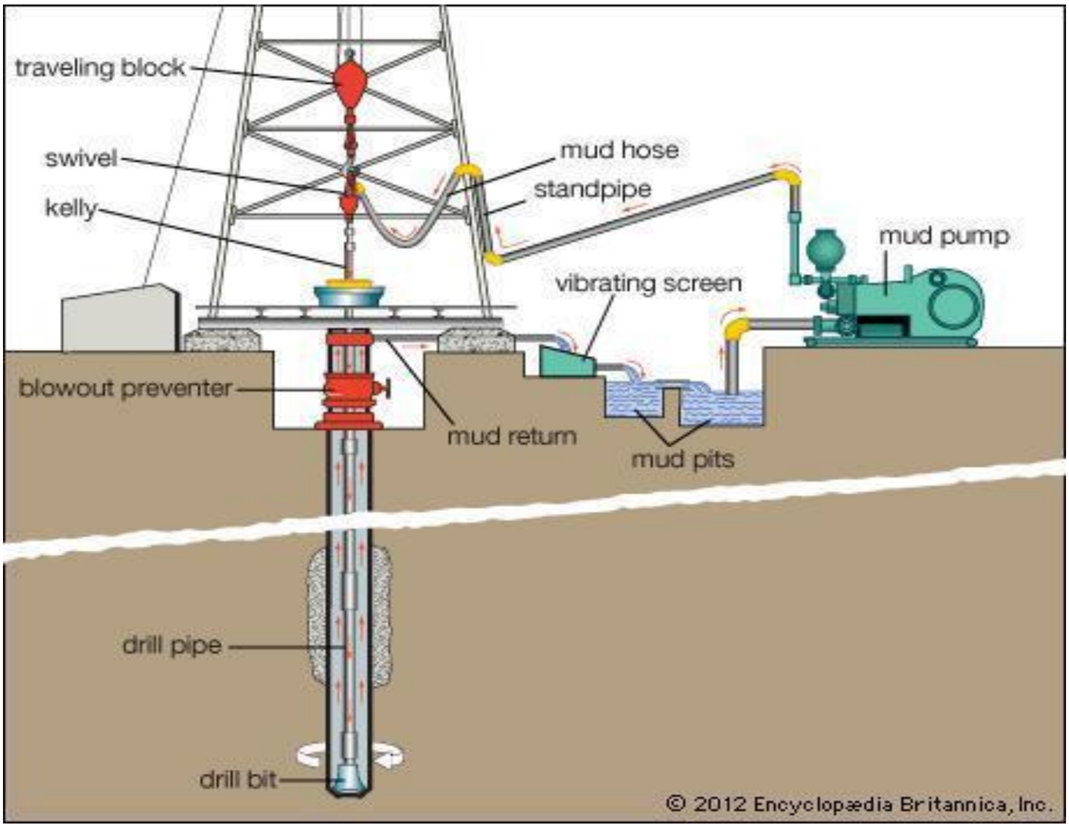


Figure 2: Circulation of drilling mud during the drilling of an oil well. Source: Encyclopedia Britannica, Inc.

2.1.1 Types of Drilling Fluids

Water-based fluids are used to drill approximately 80% of all wells. The base fluid may be fresh water, seawater, brine, saturated brine, or formate brine. The type of fluid selected depends on anticipated well conditions or on the specific interval of the well being drilled. For example, the surface interval typically is drilled with a low-density water- or seawater-based mud that contains few commercial additives. These systems incorporate natural clays in the course of the drilling operation. Some commercial bentonite or attapulgite also may be added to aid in fluid-loss control and to enhance hole-cleaning effectiveness. Water-based muds fall into two broad categories: nondispersed and dispersed (Oilfield Market Report, 2004).

Oil-based fluids are selected for their superior temperature stability, lubricity and hole stabilizing attributes (Caenn, 1996). Oil based mud's has been defined as a system the continuous or external phase of which is any suitable oil. At the present time, there are two mud systems the external phase of which are true oil mud's and invert emulsion muds. Oil based drilling fluids are very useful to drill through all types of shales, salt, gypsum and other difficult strata. It is often the mud of choice for drilling high-pressure, high-temperature zones (Sclumberger, Oilfield Glossary). Although oil muds have unique properties that are difficult to match with those of water muds, their use also causes some difficulties such as:

- High costs- The base fluids used to formulate oil muds are usually more costly than water. For example, saraline and sarapar.
- Special handling- Normally, oil muds are prepared in a liquid mud plant to ensure proper formulation and conditioning before pumping down hole. Besides, totally enclosed mud handling systems on the rig are essential to the proper operation of oil muds. Catch pans and proper disposal containers are required on the rig so that the muds are not spilled on the ground or into water bodies causing contamination.

- Environmental concerns- Probably the most important aspect driving the search for oil mud replacements is the environmental concerns associated with oil muds, especially diesel muds. It has been found that diesel is very harmful and toxic to various organisms (Caenn, 1996). Besides, tend to have poor environmental performance in terms of their ecotoxicity and their tendency to persist in cuttings piles (Department of Industry and Resources, Western Australia, Drilling Fluids Management 2006). This led to the development of mineral oil muds and then, in the early 1980s (Baroid, 1994), to modified vegetable oil muds. The latter oils, however, are much more expensive than either diesel or mineral oil.

Synthetic-based fluids are intended to replace either diesel or mineral oil, in oil-based fluids. There are a variety of distinct chemical materials available to replace the regular oils those are used in making drilling fluids. The particular fluid that is chosen depends on many factors, not on the least of which in cost. The toxicity of diesel oil is due to its aromatic content. All of the diesel replacements are aimed at either eliminating or minimizing the aromatic content, thereby making the material non-toxic or less toxic. As long as the material is within the guidelines set by local regulatory toxicity tests, it can be used in a drilling fluid.

Biodegradation of a material depends on the chemistry of the molecule of the base fluid. In general, that material which contains oxygen within their structure degrade easier. However, degradation is highly dependent upon a specific condition that impacts a fluid. Even though laboratory tests do not reflect the conditions found at the bottom of sea, they are the only way of evaluating these materials and determing their relative toxicities. There are four chemical types that are being used to replace diesel or mineral oils in muds such as esters, polyalphaolefins, ethers and also detergent alkylates. Two partially water-soluble materials, methyl-glucoside and polyalcohols, are also under development for oil replacements. For example, on esthers development Baroid’s Petrofree was the first commercially available substitute for oil muds (Carlson, 1994).

Biofuel-based drilling fluid

Biofuels are generally known as non-toxic and environmentally safe fuels, and they can be used for many applications to replace the conventional fuels such as diesel and mineral oils. One such application is for petroleum oil rig drilling in oil-based mud drilling applications. In the past, the usage of oil especially diesel oil as the continuous phase of oil-based drilling mud was widespread when drilling through sensitive producing formations and troublesome shale zones. However, diesel oil is harmful to the environment- particularly the marine environment in offshore applications and due to this extensive legislation exists in many countries to regulate this form of oil pollution. Mineral oils have been widely used as alternatives to diesel oil. Subsequently, various types of low-toxicity oils were introduced to replace the more toxic mineral oils and diesel oil-based mud system, which has proven toxic to the environment.

The use of biodiesel, such as palm oil derivative or oil from other biomass, could be considered as an alternative base fluid that is harmless to the environment. Biodiesel, defined as the mono-alkyl esters of vegetable oils or animal fats, is an environmentally attractive alternative to conventional petroleum diesel fuel (petrodiesel). Produced by transesterification with a monohydric alcohol, usually methanol, biodiesel has many important technical advantages over petrodiesel, such as inherent lubricity, low toxicity, derivation from a renewable and domestic feedstock, superior flash point and biodegradability and negligible sulfur content (Bryan R. Moser., 2009).

Tests have been undertaken to evaluate the characteristics of biodiesel as the base fluid in the oil based mud and its toxic effect on marine life. The results obtained showed that biodiesel fuels are suitable alternative to formulate oil-based drilling fluids with the necessary rheological properties. The promising qualities of these methyl esters are that they have high flash point, are non-toxic, and have good emulsion stability. In addition, they are cheaper than any mineral oils (Yassin et al., 1991). The main literature reviewed in the study of biodiesel based-drilling fluid would be an article on biofuel properties from the book entitled Applied Energy: An Introduction by Mohammad Omar Abdullah.

2.1.2 Drilling Fluid Physical Properties

Steve Devereux (1998) **s**tated that the drilling fluid properties are important to ensure the mud quality has not deteriorated and it should be treated properly if the mud quality is declined. Moreover, the mud quality must be regularly tested at the site by its specific recommended API 13B standard procedures. The properties are:

- **Density**

Mud density is widely known as mud weight in industry. This is important in maintaining well control. It is because the mud density will provide an adequate hydrostatic pressure to prevent the walls from caving in and formation fluids entering into the wellbore. In most cases, mud pressure should be higher than formation pressure to serve its function efficiently.

- **Sand Content**

There will be a presence of abrasive solid called sand in the mud and high sand content will increase wear on pumps, valves, and other equipments. However, all solids in the mud will contribute to mud abrasiveness. So, it is advisable to keep the solid content of the mud as low as possible.

- **Fluid Loss**

The fluid loss property is an indication of the mud ability to forms an effective seal against permeable formation. The formation of filter cake indicates the amount of water lost from the mud to the formation. High fluid loss mud will build up a thicker, stickier wall cake that is likely to lead to problems such as differential sticking. Ideally the mud cake should build up a thin, tough, and impermeable fairly.

2.1.3 Rheological properties of drilling fluid

Rheology refers to the deformation and flow behavior of all forms of matter. The rheologic measurements made on fluids as discussed below helps to determine how this fluid will flow under a variety of different conditions. For drilling fluids, there are 3 parameters measured which are:

- **Plastic Viscosity**

Plastic viscosity is the part of flow resistance in a mud caused primarily by the friction between the suspended particles and by the viscosity of the continuous liquid phase (Principle of Drilling fluid Control). Plastic viscosity is usually regarded as a guide to solids control. PV increases when the volume percent of solids increase or decrease when the size of particle decreases. It also represents the viscosity of mud. Low PV means mud capable drilling rapidly. High PV means the mud is too viscous which mean we have to dilute the mud so that the pump can pump the mud.

- **Yield Point**

Yield point is the measure of the electrochemical forces or attractive forces in the mud under flow conditions (Aminuddin 2006). These forces depend on surface properties of the mud solids, volume concentrations of the solids and electrical environment of the solids. This parameter helps evaluate the ability of mud to lift cuttings out of annulus.

- **Gel Strength**

Gel strength is a function of inter-particle forces. An initial 10 seconds gel and 10 minutes gel strength measurement give an indication of the amount of gellation 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.

2.1.4 Drilling Fluid Chemical Properties

Chemical properties can have a wide range of effects on drilling mud. Often chemicals are used to treat and adjust the mud so that control of other drilling fluid properties can be achieved. The chemical characteristics of the mud are mostly determined by wellbore stability considerations of the formations drilled through in a particular borehole section. One of the most important chemical properties that need to be considered is pH value. The control of pH value is needed to keep pH of mud high (between 9.5 – 10.5) to prevent corrosion.

2.1.5 Drilling Fluid Additives

Drilling fluids contain a variety of specialty chemicals (called 'additives') each having a different purpose. For example:

- Killing bacteria and adjusting pH (West et al., 2006)
- Controlling viscosity, reducing fluid loss to the formation and inhibiting equipment corrosion (Ghazia, 2011).

The additives in drilling fluids are adjusted according to the physicochemical conditions of geological formations which invariably change with depth (Ghazia, 2011). Drilling muds typically have several additives. (Air and foam fluids typically do not contain many additives because the additives are either liquid or solid, and will not mix with air and foam drilling fluids.) The following is a list of the more significant additives (OSHA USA):

- Weighting materials, primarily barite (barium sulphate), may be used to increase the density of the mud in order to equilibrate the pressure between the wellbore and formation when drilling through particularly pressurized zones. Hematite (Fe_2O_3) sometimes is used as a weighting agent in oil-based mud's (Souders, 1998).
- Corrosion inhibitors such as iron oxide, aluminum bisulfate, zinc carbonate, and zinc chromate protect pipes and other metallic components from acidic compounds encountered in the formation.

- Dispersants, including iron lignosulfonates, break up solid clusters into small particles so they can be carried by the fluid.

- Flocculants, primarily acrylic polymers, cause suspended particles to group together so they can be removed from the fluid at the surface.

- Surfactants, like fatty acids and soaps, defoam and emulsify the mud.

- Biocides, typically organic amines, chlorophenols, or formaldehydes, kill bacteria and help reduce the souring of drilling mud.

- Fluid loss reducers include starch and organic polymers and limit the loss of drilling mud to under-pressurized or high-permeability formations.

2.1.6 Lime

Lime or better known as hydrated lime and slaked lime are all common names for calcium hydroxide [$Ca(OH)_2$]. It is used as a source of calcium and alkalinity in both water- and oil-base drilling fluids. Lime, a widely available commercial chemical, is an economical source of calcium (Ca^{2+}) and hydroxyl ions (OH^-). Drilling fluid applications for lime include: increasing pH; providing excess lime as an alkalinity buffer, flocculating bentonite muds, removing soluble carbonate (CO_3^{2-}) ions, controlling corrosion, and activating fatty-acid and oil-base mud additives. A sufficiently optimum lime value used to activate the emulsifiers is often essential factors for the successful performance of a drilling fluid. The activation of fatty acids in the emulsifier's, aids in soap building in drilling mud's, directly related to sufficient emulsion formation and stability. Limiting the lime value can also impede the performance of the drilling fluid as it affects the emulsion stability and consequently causes more free water loss during HTHP filtration test. The relative importance of the effect of lime depends on the base fluid and emulsifiers used as well (as of this case). Besides, an alkaline pH which is buffered by excess lime will prevent acidic conditions from occurring which can lead to accelerated corrosion from acid gases. The solubility of lime increases with increased salinity, but decreases with increased calcium, increased pH and increased temperature. Normal treatments for lime depend on the system. The three levels of lime concentration are often described as:

- Low Lime: 0.5 – 2.0 lb/bbl → (1.43 – 5.7 kg/m3)
- Medium Lime: 2.0 – 5.0 lb/bbl → (5.7 – 14.3 kg/m3)
- High Lime: 5.0 - 15.0 lb/bbl → (14.3 - 43.0 kg/m3)

Lime precipitates soluble carbonate ions as calcium carbonate (CaCO3) as follows:
Ca(OH)2 + CO32– ® CaCO3 ¯ + 2(OH–) ………. (at pH >10.3)

Lime (lb/bbl) = CO32– (mg/l) x 0.000432 x Fw
Water-base: Excess Lime (lb/bbl) =0.26 [Pm - (Fw x Pf)]
Oil-base: Excess Lime (lb/bbl) = POM x 1.3

Where:

Fw = Water fraction from retort analysis (% water/100)

(Source: MI Drilling Fluids)

2.1.7 Properties of Palm Fatty Acid Distillate (PFAD)

Palm Fatty Acid Distillate (PFAD) is a by-product of crude palm oil refinery plant. PFAD is a light brown semi-solid at room temperature melting down to a brown liquid on heating. The main component of Palm Fatty Acid Distillate (PFAD) is free fatty acids, which are oleic, stearic and palmitic. It is used for many industries such as soap industries, animal food industries and also is used as raw materials for bio-diesel and chemical industries. It has also been used widely in pharmaceutical industries where vitamin E has been extracted commercially from PFAD for encapsulation (Gapor et.al., 1988). For the past few years, many researchers have started for solutions on challenges faced by bio-diesel industry on Palm Fatty Acid Distillate (PFAD). Palm Fatty Acid Distillate (PFAD) is potentially a valuable, low-cost raw material for bio-diesel production. Palm Fatty Acid Distillate (PFAD) also a "food vs. fuel" argument that is much debated non-issue since Palm Fatty Acid Distillate (PFAD) is generally sold as a industrial fatty acids source for non-food requests. Palm Fatty Acid Distillate (PFAD) has also been used as power plants and industrial boilers fuel.

PFAD (Palm Fatty Acid Distillated)

Figure 3: Crude and biodiesel palm fatty acid distillate

PFAD also provides a source of value-added co-products for the biodiesel producer. PFAD contains 72.7–92.6% FFA, with a small amount of unsaponifiable components (1–2.5%) and the remainder neutral oil. The figure below shows some of the general characteristics of Malaysian PFAD. (Source: Bonnie, T.Y.B., and Y. Mohtar, Characteristics and properties of fatty acid distillates from palm oil, *Oil Palm Bulletin 59*:5–11 (2009).

Table 1: General characteristics of Malaysian PFAD

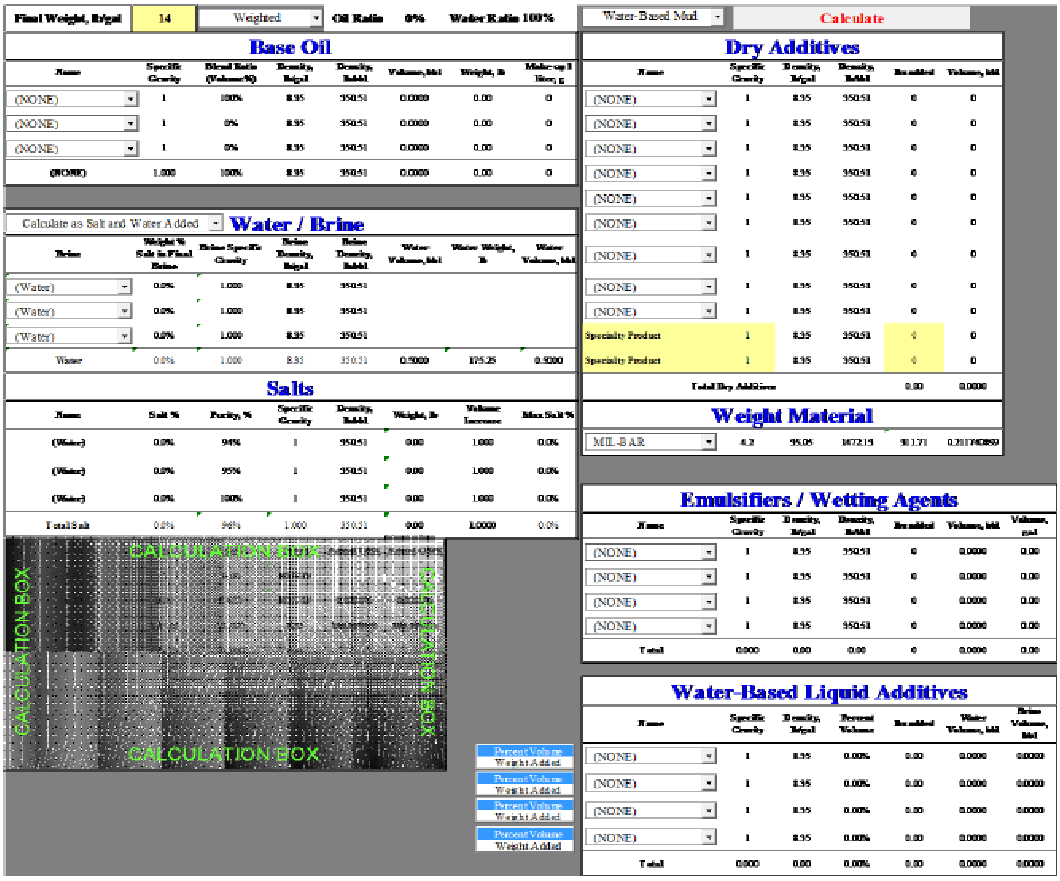
	Mean	Range
FFA—palmitic (%)	86.4	72.7–92.6
Unsaponifiable matter (%)	1.61	1.0–2.5
Saponification value (mg KOH/g)	209.5	200.3–215.4
Titer (°C)	46.7	46.0–48.3
Specific gravity @ 50°C (g/cc)	0.8725	0.8640–0.8880
Water content (%)	0.104	0.03–0.24
Iodine value, Wijs (g/100 g)	54.8	46.3–57.6

[a]PFAD, palm fatty acid distillate; FFA, free fatty acids. Source: Bonnie and Mohtar (2009).

PFAD biodiesel is an alternative fuel similar to conventional or fossil diesel which is produced from vegetable oil, and animal oils/fats. The largest possible source of suitable oil comes from oil crops such as rapeseed, soybean or palm etc. Biodiesel is a completely natural, renewable fuel applicable in any situation where conventional petroleum diesel is used. No modifications on engine are needed. Even though “diesel” is part of its name, there is no petroleum or other fossil fuels in biodiesel. Biodiesel is 100% fatty acid based. This environment-friendly fuel reduces tailpipe emissions, visible smoke and obnoxious odors. Biodiesel can also be used in blends with conventional diesel while still achieving substantial reductions in emissions. Technically, biodiesel is Fatty Acid Methyl Ester (FAME). It is formed by replacing the glycerol from each triglyceride molecule of vegetable oil with methyl from methanol (that is by the reaction of free fatty acid and methanol). The FAME is similar to petroleum diesel fuel to a diesel engine. But there is some notable difference. The biodiesel molecules are simple hydrocarbon chains, containing no sulfur, ring molecules, or aromatics that are associated with fossil fuels. Biodiesel is made up of almost 10% oxygen, making it a naturally “oxygenated” fuel

2.1.8 Mud Formulation

Firstly, the mud formulation for oil/synthetic based mud is created using the mud formulator shown in figure below. The mud formulator is an excel spreadsheet utilized to calculate the appropriate amount of products to be used to mix one lab barrel of mud which is almost 350ml in the laboratory. The final weight, type of mud, products such as weighting material, emulsifiers, viscosifiers, fluid loss agent and others are keyed into this spreadsheet and calculated.



Next, the base fluids and products are weighed according to the formulation calculated. The chemicals are then mixed according to the mixing time and order. In oil/synthetic based mud, the emulsifiers are commonly added first into the base fluid such as base oil, followed by the viscosifiers, fluid loss agent, lime, brine and finally the weighting material and mixed for an hour with mud mixers. Once the mud is mixed, the initial properties of the mud are tested.

The properties of palm fatty acid distillate drilling fluid will be compared against conventional diesel-based mud to learn the fundamental differences and especially on effect of lime on the mud. For the physical properties of PFAD mud, the plastic viscosity, yield point, gel strength, density filtration and viscosity will be measured. Then, the properties of the PFAD based drilling fluid will be adjusted with lime concentration and additives such as emulsifiers in order to match the properties and performance of conventional diesel-based drilling fluid.

The formulations are been prepared with PFAD oil and diesel used as base for the two different drilling fluids and mixed together with formulated amount of viscosifiers, fluid loss control agents, lime, brine and barite. A constant mud weight will be chosen for all formulations and mixing will be conducted in the mud laboratory.

Mineral oil, bbl	0.50 to 0.70
Primary emulsifier, lbm/bbl	3
Secondary emulsifier, lbm/bbl	7
Oil wetting agent, lbm/bbl	2
Organophilic clay, lbm/bbl	3
High-temperature stabilizer, lbm/bbl	5
High-temperature dispersant, lbm/bbl	1
Lime, lbm/bbl	10
Water, bbl	0.05 to 0.30
Barite, lbm/bbl	0 to 600

Expected mud formulation

2.2 LITERATURE REVIEW

For the study on biofuel characterization, biofuel-based drilling fluid and effect of lime, there are several research papers that were reviewed and studies in order to understand the scope of the topic. The research done was divided into to two categories which is the design of biofuel-based drilling and study on the fluid loss property of the biofuel-based drilling fluid and improves its fluid loss characteristics by using the conventional oil-based drilling fluid as the reference.

First book reviewed was the Drilling Fluids, 1st Edition, Rotary drilling series by Kate Vand Dyke (1951). This book was reviewed for understanding on the testing of drilling fluid. Based on this book, it can be said that there are 5 basic properties that will be measured for the drilling fluid which are density, viscosity, rheology, fluid loss, and solids contents. Two main tests done on testing the fluid loss behaviour of drilling fluids are API and HTHP fluid loss test. API Fluid Loss Test (low pressure, low temperature filtration test) is a test to measure a filtration of mud with ambient temperature and 100 psi differential pressure The API test equipment is shown on the Appendix A part. The HTHP testing equipment has a heating jacket so you can heat up the drilling fluid sample to the expected wellbore temperature. This equipment is usually used to simulate fluid loss under high temperature and high pressure conditions. The mud cake thickness is also taken into account as it also affects the fluid loss behaviour. The testing procedure for HTHP fluid loss test is also included in the Appendix A section. The book also discussed bout the current available methods to measure these properties (Please refer Appendix A: Equipments and Mud Tests).

Paper entitled Biodiesel-based Drilling Fluids by Wang, M., Sun, M., Shang, H., Fan, S., Liu, M. and Liu, F. (2012) was also reviewed to study on a types of biofuels. This paper discusses the rheological properties of biodiese oil and white oil. This paper shows several tests that have been taken to test the biodise and white oil and as a conclusion, the biodese oil showed better resistance to high temperature and shale expansion and as well as better filtration control with usage of organo-lignite.

Besides that, the article entitled Investigating the Effect of Electrolytes and Temperature on Rheological Properties of Jatropha oil based mud by Fadairo, A.S, Tozunku, A.K, Kadiri T.M and Falode O.A written recently in 2012 was also reviewed to aid the project. Jatropha oil is a biofuel which has been successfully used to design a drilling fluid. This paper discusses the properties of jatropha oil in the presence of sodium chloride and pottasium chloride salts, and at increasing temperature of the muds. After some modifications are done on improving the rheological properties of the jatropha oil based mud and it was revealed that jatropha oil-based mud possesses great chances of replacing diesel oil-based muds in future.

Besides that, for the study of palm fatty acid distillate biodiesel, the paper entitled Biodisel production by esterification of palm fatty acid distillate by S. Chong Kong, C. Tongurai, P.Chetpattananondh, and C. Bunyukan, published on 30 Janurary 2007 was reviewed. Production of fatty acid methyl ester (FAME) from palm fatty acid distillate (PFAD) having high free fatty acids (FFA) was investigated in this work. The PFAD biodiesel has similarity in properties with diesel fuels. However, production cost of the biodiesel is not economical when compared to petroleum based fuels. This paper generally describes the potential of palm fatty acid distillate (PFAD), a by product from production of consumable palm oil, with free fatty acid (FFA) content of 93% wt to be used as feedstock for a continuous production of biodiesel. The figure below shows the fuel properties of PFAD biodiesel studied in this research paper.

Fuel properties of PFAD biodiesel

Properties	Unit	Test method	PFAD biodiesel[a] in this work	Biodiesel specification	
				Thai standard	ASTM D6751-02
Density at 15 °C	kg/m^3	ASTM D4052	879.3	860–900	870–900
Viscosity at 40 °C	mm^2/s	ASTM D445	4.865	3.50–5.00	1.9–6.0
Flash point	°C	ASTM D93	181	120 min	130 min
Cloud point	°C	ASTM D2500	15	–	−3 to 12
Pour point	°C	ASTM D97	14	–	−15 to 10
Distillation 95%	°C	ASTM D86	335	360 max	360 max
Water content	wt%	ASTM D6304	0.03	0.05 max	0.03 max
Ash content	wt%	ASTM D874	0.0066	0.02 max	0.02 max
Carbon residue	wt%	ASTM D4530	0.07	–	–
Acid value	mgKOH/g	ASTM D664	0.33	0.50 max	0.80 max
Copper corrosion	Number	ASTM D130	1	1 max	3 max
Ester content	wt%	TLC	99.480	96.5 min	–
Triglyceride	wt%	TLC	0.000	0.2 max	–
Diglyceride	wt%	TLC	0.058	0.2 max	–
Monoglyceride	wt%	TLC	0.462	0.8 max	–

[a]PFAD biodiesel from neutralization with 1.83 v/wt% of 3 M NaOH–H_2O solution at 65 °C for 15 min followed by transesterification process using 3.85 wt% of 0.396 M NaOH–MeOH solution at 80 °C for 15 min.

Figure 4: Fuel properties of PFAD biodiesel

Addition to that, another article reviewed on Palm Fatty Acid Distillate (PFAD) is Synthesis of Biodiesel using Palm Fatty Acid Distillate by Meshach Emmanuel Ekeoma from Federal University of Technology Owerri in November 2010. This work deals with esterification of palm fatty acid distillates to produce fatty acid methyl esters which is the biodiesel in a batch reactor, using 98 % concentrated tetraoxosulphate (vi) acid catalyst. The optimum requirement of ratio of reactants, catalyst, reaction time, and temperature was studied.

Besides that, an article was reviewed on rubber seed oil is Biodiesel production from high FFA rubber seed oil by A.S. Ramadhas, S. Jayaraj, C. Muraleedharan from National Institute of Technology Calicut, India for additional information on other types of biodiesels. This paper describes a two-step transesterification process developed to convert the free fatty acids (FFA) oils to its mono-esters. The difficulty with conventional alkaline-esterification is that they often contain large amounts of free fatty acids (FFA). The first step, acid catalyzed esterification reduces the FFA content of the oil to less than 2%. The second step, alkaline catalyzed transesterification process converts the products of the first step to its mono-esters and glycerol. The important properties of biodiesel such as specific gravity, flash point, cloud point and pour point are found out and compared with that of diesel (shown in table below). This study supports the production of biodiesel from unrefined rubber seed oil as a viable alternative to the diesel fuel.

Properties of rubber seed oil in comparison with the other oils

Property	Rubber seed oil	Sunflower oil	Rapeseed oil	Cotton seed oil	Soybean oil
Fatty acid composition (%)					
(i) Palmitic acid $C_{16:0}$	10.2	6.8	3.49	11.67	11.75
(ii) Stearic acid $C_{18:0}$	8.7	3.26	0.85	0.89	3.15
(iii) Oleic acid $C_{18:1}$	24.6	16.93	64.4	13.27	23.26
(iv) Linoleic acid $C_{18:2}$	39.6	73.73	22.3	57.51	55.53
(v) Linolenic acid $C_{18:3}$	16.3	0	8.23	0	6.31
Specific gravity	0.91	0.918	0.914	0.912	0.92
Viscosity (mm^2/s) at 40 °C	66.2	58	39. 5	50	65
Flash point (°C)	198	220	280	210	230
Calorific value (MJ/kg)	37.5	39.5	37.6	39.6	39.6
Acid value	34	0.15	1.14	0.11	0.2

Figure 5: Properties of rubber seed oil in comparison with other oils

CHAPTER 3: METHODOLOGY

3.1 Introduction

The methodology of this project covers the experimental works which include characterization of palm fatty acid distillate, blending of palm fatty acid distillate with palm olein with different ratios for economic considerations; preparation of palm fatty acid distillate based drilling fluid and studies the effect of lime on the newly developed drilling fluid. This chapter discusses on the project research methodology, methods used for property analysis, experimental procedures for all experimental works stated above, tools and chemicals needed for experiments, as well as the Gantt chart for FYP 2.

3.2 Research Methodology

3.2.1 1st Stage: Project Planning

The problems related to the project are identified and the importance of the project is determined. The objective and also the scope of study are outlined and the feasibility of the project work is making sure to be within the time frame given. The solution to the problem statement is studied and the types of materials and tools used for the experiment are also indentified through literature review.

3.2.2 2nd Stage: Characterization of Palm Fatty Acid Distillate

The palm fatty acid distillate used in this experiment is characterized first. The properties which are analyzed include the density, specific gravity, kinematic viscosity, flash point, pour point and acid value. The palm fatty acid distillate used in this experiment is obtained from UTP Research and Development Department Laboratory.

3.2.3 3rd Stage: Blending of Palm Fatty Acid Distillate with Palm Olein

The palm fatty acid distillate is blended with different proportions with palm olein in order to reduce the cost of the production as using 100% biodiesel alone could increase the cost. The kinematic viscosity of different blending of palm fatty acid distillate and palm olein are determined.

3.2.4 4th Stage: Development of PFAD Drilling Fluid

The palm fatty acid distillate drilling fluid formulation is prepared. The equipment and chemicals required for the experiments are indentified with a clear experimental procedure outlined and planned. The equipment and methods needed to analyze the properties samples are also indentified. After that, the experimental works will be conducted in the laboratory of Scomi Oiltools Sdn. Bhd to obtain the data and results.

3.2.5 5th Stage: Comparative study on effect of lime

The effect of lime on the newly developed palm fatty acid distillate drilling fluid is analyzed. Different amount of lime concentration is used to aid in this study. The comparative study of drilling fluid with different amount of lime concentration is done to obtain optimum conditions for design of an optimum drilling fluid.

The flowchart that summarizes the research methodology and project activities is shown in Figure below:

Figure 6 : Research Methodology Flowcart

Prototype

Sample Preparation

- Characterization of palm fatty acid distillate oil
- Blending of palm fatty acid distillate and palm olein
- Chemical Additives

Parameters

Sample Testing

- Physical properties.
  Yield point
  Plastic viscosity
  Density
  Gel strength
  Filtration
- Comparative study

Theory

- Wide range of differences expected to be seen between PFAD oil based drilling fluid with different concentrations of lime
- The effect of lime is expected to have major differences on the behaviour of PFAD based drilling fluid.

Outcome

Result

- Comparison between the efficiency and properties of Rubber seed oil based drilling fluid and Diesel based.
- Comparison on the fluid loss behaviour.

3.3 Analysis

Before developing the palm fatty acid distillate drilling fluid, the PFAD needs to be characterized to determine its characteristics and properties. The PFAD biodiesels needs to be characterized and analyzed for its properties and to be compared with international biodiesel standards to verify their qualities and also for other comparative studies. The property analysis covers:

1. Density
2. Specific Gravity (SG)
3. Kinematic Viscosity
4. Flash Point (FP)
5. Pour Point (PP)
6. Acid Value (FFA Content)

3.3.1 Density and Specific Gravity

The density measurement is carried out using the Anton Paar DMA 4500 M Density Meter in accordance with the testing standard of ASTM D4052. The testing temperature if the density meter is set to be 20’C. A sample of around 2ml is prepared and injected into the density meter’s inlet nozzle using the syringe. The readings for the density and specific gravity are displayed on the panel.

3.3.2 Kinematic Viscosity

The kinematic viscosity if the sample is determined using the Koehler Kinematic Viscosity Bath equipment. The equipment is a constant temperature bath series with advanced temperature control circuitry and integrated timing features for convenient, accurate glass capillary viscometry determinations which satisfy ASTM standards.

3.3.3 Flash Point

The Protest Cleveland Open Cup Instrument (CLA 5) with auntomated flash point analyzer is used to determine the ASTM D92 Flash Point of the samples.

3.3.4 Pour Point

The 1SL CPP 5Gs analyzer is used to determine the pour point through the testing methods of ASTM D25OO and ASTM D97 respectively.

3.3.5 Acid Value

The acid value is determined through the titration if potassium hydroxide (KOH) based on the official method of AOCS (Cd 3d-63) (AOCS) (Darnoko & Cheryan, 2000) used for low values of acid value.

3.4 Blending

Before the palm fatty acid distillate is used as base oil in drilling fluid, the PFAD needs to be blended with palm olein which is conventional palm oil for cost reduction as palm olein is less expensive and easily available. The kinematic viscosity of the blending is determined and expected to be as low as possible (generally below 10cp) to be suitable to be used as base oil in developing a drilling fluid. The palm fatty acid distillate and palm olein are blended in different ratios as shown in table below:

Palm Fatty Acid Distillate (PFAD) (%)	**Palm Olein (%)**
100	0
90	10
80	20
70	30
60	40
50	5
40	6
30	70
20	80
10	90
0	100

Table 2 : Different percentages blend of palm fatty acid distillate and palm olein

3.5 Mud Formulation of Base Fluid

The next vital step is the development of a new drilling fluid using the palm fatty acid distillate. The best blend of oil is selected after the characterization and analyzing the kinematic viscosity of the blended oils. Before the drilling fluid is being mixed, mud formulation should be done for each base fluid that is going to be tested.

3.6 Mud testing

Table 3 below shows the activities and mud tests done on the drilling fluid as soon as it is been mixed.

Activities	Description	
Preparation of mud formulation	- Prepare a mud formulation for the biofuel-based mud (PFAD) and diesel mud which will be for comparison purpose - Chemical Additives - Tools required (mud formulator)	
Mud to be tested with following parameters	Properties	Tools Required
	Density	Mud Balance
	Viscosity	March Funnel
	- Plastic Viscosity - Gel Strength - Yield Point	FANN (Model 35A) Viscometer
	- Filtrate Volume	High Pressure High Temperature Filter press

Table 3 Mud tests

3.7 Comparative study on effect of lime

Generally, lime used as additive has certain functions in drilling fluids such as to increase Ph, as alkalinity buffer and most importantly for activating fatty acid and oil-based drilling fluid additives. For most of oil base mud, lime (Ca (OH) 2) is used in the system in order to perform chemical reaction with fatty acid emulsifiers. Typically, 3 to 5 lb/bbl of lime are added in the drilling mud so that there are enough hydroxide (OH-1) ions to keep emulsion stability in a good shape. As for this study, palm fatty acid distillate has already contained considerable amount of fatty acid in them naturally and thus, the addition of different concentration of lime will definitely have significant impact on the behaviour of the drilling fluid. Thus, the last phase of this project focuses on the comparative study of drilling fluid with different amount of lime concentration to help in obtaining optimum conditions for design of an optimum PFAD drilling fluid.

3.8 Tools for Experiment

1. Rotary Evaporator (consists of water bath as temperature controller, vacuum pump and distillation column).
2. Koehler Kinematic Viscosity Bath equipment
3. Hot plate with magnetic stirrer
4. Anton Paar DMA 4500 M Density Meter
5. Bohlin Gemini 2 Rotational Rheometer
6. Brookfield Viscometer

7. Petrotest Cleveland Open Cup Instrument (CLA 5) automated flash point analyzer

8. ISL CPP 5Gs Cloud Point and Pour Point Analyzer

9. Mud Formulator Spreadsheet

10. Hamilton Beach Mixer

11. FANN Model 35 A (VG meter)

12. HTHP filter press

13. Electric Stability Meter (ES meter

3.9 Chemicals and Additives for Experiment

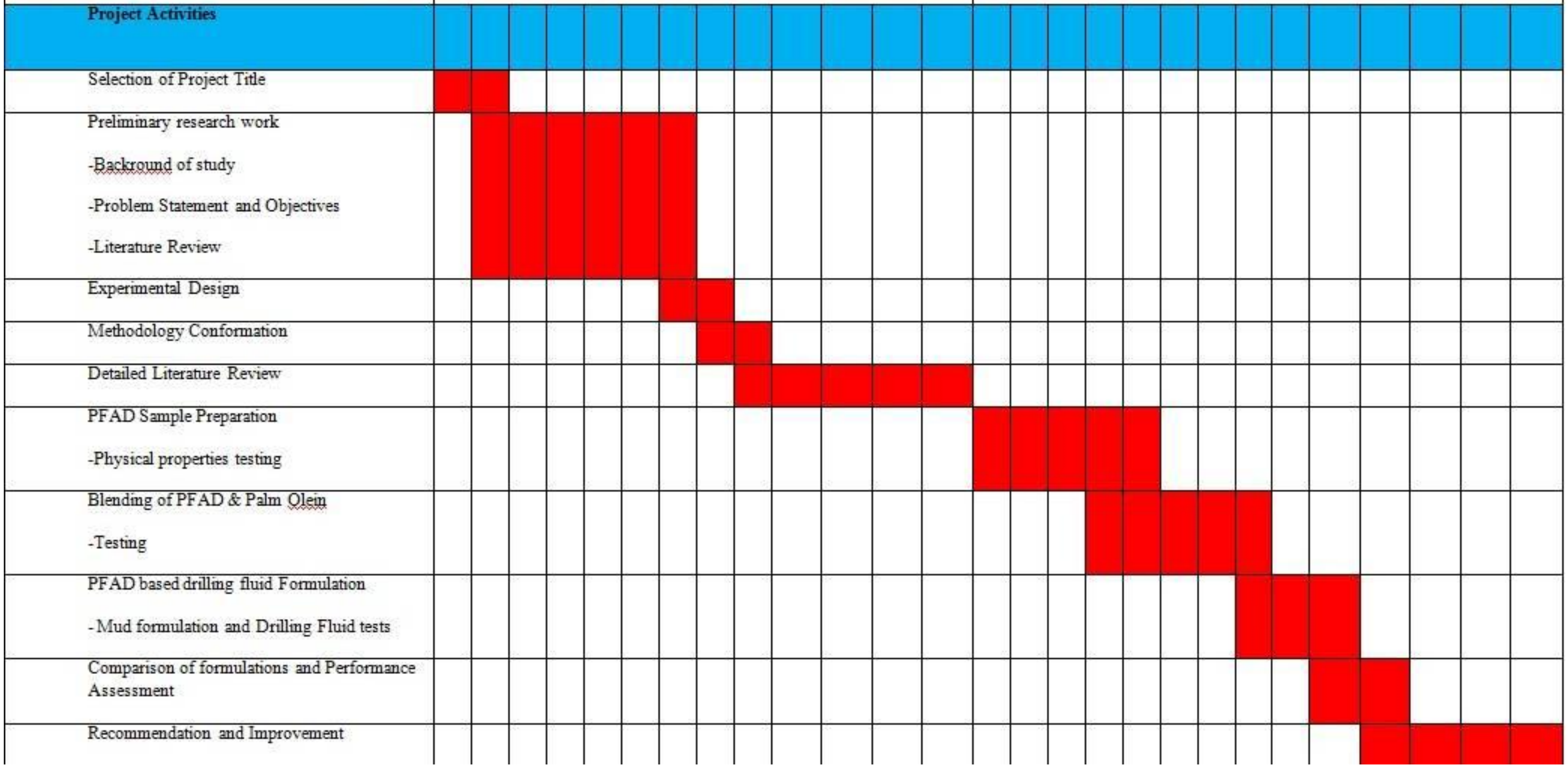
1. Palm Fatty Acid Distillate Oil (PFAD)
2. Diesel Oil
3. Primary Emulsifier (MUL P)
4. Secondary Emulsifer (MUL S)
5. Viscosifier (CONFI-GEL)
6. Fluid loss control agent (CONFI-TROL)
7. Lime
8. Calcium Chloride
9. Barite (DRILL-BAR)

3.10 Key Milestones

Some of the key events and activities that will take place during the Final Year Project are as follows:

1. Finalizing the project title from project supervisor.
2. Thorough background study on the topic given via online articles, journals and books.
3. Identification of the topic problem statement and objectives of the project.
4. Thorough research on the literature review.
5. Plan the methodology to be used in this project and the flow of the project.
6. Completion and submission of Extended Proposal to project supervisor.
7. Proposal defence with project supervisor and panel of examiners.
8. Completion and submission of FYP I Interim Report to be reviewed by project supervisor and panel of examiners.
9. Experimentation works continue and results are recorded
10. Submission of progress report to be reviewed by project supervisor.
11. Pre- SEDEX poster evaluation of project by panel of examiners.
12. Submission of dissertation (soft bound) and technical report to be reviewed by project supervisor and panel of internal and external examiner.
13. Oral presentation (VIVA) of project to be assessed by project supervisor and panel of internal and external examiner.
14. Submission of Project dissertation (hard bound) after critiqued by project supervisor and panel of examiners.

3.6. Project Gantt Chart



CHAPTER 4: RESULTS AND DISCUSSIONS

4.1 Characterization of Palm Fatty Acid Distillate

The palm fatty acid distillate used in this study is obtained from plant in UTP Research and Development Laboratory. The oil appears to be brown in colour. The oil which is obtained is first heated at 70^{o}C for around 30 minutes. This is to ensure that the methanol and excess fatty acids are removed from the biodiesel sample which is stored. This test is conducted using the rotary evaporator (consists of water bath as temperature controller, vacuum pump and distillation column) to heat up the biodiesel and remove methanol. Later, the oil is characterized by analyzing its properties such as density, specific gravity, kinematic viscosity, flash point, pour point and acid value. The results of the analysis are shown below.

Table 4 Results for the Characterization of Palm Fatty Acid Distillate

Property	Unit	Testing Method	Value
Density	kg/m3	ASTM D4052	908.25
Specific Gravity	-	ASTM D4052	0.910
Kinematic Viscosity (at 40^{0}C)	cSt	ASTM D445	4.8
Pour Point	^{0}C	ASTM D97	16
Flash Point	^{0}C	ASTM D92	132
Acid Value	M KOH/g	AOCS (Cd 3d-63)	0.33

The results obtained from the characterization of PFAD biodiesel is then compared with several common base fluid such as Sarapar 147, Saraline 200 and Diesel oil in order to analyze the properties. The table below shows the properties of Sarapar 147, Saraline 200 and Diesel oil.

Table 5 The comparison of PFAD properties and some common base fluids

Property	Unit	Diesel	Sarapar 147	Saraline 200	PFAD Sample
Specific Gravity	-	0.865	0.773	0.783	0.910
Kinematic Viscosity (at 40'C)	cSt	2.0	2.5	3.0 - 4.0	4.8
Pour Point	^{0}C	-17.7	12	-18	16
Flash Point	^{0}C	37.8	120	95	132

Based on the results obtained through characterization and comparison with Sarapar 147, Saraline 200 and Diesel oil, the PFAD biodiesel can be used as continuous or base fluid of drilling fluid. This is because; the PFAD satisfies most of the basic requirements for base oil for a drilling fluid.

Firstly, the PFAD oil has a low Kinematic Viscosity which has to be low as possible (usually less than 10) and the PFAD has a value of 4.8 which is almost the same as diesel, sarapar and saraline as shown on table above. This allows the oil-based mud to be formulated at lower water oil ratios and gives better a rheology.

The requirement for flash point is that, it should be greater than 100^0F. Higher flash point will minimize fire hazards as less hydrocarbon vapours generates above the mud. In this case, the flash point is 132^0 F, which means that PFAD satisfies the flash point requirement of more than 100^0F and it is regarded as safest of all alternative fuels.

On the other hand, the pour point should be lower than the ambient temperature. The pour point of PFAD is 16 ^{0}C thus it makes it easier for pump ability of mud from storage tanks.

As for the acid value of PFAD it is 0.33. Acid value (or “neutralization number “or “acid number”) is the mass of potassium hydroxide (KOH) in milligrams that is required to neutralize one gram of chemical substance. More acid value means free fatty acid. This free fatty acid interferes in transesterification with methanol. Lower the acid value higher the yield or quality of biodiesel. Thus, PFAD is regarded as one of the highest quality of biodiesel.

4.2 Blending of Palm Fatty Acid Distillate with Palm Olein

The PFAD biodiesel is considered to be expensive as compared to conventional diesel and palm oil. Thus, taking the economic value into considerations the PFAD was blended with Palm Olene in different ratios as shown on table below. The kinematic viscosity of different blends of palm fatty acid distillate and palm olene was determined and compared. The results of the analysis are shown in Table 6 and line graph below:

Table 6 Different mixing ratio of PFAD and palm olene and its kinematic viscosity

No	**Palm Fatty Acid Distillate (PFAD) (%)**	**Palm Olene (%)**	**Kinematic Viscosity (cSt)**
1.	100	0	4.80
2.	90	10	6.42
3.	80	20	8.06
4.	70	30	11.34
5.	60	40	17.55
6.	50	50	21.85
7.	40	60	26.15
8.	30	70	31.08
9.	20	80	35.38
10.	10	90	37.49
11.	0	100	39.04

The line graph below shows the value of increasing kinematic viscosity when a higher ratio of palm olene is used. It can be concluded that, the kinematic viscosity of oil increases as the ratio of PFAD to palm olene increases.

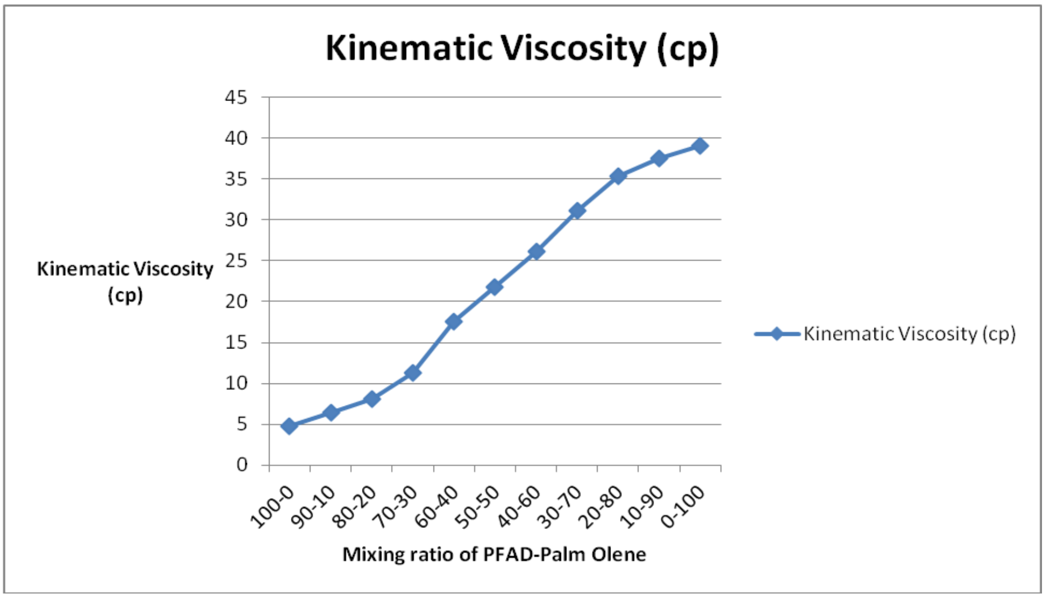


Figure 8: Kinematic viscosity

Based on the results above, it is observed that the 100% biodiesel has the lowest viscosity of 4.8 followed by other blends accordingly (refer to line graph above). The kinematic viscosity of all the conventional base oils such as diesel, sarapar and saraline are less than 10cSt which makes them an ideal continuous base fluid. The closest viscosity to the biodiesel would be the (90% PFAD biodiesel, 10% palm olene) and (80% PFAD biodiesel, 20% palm olein) which has a value less than 10.

On the other hand, all the other blends of PFAD and palm olene has values more than 10cSt which is considered too viscous to be used as base fluid in drilling fluid. Commonly, any oil with high kinematic viscosity (>10cSt) are generally not recommended because a higher oil/water ratio will be required and consequently it affects the rheological readings.

Based on the results above, 100% of PFAD biodiesel records the lowest kinematic viscosity value which is 4.8 and a value which is close to the conventional diesel oil, sarapar 147 and saraline 200. Thus, the 100% PFAD biodiesel is the best candidate out of all the blends and it has been selected to be used as base oil for this biofuel-based drilling fluid development. The other two blends, which is the (90% PFAD biodiesel, 10% palm olein) and (80% PFAD biodiesel, 20% palm olein) is recommended to be used as base oil after an optimized formulation using the 100% PFAD biodiesel is obtained. The optimized PFAD biodiesel can be used as a base or reference and this could well aid in the optimization of the two other blends in a short period of time.

4.3 Palm Fatty Acid Distillate Drilling Fluid

After verifying that, the blend that can be used as base oil for drilling fluid is the 100% biodiesel due to its low viscosity and suitable physical and chemical properties. The biofuel-based PFAD drilling fluid is mixed according to the basic mud formulation prepared. The table 7 below shows the basic mud formulation.

Table 7 Basic mud formulation

Composition	**Scomi Name**	**Mixing order**	**Mixing Time (Min)**
Palm Fatty Acid Distillate–Base oil	-	1	
Primary Emulsifier	CONFI- MUL P	2	2
Secondary Emulsifier	CONFI- MUL S	3	2
Viscosifier	CONFI-GEL HT	4	5
Fluid Loss Control	CONFI TROL	5	2
Lime	-	6	2
Water, bbl	-	7	15
$CaCl_2$	-		
Barite	DRILL BAR	8	5

The table above shows the basic mud formulation which is usually done using an excel spreadsheet utilized to calculate the appropriate amount of products to be used to mix one lab barrel of mud which is almost 350 ml in the laboratory. The final weight, type of mud, products such as weighting material, emulsifiers, viscosifiers, fluid loss agent and others are calculated accordingly. The chemicals are then mixed according to the mixing time and order and this basic mud formulation acts as the basic outline in which all the mud samples are mixed.

- Base oil - PFAD sample and Diesel (for reference purpose)
  - PFAD Biodiesel which is the base fluid used in this project.
  - Diesel which is the conventional base fluid used for reference purpose

- Primary and Secondary Emulsifier - CONFI-MUL P and S
  - Both the primary and secondary emulsifiers which are set at constant value in this study for analyzing the effect of lime
  - Only one test has different concentrations of it for comparison purpose

- Viscosifier - CONFI-GEL HT
  - The viscosifier is the Organophillic bentonite which is used at a constant value as well.
  -

- Fluid loss control agent - CONFI TROL HT
  - The fluid loss control agent that is used to form filter cake and subsequently help control loss of fluid.

- Lime
  - To activate fatty acid emulsifiers, oil-based additives and maintain alkalinity
  - Used as the manipulated variable in this project where different concentrations of lime are used to study the effects of it.

- Fresh water and $CaCl_2$
  - Used as brine to hydrate the viscosifier and reduce the volume of oil.
- Barite – DRILL BAR
  - Used to increase the density of the mud.

4.3.1 Results:

The biofuel-based drilling fluid using the palm fatty acid distillate is mixed with different concentration of lime and necessary mud tests are conducted. The concentration of primary emulsifier and secondary emulsifier is constant for the Test 1 till Test 5 except Test 6, where the primary and secondary emulsifiers are varied for comparison purpose. And, the test 7 uses diesel base oil which is used for just referencing purpose as diesel is already used as conventional base fluid and this is to aid in determining the criteria's the biofuel-based drilling fluid must satisfy after undergoing series of optimization in future.

The rheology reading is taken before and after hot-roll for 16 hours. On the other hand, the retort kit, HTHP and density checks are done after hot-roll. The results obtained are presented on the table below.

Test 1 = PFAD with **0 ppb lime**

Test 2 = PFAD with **2.0 ppb lime**

Test 3 = PFAD with **5.0 ppb lime**

Test 4 = PFAD with **6.0 ppb lime**

Test 5 = PFAD with **10.0 ppb lime**

Test 6 = PFAD with **0 ppb lime** and **1.0 ppb of CONFI-MUL P**(primary emulsifier) and
8.0 ppb of CONFI-MUL S(secondary emulsifier)

Test 7 = Diesel Oil

- **Rheology of PFAD drilling fluid before hot-roll**

Table 8 : Rheology Test Before Hot-roll results

Formulation	**Test 1 (0 Lime)**	**Test 2 (2.5 Lime)**	**Test 3 (5.0 Lime)**	**Test 4 (6.0 Lime)**	**Test 5 (10.0 Lime)**	**Test 6 (0 Lime, 1PE & 8 SE)**	**Test 7 (Diesel)**
Mud weight (ppg)	12	12	12	12	12	12	12
Rheology Temperature(◦F)	120	120	120	120	120	120	120
600 rpm	154	218	OS	OS	OS	171	96
300 rpm	111	169	250	OS	OS	133	67
200 rpm	89	156	224	OS	OS	114	55
100 rpm	61	135	191	286	291	92	43
6 rpm	21	101	136	196	196	52	27
3 rpm	19	89	131	175	177	47	25
Plastic Viscosity, cP	43	59	-	-	-	68	29
Yield Point, lb/100 ft^2	68	110	-	-	-	65	38
Gels, 10 sec	43	85	125	155	150	44	27
Gels, 10 min	48	89	129	163	158	46	33
Electrical Stability	1786	949	847	-	-	676	1267

- **Rheology, HTHP and retort kit test after hot-roll for 16 hours at 275◦C**

Formulation	**Test 1 (0 Lime)**	**Test 2 (2.5 Lime)**	**Test 3 (5.0 Lime)**	**Test 4 (6.0 Lime)**	**Test 5 (10.0 Lime)**	**Test 6 (0 Lime, 1 PE, 8 SE)**	**Test 7 (Diesel)**
Mud weight (ppg)	12	12	12	12	12	12	12
Rheology Temperature(◦F)	120	120	120	120	120	120	120
600 rpm	114	146	232	OS	OS	104	63
300 rpm	82	110	153	OS	OS	72	37
200 rpm	69	96	124	OS	OS	60	29
100 rpm	54	78	91	293	OS	46	20
6 rpm	39	60	51	213	OS	27	10
3 rpm	40	59	49	202	OS	25	9
Plastic Viscosity, cP (ALAP)	32	36	79	-	-	32	26
Yield Point, lb/100 ft^2	50	74	74	-	-	40	11
Gels, 10 sec	28	57	40	194	-	23	10
Gels, 10 min	33	60	53	198	-	26	15
Electrical Stability	1107	861	966	1321	-	531	847
OWR							
Oil,ml	28.5	22	24	23	25.5	27	31
Water,ml	8	11	12	7	7.5	10	8
Solids,ml	13.5	17	14	20	17	13	11
HPHT at 275°F and 500psi, ml (30min)							
Water,ml	1.7	2.4	0.7	0	0	0	0
Oil,ml	17.5	16	16	14.7	0.5	20.5	0.4
Total,ml	38.4	36.8	33.4	14.7	1	41	0.8

4.3.2 Discussion:

The factors that be considered as base of comparison between all different types of mud samples would be as follows:

1. Plastic Viscosity (PV) and Yield Point (YP)
2. Electrical Stability (ES)
3. Gel Strength
4. HTHP fluid loss and free water content
5.

Basically, to assist in the analysis of results, the values of criteria after hot-roll is usually taken into considerations. This is because; the hot rolling process of the fluid is conducted to assess the impact that temperatures > 250°F have on performance. Over time, high temperatures can degrade the components of a drilling fluid, and alter its performance. The fluid is hot-rolled in a pressurized cell at the desired test temperature to simulate the fluid under drilling conditions. Thus, the data's obtained after hot-rolling has a better image of the formulation's performance and reliable to be used in comparison.

4.3.4 Plastic Viscosity (PV) and Yield Point (YP)

Plastic Viscosity is the resistance to flow due to mechanical friction of the properties. It is affected by the solids concentration, size and shape of the solids and the viscosity of the fluid phase in the mud. The higher the volume percent of solids, the higher the surface area and thus the higher the PV will be. High PV can also be caused by a viscous base fluid and by excess colloidal solids.

Yield Point (YP) is defined as the electrochemical forces between particles. YP is used to evaluate the ability of the mud to lift cuttings out of the annulus. The charges on the surface of the particles dispersed in the fluid phase are what cause the electrochemical forces to present. YP is highly dependent on the surface properties of the mud solids, the volume concentrations of the solids and the ionic environment of the liquid surrounding the solids.

In general, PV indicates the flow characteristics of the mud when it is moving rapidly, whereas YP indicates the flow characteristics when the mud is moving slowly or at rest. In both cases, higher values indicate a more viscous mud. The PV and YP are determined using the following formula:

PV = 600RPM - 300RPM

YP = PV - 300RPM

The table 10 below shows the comparison of all the 5 different concentration of lime, before hot-roll (BHR) and after hot-roll (AHR).

Table10: Plastic viscosity (PV) and Yield Point(YP) results

Mud Formulation	**Test 1 (0 lime)**		**Test 2 (2.5 lime)**		**Test 3 (5 lime)**		**Test 4 (6 lime)**		**Test 5 (10 lime)**	
	BHR	AHR	BHR	AHR	BHR	AHR	BHR	AHR	BHR	AHR
PV	43	32	49	36	-	79	-	-	-	-
YP	68	50	120	74	-	74	-	-	-	-

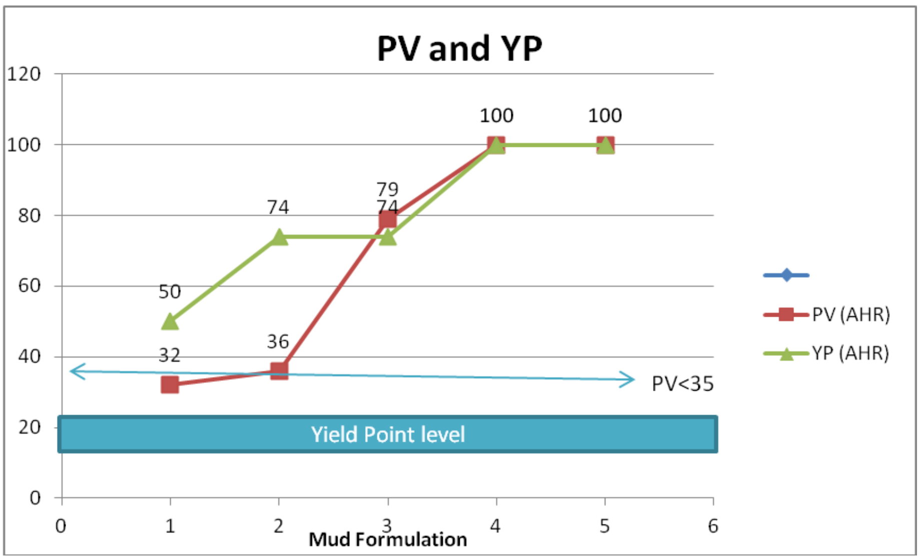


Figure 9: Graph-Plastic viscosity (PV) and Yield Point(YP) for (AHR) results

The generally acceptable range of Plastic Viscosity (PV) criteria should be below (<35) and Yield Point criteria should be in the range of (15 to 25). Thus, based on the results above, all the five mud formulations do not satisfy the requirement of the Plastic viscosity and Yield Point criteria before hot-roll. However in terms of PV after hot-roll, the mud formulation with 0 ppb lime concentration has a PV which is less than 35 and the formulation with 2.5 ppb lime has a value close to required criteria. On the other hand, the YP for all formulations BHR and AHR have values which are way above the acceptable range of 15 to 25.

This high range of PV and YP is can be due to high solid content in the drilling fluid both barite and other solids. Another reason could be excessive clay or viscosifier concentration as clay in the system chemically reacts with oil based mud causing high rheology. In this case, reducing the viscosifier concentration will help in producing stable rheology reading.

From the graph, we can deduce that the lime concentration directly affects the values of PV and YP. Both PV and YP increase with increasing concentration of lime. Basically, YP and PV increase when an invert emulsion is formed. It can be assumed that increasing lime concentration reacts with the fatty acids of the primary emulsifier and as well as the natural fatty acids present in the PFAD and forms colloidal solids or invert emulsion. Therefore, the solids concentration increases, subsequently increasing the viscosity of the drilling fluid, together with PV and YP.

4.3.5 Electrical Stability (ES)

The Electrical Stability (ES) is one of vital properties for oil based mud. It shows the voltage of the current to flow in the mud. The ES number represents mud emulsion stability. Basically, the oil base fluid is non conductive material. Therefore, the base fluid will not transfer any current. Only the water phase in the mud will conduct the electricity. If the mud has good emulsion, you will have nigh number of ES. On the other hand, if the emulsion of the mud is bad, you will have low ES value.

Table 11: Electrical stability results

Mud Formulation	Test 1 (0 lime)	Test 2 (2.5 lime)	Test 3 (5 lime)	Test 4 (6 lime)	Test 5 (10 lime)
BHR	1786	949	847	>2000	>2000
AHR	1107	861	966	1321	>2000

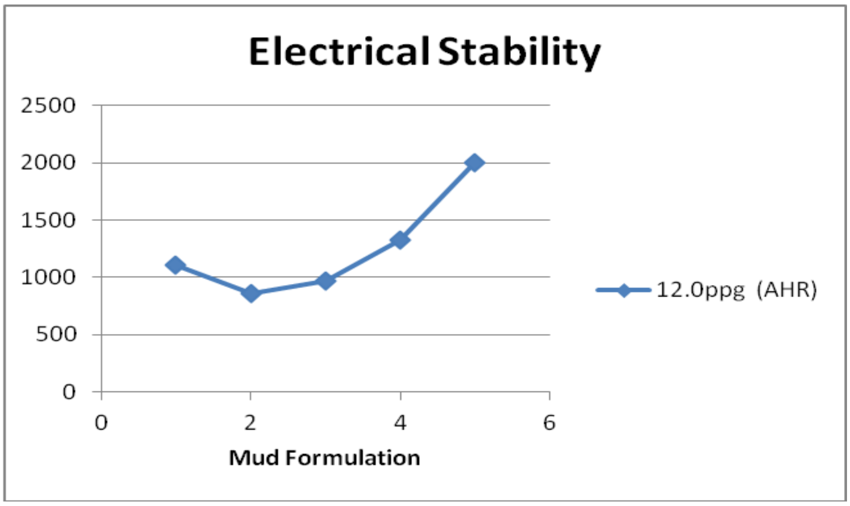


Figure 10: Graph -Emulsion stability for (AHR) results

The acceptable range of value of ES is it should be more than **(>500)**. Based on the results, all mud formulations satisfy the requirements of the Electrical Stability criteria. Thus it can be said that the emulsifier is compatible with the PFAD biodiesel and gives stable water in oil emulsion. The graph plot clearly shows that the electrical stability for all the mud samples are more than the required criteria and it keep increases with increasing concentration of lime in the drilling fluid.

4.3.6 Gel Strength

The gel strengths readings are also determined from the viscometer. The gel strengths will indicate the behaviour of the mud over time when it is in static condition, that is, when the mud circulation is stopped. The gel strength is the shear stress of drilling mud that is measured at low shear rate after the drilling mud is static for a certain period of time. The gel strength is one of the important drilling fluid properties because it demonstrates the ability of the drilling mud to suspend drill solids and weighting material when circulation is ceased. Gel strength is run for 10 seconds and 10 minutes. The difference between the two readings will then be used to indicate whether the mud has progressive gel or not. Progressive gel is a term used when the difference between the two gel strength readings are too large, which is undesirable. Progressive gels are caused by high solids concentration which leads to flocculation. If flocculation occurs, there may be a need to increase the power of the pump to circulate the mud again after being at rest, which is cost consuming.

Table 12: Gel strength for AHR results

Mud Formulation	**Test 1 (0 lime)**		**Test 2 (2.5 lime)**		**Test 3 (5 lime)**		**Test 4 (6 lime)**		**Test 5 (10 lime)**	
	BHR	AHR	BHR	AHR	BHR	AHR	BHR	AHR	BHR	AHR
10 sec	43	38	85	57	125	40	155	194	150	-
10 min	48	33	89	60	129	53	163	198	158	-

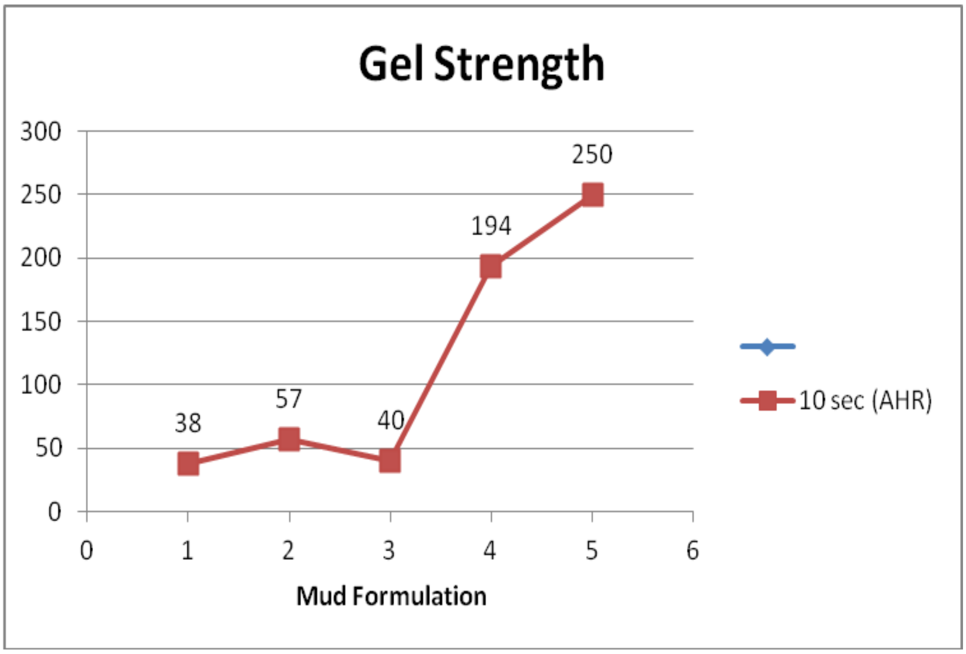


Figure 11: Graph- Gel strength 10sec (AHR) results

Based on the results above, it can be observed that the readings between the two readings of 10 sec and 10 min are not very large. Thus, all the mud samples have no progressive gel. However, the values are relatively high and if the mud has the high gel strength, it will create high pump pressure in order to break circulation after the mud is static for long time. It is commonly caused by over treatment with organic gelling material or due to build up of fine solid particles in the mud. Therefore, the mud must be treated by adding chemicals or diluting with fresh base fluid or by lowering the viscosifier concentration. The graph plotted for 10 sec after hot roll clearly shows the high value of increasing gel strength with higher concentration of lime. This may be caused by high viscosity of the drilling fluid due to reaction of lime with primary emulsifier's fatty acid and also the natural PFAD's fatty acid causing more colloidal solids formation.

4.3.7 HTHP Fluid Loss and Free Water Content

The high temperature, high pressure (HTHP) water loss is an method of testing the fluid loss in a differential pressure of 500 psi across the filtration medium at 275'F. This test is designed to give some idea of the performance of the mud under downhole conditions. The HTHP API test determines the relative amount of filtrate that may be lost and resulting mudcake thickness. In this project, in the optimization stage, the mud cake thickness is not considered and only the filtrate level and free water content are analyzed. The HTHP is done on the mud sample after hot-roll. Table 13 below shows the results of the HTHP fluid loss test:

Table 13: HTHP fluid loss results

Mud Formulation	Test 1 (0 lime)	Test 2 (2.5 lime)	Test 3 (5 lime)	Test 4 (6 lime)	Test 5 (10 lime)
HTHP Filtrate	38.4	36.8	33.4	14.7	1
Free Water	1.7	2.4	0.7	0	0

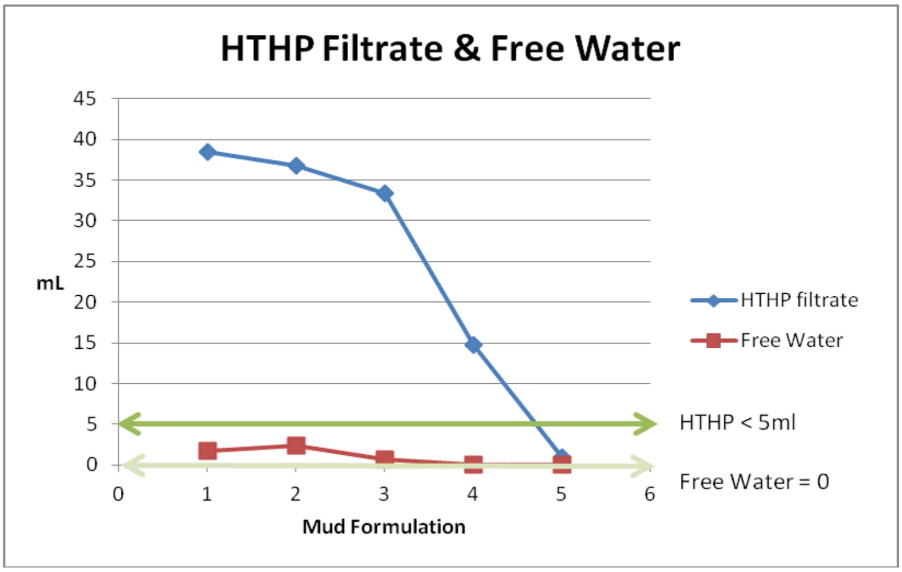


Figure12: Graph of HTHP fluid loss and Free Water Content result

The acceptable filtrate loss is less than 5ml (<5ml). Thus, it can be observed that only Test 5 which has 10 ppb concentration of lime satisfies the criterion, which is to have a HTHP filtrate of less than 5 and no free water content. All the other formulations did not satisfy the criteria and have excessive filtrate level. However, Test 4 and Test 5 have no free water content, which is an indication of good emulsion stability. This could be due to presence of right concentration of primary emulsifier, lime and ideal amount of fluid loss control agent. On the other hand, the Test 1, Test 2 and Test 3 has considerable amount of water. The problem of high fluid loss and free water content are probably due to weak emulsion stability. The solution is then to treat the mud with primary emulsifier and lime (systematically, when the mud is treated with primary emulsifier, lime must be added) and increasing the fluid loss control agent concentration accordingly. The graph plot clearly shows the effect of different concentrations of lime on the HTHP filtrate and free water content. The HTHP filtrate decreases with increasing concentration of lime while the free water content decreases with increasing concentration of lime. A sufficiently optimum lime value used to activate the emulsifiers is often essential factors for the successful performance of a drilling fluid. The activation of fatty acids in the emulsifier's, aids in soap building in drilling mud's, directly related to sufficient emulsion formation and stability. Limiting the lime value can also impede the performance of the drilling fluid as it affects the emulsion stability and consequently causes more free water loss during HTHP filtration test. Thus, it can be said that lime does play a big role in the fluid loss characteristics and emulsion stability of a PFAD biofuel- based drilling fluid which contains natural fatty acids and where further optimization is required for optimum design of drilling fluid.

CHAPTER 5: CONCLUSION & RECOMMENDATION

The aim of the project to study the effect of different concentrations of lime on a biofuel based drilling fluid, which is the PFAD biodiesel drilling fluid is achieved. The other objectives are also satisfied which includes:

1. 1. The PFAD sample was characterized and prepared to be used as continuous phase for drilling fluid in terms of physical properties comparison with conventional oil and the blending of PFAD sample with palm olein.

2. The PFAD biofuel-based drilling fluid was formulated and tested with various drilling fluid tests.

3. The effect of different concentrations of lime is studied and evaluated in terms of its plastic viscosity, yield point, gel strength, electrical stability(ES), HTHP fluid loss and free water content.

4. The results were compared and the formulation's performances were assessed for further optimization recommendations.

The comparative study covers the characteristics and performance and as well as the main part which is the effect of lime on different drilling fluid properties. Based on the results and discussions, it can be said that different concentrations of lime do play an important role in developing a new drilling fluid system, especially when using a biofuel with significant amount of natural fatty acids in them.

Based on the analysis done, it is understood that all the formulations require further optimization in order to improve its performance and efficiency of the PFAD based drilling fluid. The different concentrations of lime need further study and optimization by testing with more variety of lime concentrations and as well as the emulsifier concentrations, which are deduced to be directly related on the behavior of the drilling fluid.

Based on the objectives stated, the author can conclude that, from the characterization and properties comparison, the palm fatty acid distillate (PFAD) is found to have acceptable base oil properties. By comparison with other conventional base oils such as diesel, sarapar 147 and saraline 200, PFAD oil certainly has the criteria of a base fluid. Therefore, it can be treated further to be use commercially as a base fluid in drilling operation. However, further research by blending or repeated transesterification process to produce thinner and less viscous oil can be done for further improvement.

As for the rheology test result, PFAD, does not acquire much of an ideal base fluid to be use in drilling operation. But it can be further optimized in future to some extent by changing the composition of additives in the mud formulation and testing for better PV, YP and gel strength values. The lime which acts as an activator for the primary emulsifier, which is also a fatty acid must be controlled. Once the amount of emulsifiers is designed it is of primary importance to optimize the lime content of the mud. There are problems encountered in this design of PFAD biofuel based drilling fluid is because the palm distillate itself contains significant amount of fatty acids. Thus, the lime content must be optimized in future by varying the concentration of primary emulsifiers by maintaining a significant amount of lime. This is because, based on the results, the free water content and HTHP filtrate increases with decreasing amount of lime content. This can be encountered by probably increasing the fluid loss control agent or sustain the benefits of lime. As lime has an important function on maintaining the mud stability and from literature review, it has been held that excess of lime benefits the mud whereas lack is detrimental to its stability.

In a nutshell, this particular project experiment was very beneficial with all the objectives of this project are achieved, that is to study on the effect of lime concentration on a biofuel-based drilling fluid which is to aid in further optimization to come up with an environmentally safe biofuel-based drilling fluid that could match the conventionally available diesel and mineral oil-based drilling fluids.

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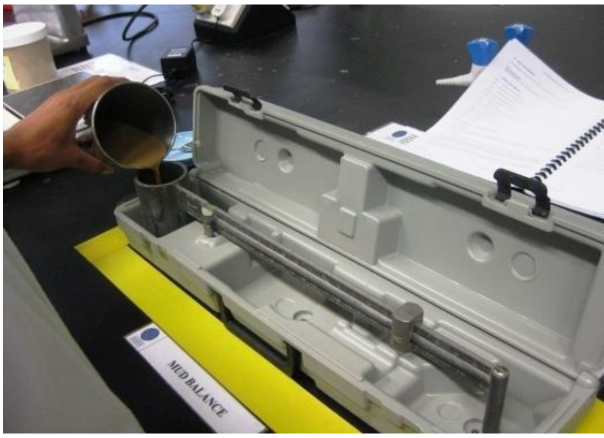
APPENDIX

Equipments and Mud Tests

Basic properties that will be measured for the drilling fluids are:

a) Mud Density Test

The Mud Balance is used for mud weight determinations and is the recommended equipment in the API standard procedures for testing drilling fluids. The mud balance is accurate to within +/- 0,1 lb/gal (or 0.5 lb/ft3, 0.01 g/ml , 10 g/l). It is designed such that the mud 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 the graduated scale. A level bubble is mounted
on the beam to allow accurate balancing.



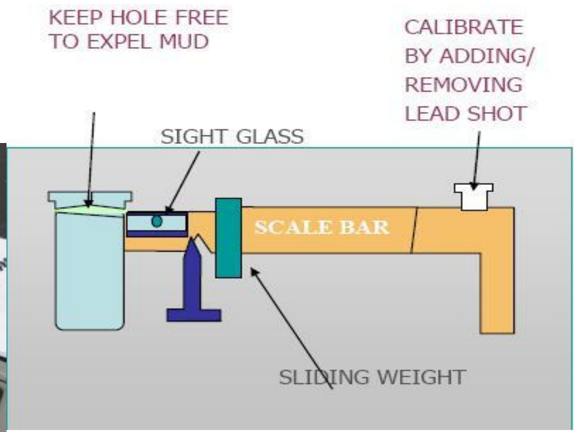


Figure 4: Mud Balance

b) Viscosity Test

The Marsh Funnel Viscometer is used for routine viscosity measurements. The results obtained are greatly influenced by rate of gel strength and density. The latter varies the hydrostatic head of the column of mud in the funnel. Because of these variations, the viscosities obtained cannot be correlated directly with those obtained using the rotational viscometers, and therefore can only be used as an indicator of mud stability, or relative changes to mud properties. The funnel viscosity is a good quick guide to whether water based mud (WBM) is thickening or thinning.

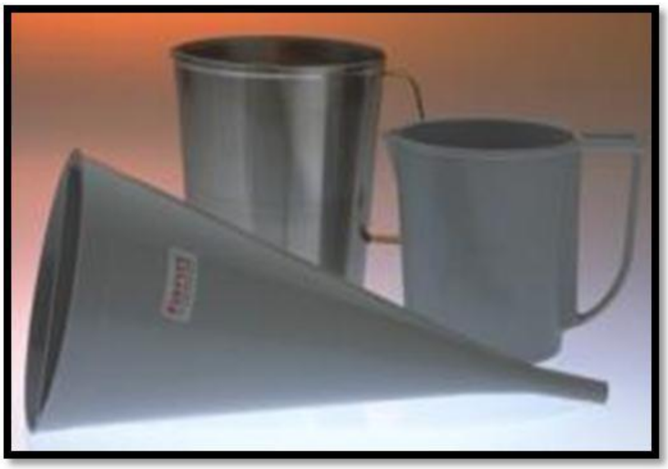


Figure 5: Marsh Funnel

c) Rheology Test

Rheology basically can be defined as the science of the deformation and/or flow of solids, liquids and gases under applied stress. In drilling, it is still a common practice to express the flow characteristics in terms of simple viscosity terms such as the constants used in the Bingham Plastic and Power Law models.

Most drilling fluids have critical yield stress which must be exceeded before flow is initiated. This characteristic follows the Bingham Plastic model closely. However, the Bingham model could not accurately describe the fluid rheological characteristics in all drilling situations due to its inability in describing fluid flow over a long shear rate range. In this case, the Power Law model is then thought to be more accurate in describing the flow characteristics of drilling fluids over the shear rate ranges experienced in the wellbore. The only downfall of this model is that it does not fully describe drilling fluids as it does not have a yield stress and underestimates low shear rate viscosity. Due to these reasons, a modified Power Law (Herschel-Bulkley model) is produced from the results of the previous models (Bingham, Power Law, and Newtonian).

The diagram below shows the differences between the Bingham Plastic, the Power Law and the modified Power Law models. The modified Power Law which is located in between the two other models shows more resemblance to the flow profile of a typical drilling mud.

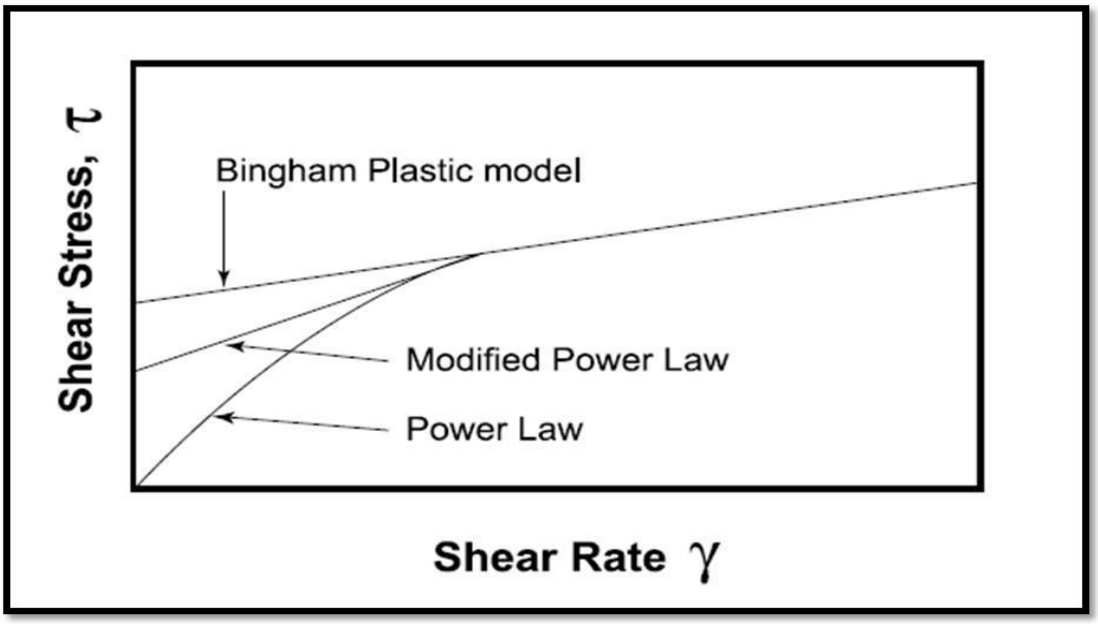


Figure: Comparison between Bingham Plastic, Modified Power Law and Power Law models. (Taken from Scomi Oiltools Handbook)

The rheology test is carried out on the viscometer unit. The viscometer has 6 different speeds (600, 300, 200, 100, 6 and 3 rpm). At each speed, a reading will be taken and then from these 6 readings, the Plastic Viscosity (PV) and the Yield Point (YP) can be determined. PV is the resistance to flow due to mechanical friction of the properties. It is affected by the solids concentration, size and shape of the solids and the viscosity of the fluid phase in the mud. The higher the volume percent of solids, the higher the surface area and thus the higher the PV will be.

Figure 6: Viscometer Unit

YP is the initial resistance to flow caused by electrochemical forces between the particles. The charges on the surface of the particles dispersed in the fluid phase are what cause the electrochemical forces to present. YP is highly dependent on the surface properties of the mud solids, the volume concentrations of the solids, and the ionic environment of the liquid surrounding the solids.

In general, PV indicates the flow characteristics of the mud when it is moving rapidly, whereas YP indicates the flow characteristics when the mud is moving slowly or at rest. In both cases, higher values indicate a more viscous mud. The units for PV is centiPoise (cP), whereas for YP is lb/100 ft^2.

The gel strengths readings are also determined from the viscometer. The gel strengths will indicate the behavior of the mud over time when it is in static condition, that is, when the mud circulation is stopped. Gel strength is run for 10 seconds and 10 minutes. The difference between the two readings will then be used to indicate whether the mud has progressive gel or not. Progressive gel is a term used when the difference between the two gel strength readings are too large, which is undesirable. Progressive gels are caused by high solids concentration which leads to flocculation. If flocculation occurs, there may be a need to increase the power of the pump to circulate the mud again after being at rest, which is cost consuming.

Figure 6: Viscometer

e) High Temperature High Pressure (HTHP) Filtrate Test

The HTHP Filtrate Test is basically of the same concept as the API Filtrate test, except that it is usually done at temperatures up to $350^{O}F$ and with differential pressure of 500 psi. As the standard API cell is twice the area of the HTHP cell, the amount of the filtrate collected from HTHP test must be doubled. The amount of fluid loss was taken at the value of cc/30min. Due to its high temperature and high pressure nature; care must be taken seriously while performing this test. This equipment help simulate the same condition in a HTHP wellbore.

f) Retort Analysis

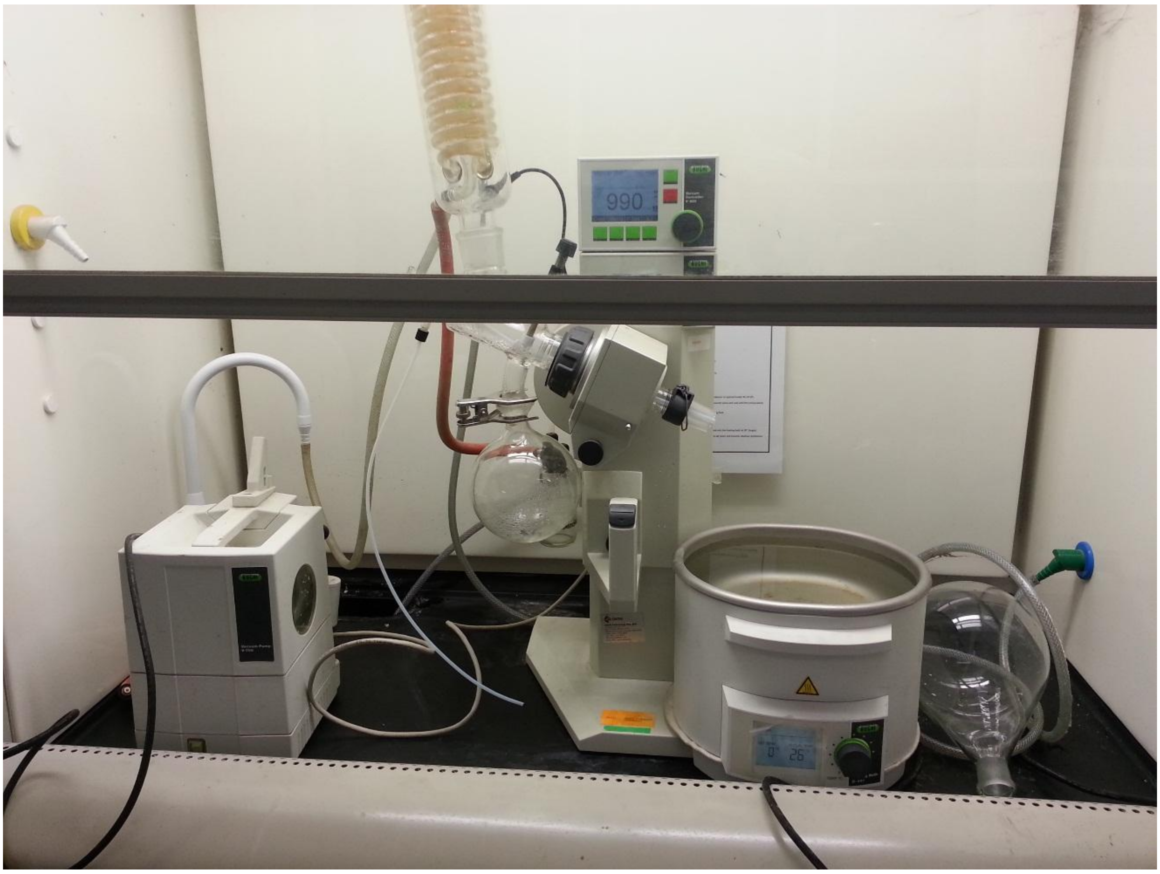
The equipment used for the retort analysis is the retort kit unit. The retort kit works like a distillation column. Each unit has a sample cup (50 ml), liquid condenser and a thermostatically controlled heating element. The test is usually carried out at $950^{O}F$ for SBM and $450^{O}F$ for WBM. Mud in the sample cup will be heated up inside the heating element. As oil and water evaporates and condensed in the condenser, solids will remain inside the cup. The oil and water will be collected in a measuring cylinder placed directly at the end of the condenser. As oil and water has different density, the amount of each liquid can be easily determined. Then, the solids content can be determined by subtracting the total amount of mud (50 ml) with the total amount of water and oil.

Equipments for preparing PFAD:

Aim / purpose : Remove methanol from the

Equipment used : Rotary Evaporation R-215

Date : 3 June 2013



Procedure

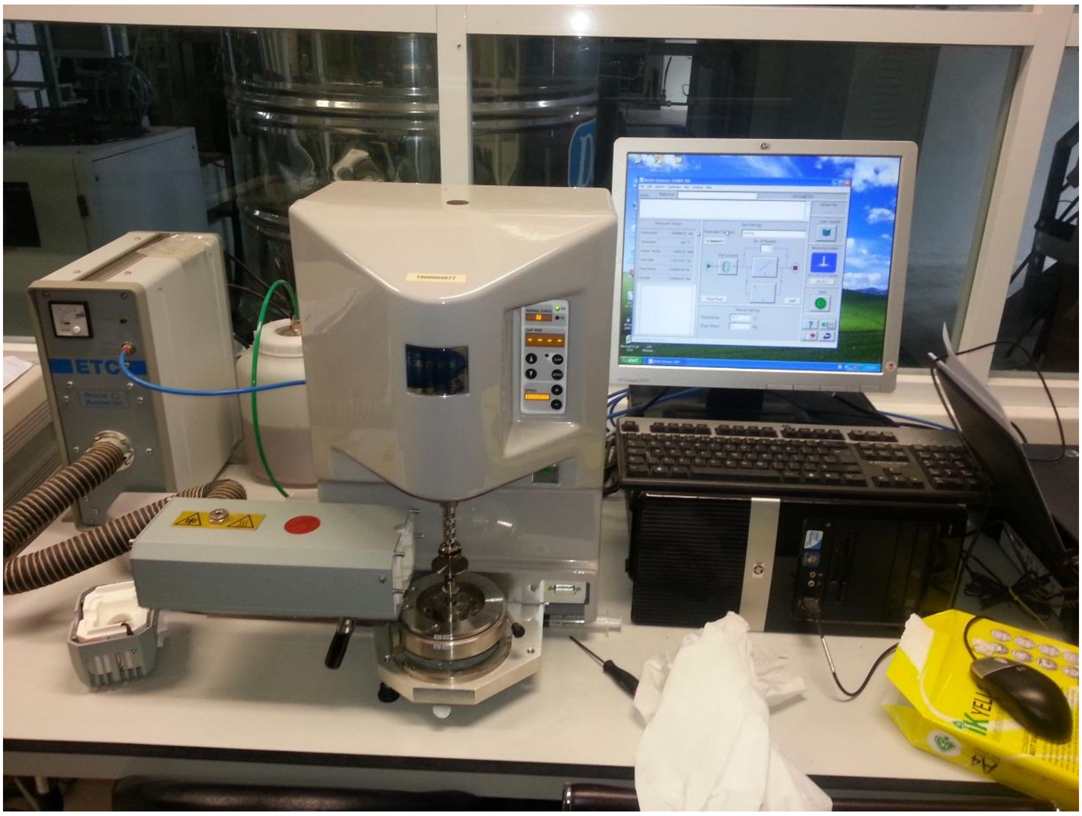
1. Sample is taken about 2 litre from the biodiesel machine
2. Then, the sample is putted inside the conical flask provided at the equipment of rotary evaporation
3. The conical flask is soak in water for the purpose of heat transferring (T = 70^{O}C)
4. The sample is leaving for about 30-45 minute
5.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

7. **Equipments used to measure viscosity**

Aim / purpose : To test the viscosity of the sample (mixed of PFAD +PO)

Equipment used : Bohlin Rheometer

Date : 11 June 2013



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 repeated with the other 10 samples with different blending amount of PFAD and Palm Oil
6. The results obtained is recorded.

Aim / purpose : To test the viscosity of the sample (mixed of PFAD +PO)

Equipment used : Brookfield Cap 2000+ Viscometer

Date : 26 June 2013

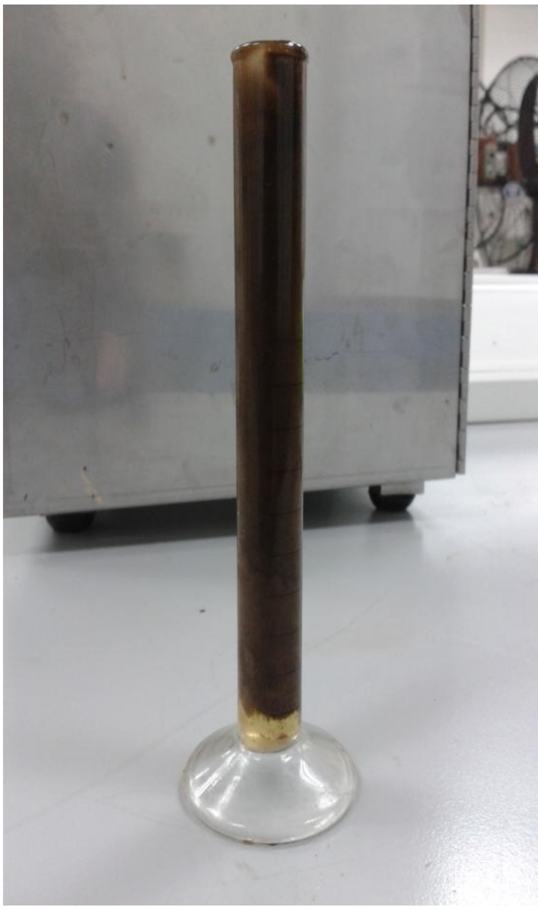
Procedure

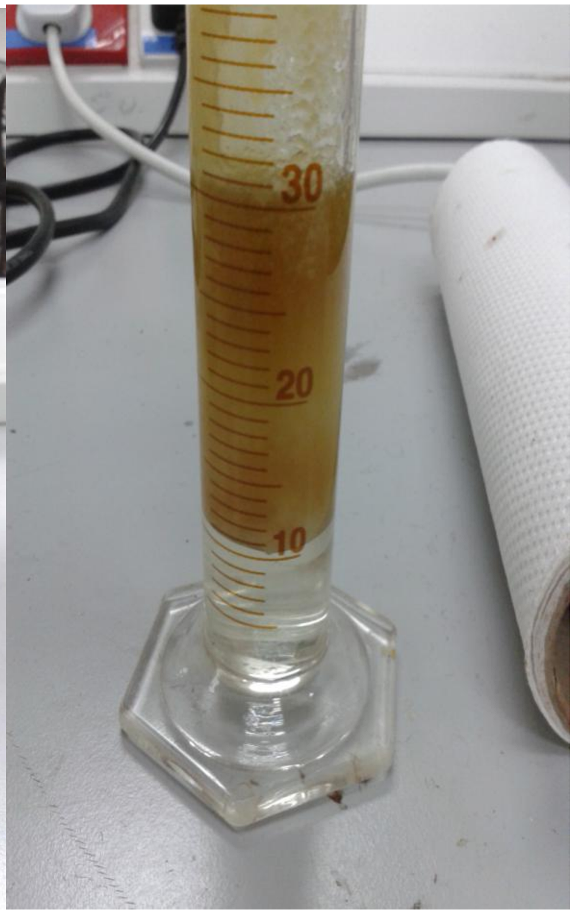
1. Small amount of sample is put on the plate
2. Temperature is set at 40^{o}C and the time is set for 60 seconds at 500 rpm.
3. After each sample is tested, the plate is cleaned carefully to prevent any scratch on the plate and contamination from occurred which may affect the reliability of results.
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 (1/2/3/4/5/6) with the other 10 samples (different blending amount of PFAD and Palm Oil)
6. The results obtained is recorded.

Photos taken during experiment:

1. **Mud in Mud Cup**

2. Mud in Aging Cell





Product HTHP Filtrate

Product of Retort Test