EVALUATION OF PALM OIL BASED FATTY ACIDS FOR SYNTHESIS OF EMULSIFIER IN DRILLING FLUIDS

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CERTIFICATION OF APPROVAL

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Arun S/O Balachandran

A project dissertation submitted to

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September 2012

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 that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(ARUN S/O BALACHANDRAN)

ABSTRACT

Drilling mud play a major role in the process of drilling and production and one of its main functions is to provide hydrostatic pressure to prevent kick. Drilling fluids could be categorised as oil based mud and water based mud. The oil based mud functionality outweighs the functionality of water based mud though oil based mud requires emulsifying agent to provide stability to it. This project titled: THE USAGE OF PALM OIL BASED FATTY ACIDS AS AN EMULSIFYING AGENT FOR DRILLING FLUIDS is to determine the ability of emulsifying agent, under high pressure and high temperature, to maintain the stability of oil based mud under high pressure and high temperature. This research will be a stepping stone for future research of the potential drilling fluid additives which is obtainable from abundant local resources. The potential usage of palm products from Malaysia as an emulsifying agent for drilling fluids is still in the experimental stage though it has high potential. Through my research I wish to look into the potential of palm oil based fatty acids able to compete with other non-biodegradable products. The problems caused by using current type emulsifier is mostly due to the nature of the product which does not decompose naturally and may cause a significant impact on the environment if proper treatment is not made before disposal. Other than that, concerning the cost of importing the product which is considerably high. This project involves a lot of lab work and experiments in order to test the effectiveness of the product and could potentially help to identify the optimization usage of palm oil based fatty acids as emulsifying agent for drilling fluids used in drilling operation. The main properties that will be investigated are the emulsion stability, plastic viscosity, yield point, gel strength and HTHP of the mud with the new emulsifier. The test results shows some interesting results in favour of the new emulsifier and will be explained in depth in the results and discussion part.

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[1.1] Project Background

Major Function of Drilling Fluids

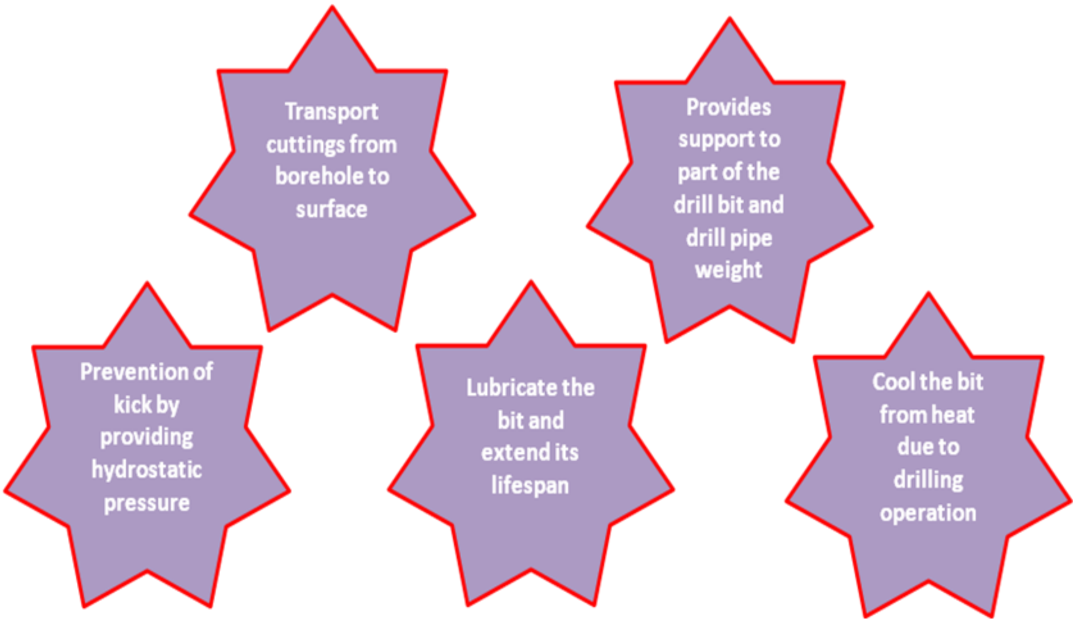


Figure 1.1: Shows some of the main functions of Drilling fluid or Drilling Mud

Drilling fluids are often used while drilling hydrocarbon formations which involves oil and natural gas wells, and on exploration drilling rigs and also on simpler boreholes such as water wells. This liquid form of drilling fluid is often known as drilling mud and can be grouped in three categories which are water based mud (which could be either dispersed and non dispersed), non-aqueous mud, which is known as oil based mud and finally gaseous drilling fluid which is made up from a wide range of gasses.

The main function of drilling fluid is to provide a hydrostatic pressure which is slightly higher than the formation pressure in order to prevent the formation fluids from entering the well and cause kick. Kick happens when the hydrostatic pressure inside the well is lower than that of the formation pressure and leads to blowout if proper well control procedures are not taken quickly. For a gas or oil influx well control procedures should be taken quickly compared to a water based influx. Other than that, the drilling fluid also functions as a lubricating agent and cooling agent for the drilling bit and prevents damages to the bit due to friction and high temperature. Drilling fluids also remove cuttings from the well and suspend cuttings while drilling is paused and the drilling assembly is brought out of the hole. The proper drilling fluid should be selected for a particular job to avoid formation damage and to limit corrosion.

Our topic of interest is for oil based mud, which have a variety of application in the drilling industry. There are two types of well fluids mainly used which are water based fluids, or water based mud and oil based fluid or oil based mud. Water based muds are cheaper compared to oil based muds and more ecologically acceptable though oil based muds have clear operational advantage particularly when it comes to drilling in high pressure and high temperature (HP/HT) or drilling deep wells because they are good friction reducing agents , the fluid is inert to the formation as they do not react with the formation unlike water based fluids which have a high composition of water, particular with clay formation, and finally the damage to the production zone is slight.

Oil based muds are mostly water-in-oil emulsions which contain 5% up to 40& by volume of a dispersed aqueous saline phase. Oil based muds are usually a composition of three types of compounds which are emulsifiers to stabilise the emulsion, organophillic clays to control the rheological properties and weighting materials which consists of barium sulphate(Barytine) which functions to adjust the density of the drilling fluid , usually used to increase or decrease the hydrostatic pressure.

What we are interested to see is how the emulsifier agent acts in an oil based drilling fluid. We encounter two major problems when using drilling fluids to drill in very deep wells. The problems are:

1. ***The emulsifying agents stabilising the emulsion must maintain the water droplets in an emulsion up to temperatures close to 200° C. If the emulsion is separated by coalescence of water droplets, it loses its rheological property.***
2. ***The emulsification agent must not only be effective but also induce a non-toxic based environment***

We are interested in using fatty acid based emulsification system and test out its ability to ensure the stability of the mud and also find out its effects to the environment.

[1.2] Problem Statement

Major Problems Encountered

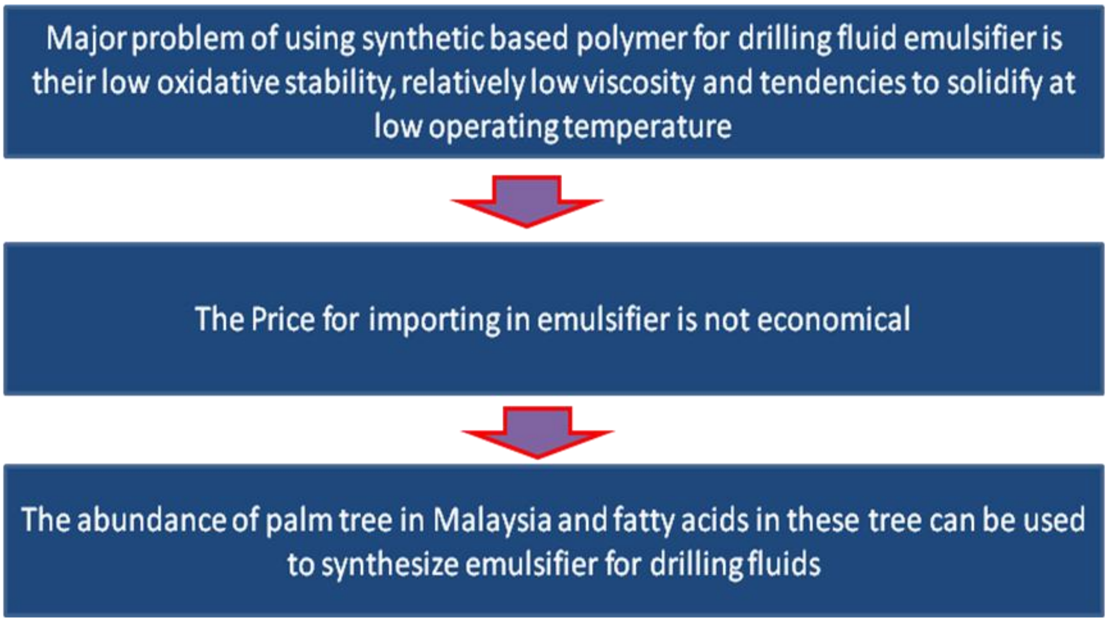


Figure 1.2: Shows the major problems encountered for this project

Our area of interest is to test the ability of palm oil based fatty acids as an emulsifiers to maintain the stability of an oil based drilling fluid. The **Schlumberger Oilfield Glossary** defines Oil based emulsifiers as “An Oil-Mud emulsifier lowers the interfacial tension between oil and water, which allows stable emulsions with small droplets to be formed.”According to the oilfield glossary, these emulsifiers will surround water droplets, like an encapsulating film, with the fatty acid component extending into the oil phase. Oil-mud emulsifier’s drops behave like a small osmotic cell where the emulsifier’s drops acts like a semi-permeable membrane which water can move but ions could not pass through. We are to test whether this stable emulsion could be achieved using emulsifiers synthesised by fatty acids from palm oil.

The problems that may arise from using emulsifiers synthesise from palm oil are such as:

- Does the emulsifier or additive able to maintain the stability of the oil based mud emulsion in high pressure and temperature (referring to deep wells)?
- Is the emulsifier produced is environmentally friendly? (Meaning is the emulsion derived from the fatty acids from palm oil and non-toxic?)

- In an economical point of view, is the cost of production of these emulsifier cheaper or expensive compared to normal diesel based emulsifiers?
- Is it feasible for the emulsifier is produced in large volume?
- Is the product having high or low biodegradability, particularly in anaerobic conditions?
- Does the product have severe impact on the seafloor sediments and food chain?

these are the few problems that have been identified when using emulsifiers synthesised from fatty acids from palm oil and of course the most important function of these emulsifier is it should be able to maintain the stability of the oil based mud emulsion under high pressure and temperature and this could only be confirmed once sufficient lab data has been obtained.

[1.3] Objectives

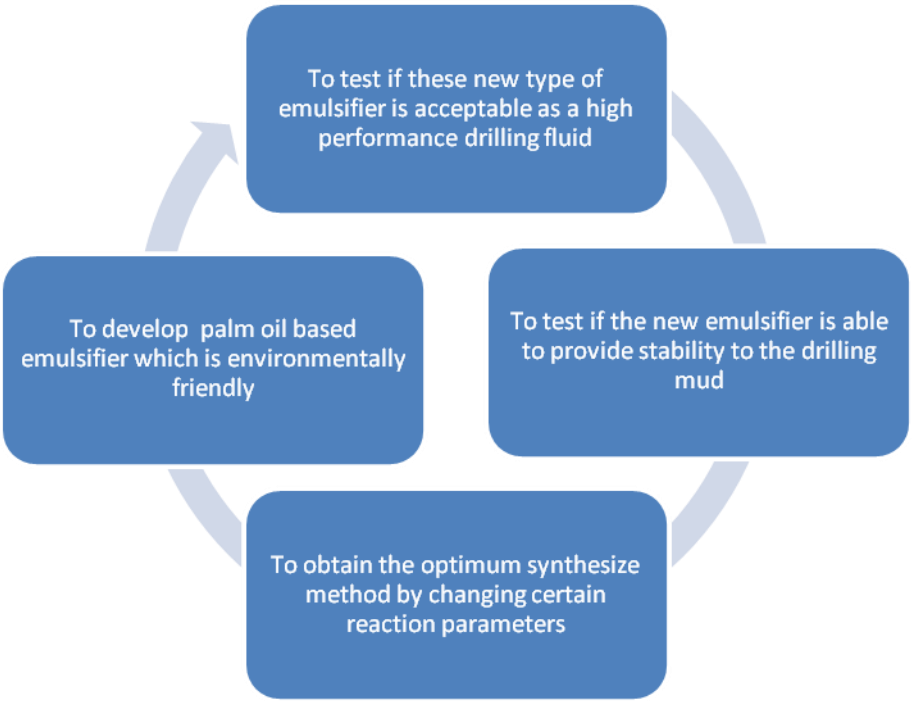


Figure 1.3: Main Objectives

Oil based drilling fluids or oil based mud require proper emulsifying agent to maintain the stability of the oil and water emulsify. the usage of palm oil based fatty acids as an emulsify is to test whether this emulsify is able to provide all the similar attributes as a commercial emulsifier and at the same time able to provide extra functionality. The objective of this experiment is to:

- ***test whether the palm oil based fatty acid can act as a substitute emulsifier from commercial emulsifiers***
- ***to test whether the emulsifier synthesised is able to provide stability to the oil based mud without affecting the rheology of the drilling fluid***
- ***to determine whether the emulsifier used is non-toxic to the environment***
- ***to determine if the emulsifier has high or low biodegradability property in anaerobic conditions***

[1.5] Scope of Study

The oil and gas industry has to react to major challenges, improving both chemical stability of the emulsifier system and its impact to the environment. The latter factor covers both occupational health and ecological hazards resulting from contact with the fluids themselves and wastes that are generated, mainly which are drill cuttings. In order to maximize technical performance of emulsifying surfactants for mud systems, it is necessary to implicate temperature stable functions such as imadazolines or amides to link the hydrophobic chain to the polar head. At the same time, it is also important to minimise ecotoxicological effects.

The development of emulsifier from fatty acids of palm oil is expected to be more environmentally friendly compared to the commercialised emulsifier from diesel. This is to promote using emulsifying agents which are non-toxic and are able to biodegrade naturally.

Other than that, from and economical point of view, Malaysia is a country rich of natural resources especially in the palm oil industry. This is one of the methods to promote the income of Malaysian economics and also to promote on buying Malaysian Made products. More people will be hired in the process of producing emulsifiers from palm oil thus incredibly reducing the number of un-employment in the country.

Therefore due to the benefit gained environmentally and for the country, I am positive that the study of using emulsifiers synthesised from palm oil will be a stepping stone for a better future for the country and the world

[1.5] Relevancy of Project

This project will help me understand in depth about the functions of drilling fluid as well as the importance of the compositions that make up a drilling fluid. These understandings are important as such to enable me to become a better petroleum engineer and also because it is related to the field I am majoring in. Other than that, I also hope that I will be able to make use of the natural resource of this country and indirectly help to promote the economics of this country. We also hope to produce a more environmentally friendly type of drilling fluid which will not cause natural problems if it is released to the environment.

[1.6] Feasibility of the Project

This project is feasible within the time frame provided as all the main result for this research is obtain through lab work. The equipments and material required to conduct this research has been prepared and the necessary party involved has agreed to provide these materials within the suggested time frame. Other than that the material are also not highly priced and is affordable from a student's perspective.

CHAPTER 2: LITERATURE REVIEW AND THEORY

[2.1] Drilling Fluids

One of the major functions of drilling fluid is in aiding the drilling of boreholes into the Earth. It is commonly used in natural gas wells and on exploration phase drilling rigs. Drilling fluid is also often known as drilling mud and can be categorized into three major groups which are water based mud (which could be either dispersed on non-dispersed), non aqueous mud or also known as oil based mud (one of most important type of drilling mud and is commonly used in drilling operation due to its high functionality) and synthetic based mud which has all the similar properties of an oil based mud but posses less harm to the environment.

The functionality of drilling fluids is as follows:

[a] Remove cuttings from well

[b] Control formation Pressure

[c] Seal Permeable Formations

[d] Cool, lubricate, and support the bit and drilling assembly

[e] Suspend and release cuttings

[f] Minimizing formation damage

[g] Maintain wellbore stability

[h] Transmit hydraulic energy to tools and bit

[i] Control corrosion

[j] Facilitate cementing and completion

Nowadays drilling into deeper, longer and more challenging wells is possible due to the advancement in the drilling technology including the usage of more efficient and effective drilling fluids. Drilling fluids are water, oil or synthetic based and the composition of each type of fluid provides a different solution in the well. It is the responsibility of a drilling fluid engineer to oversee the drilling, adding drilling fluid additives and whatever is required to be made.

In addition of considering the chemical composition of the drilling fluid and the properties of the well, a drilling fluid engineer must also consider the environmental impact when prescribing the type of drilling fluid necessary for the well. Disposal of drilling fluids after they are used is also a major challenge faced by the oil and gas industry, though recent advancements in technology has enable the methods for safe disposal and recycling of drilling fluids.

[2.2] Emulsion

Emulsion is a major part in a drilling fluid. Schlumberger oil glossary defines emulsion as a dispersion of one immiscible liquid into another through the usage of chemicals that reduce the interfacial tension between two liquids in order to achieve stability. The usage of emulsion is not limited to the Oil and gas industry but is also widely popular in other industries including food and cosmetic industries.

Some of the major advantages of using emulsion based drilling fluids are:

[1] Achievement of greater drilling depth and speed

[2] Fluid loss is reduced

[3] The drilling fluid could be handled much effectively

[4] Achieving better lubrication

According to the research done by Doyne L.Wilson, it says that excellent electric logs are obtained in emulsion drilling fluids by using normal electric survey equipments and this may be of the prime importance in a wild cat well where the operator desires a better drilling fluid than clay-water mud, but does not wish to use an oil based mud.

There are two types of emulsion which are oil in water emulsion and water in oil emulsion and the difference between these two types of emulsion is the difference in percentage of concentration of oil or water in each phase. As for oil in water type of emulsion the heavier or the carrying/dominant phase is water and oil is present as the dispersed phase while for water in oil emulsion the dominant phase is oil and the water is in dispersed phase.

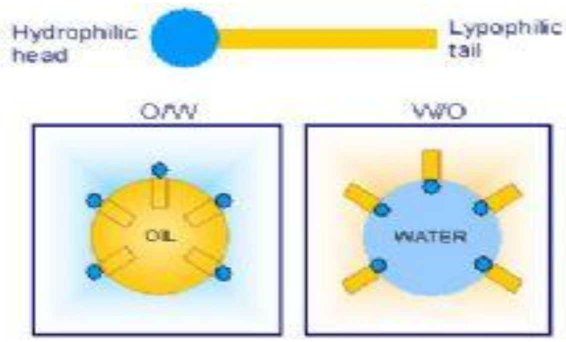


Figure 2.1: *Shows the type of emulsion; the right is a water in oil type while the left side shows a oil in water type emulsion*

[2.2.1] Oil in Water Emulsion Drilling Fluids

Water in oil type emulsions drilling fluids is used in aqueous formation to prevent damage in formation, and is also used as the treatment of oil producing formations. The advantages of using this type of emulsion drilling fluid is it does not cause any pollution to the environment if disposed. The advancement in drilling technology has open gates in optimizing the usage of water based drilling fluids. Based on a research conducted by Chee P. Tan, Calum J. Drummond and Fersheed K. Mody, the new generation water based drilling fluids that have been developed performs essentially like an oil based muds in terms of shale stabilisation. practical mud design guidelines that have been developed also can be used to optimise the drilling fluid design, in terms of mud weight, salt type and salt concentration, to manage efficiently time dependent well bore instability in troublesome shale formations.

[2.2.2] Water in Oil Emulsion Drilling Fluid

The Solids in an Oil based Fluid or oil based mud is oil wet, meaning the oil phase is the dominant phase, and all the additives are oil dispersible and the filtrate of the mud is oil. The water which is present is emulsified in the oil phase. Oil based mud can be classified as Invert emulsions and all oil mud. According to the Schlumberger Oilfield Glossary, invert emulsion is defined as an emulsion in which the oil is the continuous or external phase and water is the internal phase. Invert emulsion usually refers to oil based mud. All oil emulsion is defined as using 100% oil without any water phase. Basically the amount of oil percentage present will define the type of oil based fluid. The oil used in these types of oil based fluids can range from crude oil, refined oil such as mineral oils or diesel, or the non petroleum organic fluids which presently available. These latter type of fluids are usually known as inert fluids, pseudo-oils, non-aqueous fluids and synthetic fluids-are now considered more environmentally friendly than diesel or mineral oils.

According to the article entitled Synthetic Drilling Fluids –A Pollution Prevention Opportunity For The Oil And Gas Industry, by John A Veil, Argone National Laboratory, Christopher J Burke and David O Mosses, SBM have drilling and operational properties similar to Oil based Mud systems and are used in many situations where the properties of WBM will limit performance and the advantages of SBM are as listed:

- *Less waste is produced from recyclable product*
- *Elimination of diesel mud base lessens the pollution hazard, improves worker safety and diminish irritant properties*

- *Increases use or horizontal drilling reduces the areal extent and the environmental impacts of offshore oil and gas operation*
- *Shortened Drilling time results in reduced air emissions from drilling Power Sources*
- *Improved drilling performance decreases waste generating incidents such as pipe stuck in the hole*

Invert emulsion of oil muds is formulated to contain moderate to high concentrations of water (Amoco production company drilling fluid manual 1994). Oil based mud and synthetic based mud have a lot of advantages in functionality compared to WBM, which includes temperature stability, tolerance to contamination and corrosion protection (Dye et. al. 2006) and according to the Norwegian Oil Industry Association Working Group (1996)

[2.3] Emulsifying Agent

In the recent years, many types of additives and emulsifiers have been used to maintain the stability of OBM's emulsion. In an article by Penny Keay, emulsifier is defined as it is used whenever you want to mix two components and keep them from separating example oil and water. Emulsifier is used when one part is oil based and the other part is water based. The Schlumberger Oilfield Glossary defines emulsifier for oil based mud as a chemical used in the preparation and maintenance of an oil-or synthetic base drilling fluid that forms water in oil emulsion(invert emulsion). An oil mud emulsifier reduces the interfacial tension between oil and water, which subsequently allows a stable emulsion with small droplets to be formed. Emulsifiers can be calcium fatty acid soaps made from various reactions of fatty acids and various ethanolamine compounds. These emulsifiers surround water droplets, like an encapsulating film, with the fatty acid component extending into the oil phase. Emulsifier molecules that cannot fit around drops form clusters (micelles) in the oil phase or absorb onto solids. Examples of emulsifier available in the market are such as GLO OBM PEMUL 1000 from Global Drilling Fluids & Chemical LTD and Halliburton's SEM-7 emulsifier.

Biodegradability of drilling fluids is a major issue and has become a basic requirement for disposal of drill cuttings in offshore operations, especially in Europe. Due to their ready biodegradability, fatty acid esters are very popular. While ester enjoy advantages over synthetic based fluids in terms of biodegradability, the performance of drilling fluid still remains substanded. The ester-based fluid undergo hydrolysis in the presence of lime at temperatures exceeding 200 degrees Ferhenheit (93 degrees Celcius). According to the research done by Arvind D. Patel on the ' Negative Alkalinity Invert Emulsion Drilling Fluid

Extends the Utility of Ester Based Fluids', a new approach to the stability of ester-based fluids is anew, negative-alkalinity ester based drilling fluid that is stable at temperatures up to 350 degrees Ferhenheit (177 degrees Celcius). the thermal stability and performance characteristics of the ester based mud are superior compared to the conventional ester based mud formulations.

The surfactant used in this negative alkalinity ester-based mud is a very stable invert emulsion surfactants under slightly acidic conditions and are superior compared to conventional invert surfactants as this surfactant is able to form unstable invert emulsions in the presence of lime or reserve alkalinity.

$$R\text{-}NH_2 \underset{OH^-}{\overset{H^+}{\rightleftharpoons}} R\text{-}NH_3 \text{ (invert emulsifier)}$$

In another study conducted by Fadairo Adesina, Ameloko Anthony, Adeyemi Gbadegesin, Ogidigbo Eseoghene and Airende Oyakhire on the 'Environmental Impact Evaluation of a Safe Drilling Mud', oil extracted from the non food plant seed(jatropha and canola seeds) were used as the base fluid for drilling mud samples in laboratory, oil in water emulsion was made using oil-water ratio of 70 to 30, 200 ml of oil, 300 ml of water and 50 g of bentonite ad barite was finally added to build the density up to 10 ppg. The mud samples were formulated in three different fluids which are diesel, jatropha and canola oil based mud samples. A set of laboratory test were then conducted on the drilling fluid samples such as filtration test, pH test, viscosity and density test and the degree of safety to the environment was also taken into consideration.

The result obtained showed that jatropha oil based mud had the lowest viscosity, whic implied less resistance to flow and less fluid pressure losses. The toxicity test also confirmed that the jatropha oil was safer and less harmful plant life and soil organism compared to diesel which was the most toxic among the three samples. The overall test result proved that jatropha oil based mud pose a great chance of being among the technically and environmentally viable replacement for the convectional diesel oil based mud.

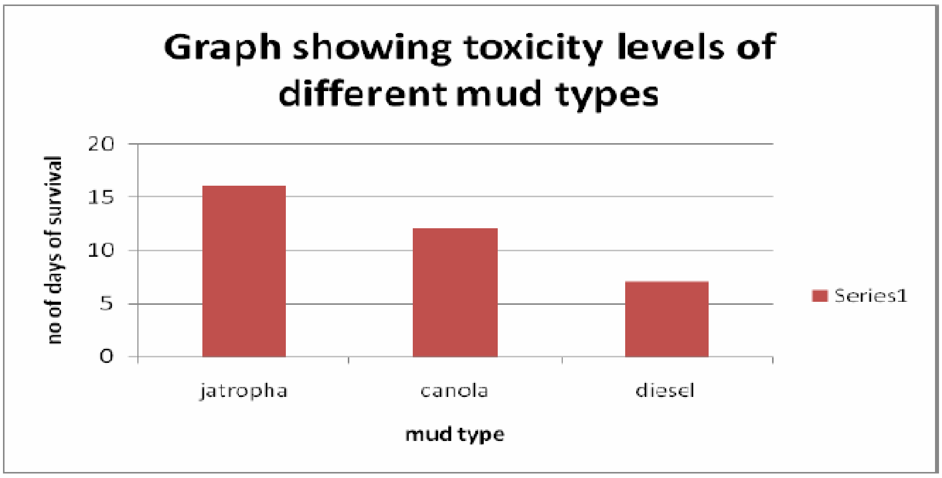


Figure 2.2: Graph showing the toxicity level of each type of oil based mud tested

In another research about the usage of ester in formulating oil based mud by Didier Degouy and J-F, Argillier , Andre Demoulin and Firmin Velghe on their paper " Biodegradable Muds: An Attractive Answer to Environmental Legislation Around Offshore Drilling", mentioned that esters used in these new oil-based muds have an anaerobic biodegradability of more than 60% after 2 months and aerobic biodegradability of 85% after 4 weeks.

The original formulation developed, maintained very good properties up to 140°C. The performance of the mud was based on proper choice of ester quality, emulsifying system and additives type, and on a severe control of lime concentration to avoid any risks of ester hydrolysis in conjunction to pH and temperature. The behaviour of the mud was also studied under bottom hole conditions, by using a homemade HT/HP flow loop, during aging. Contamination test proved the ability of ester to resist contaminations such as sea water, drilled solids, and cements during drilling process.

For the emulsifiers selection process, several commercially available emulsifiers have been tested to enhance the invert emulsion stability. The stability has been studied as a function of different parameters:

[1] Emulsifier concentration

[2] Volume fraction of water in the invert emulsion

[3] Presence of electrolytes

[4] Temperature

These tests results solely influenced the selection process of esters and emulsifying systems that form the invert fluid. Due to the linearity of the chemical chains, the ester extracted from natural oils and fats are perfectly biodegradable, following the European normalised method 84/449/EEC-L 251, these esters have an aerobic biodegradability higher than 85% after 4 weeks and already higher than 60% after 10 days. Moreover, natural oil's esters are environmentally friendly for the benthic fauna. The toxicity of ester used in this study is very low and the LC 50 following Crangon-Crangon test is above 6000mg/l. On the other hand the oral toxicity of the products are tested on lab rats and the LD 50 value is regularly above 2000 mg/kg of rat.

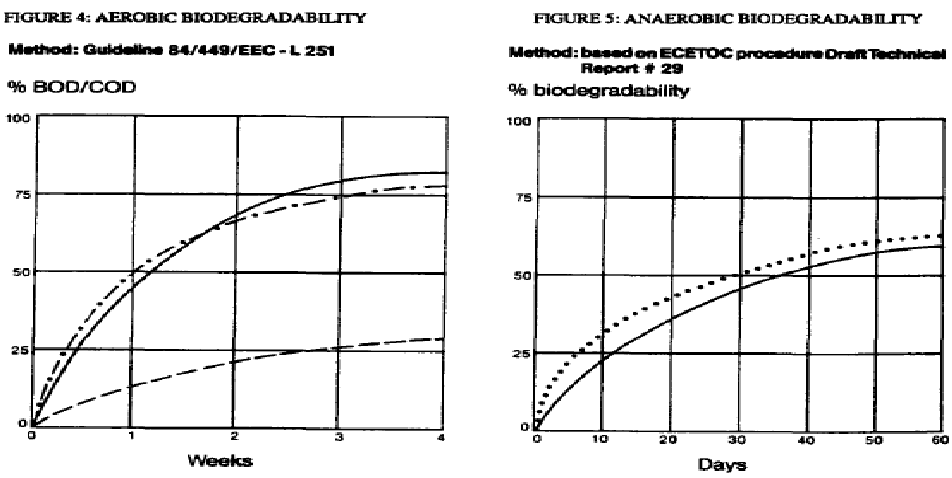


Figure 2.3: The left side shows the aerobic biodegradability and right side shows the anaerobic biodegradability of the tested ester

[2.4] Theory

Basically, drilling fluids can be grouped into three main branches which are Oil based mud and synthetic based mud and Water based mud. The most commonly used type of mud are oil based mud and synthetic based mud due to their functionality exceeding that of WBM. Oil based mud and synthetic based mud require special emulsifier agents to ensure that the oil phase and water phase mix and form a stable emulsion in a oil based fluid.

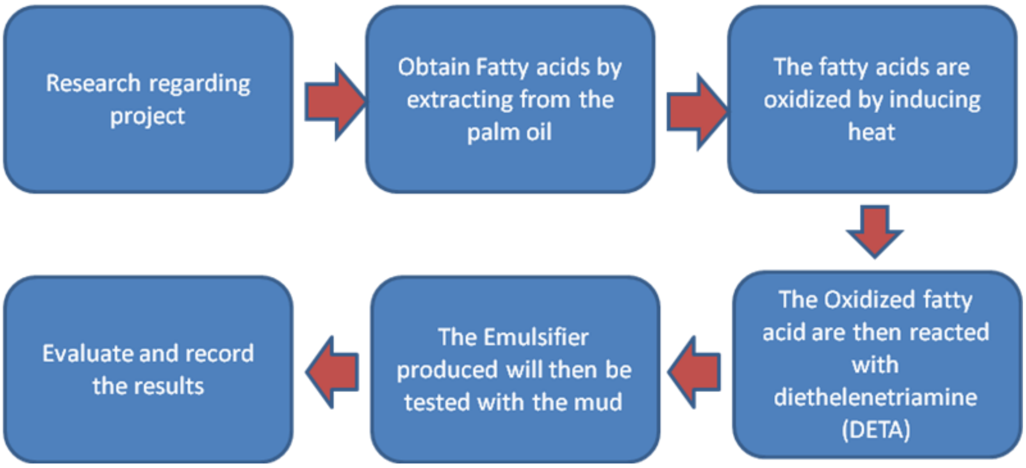
Emulsions can be categorised as primary emulsion and secondary emulsion. Secondary emulsion alone could not help Oil based fluids to achieve stable emulsion. WBM are regarded as not harmful to the environment and therefore generally used in offshore drilling. However, OBM has provided a number advantages compared to WBM such as superior borehole stability, thinner filter cake, excellent lubricate and less risk of stuck pipe. The major disadvantage of OBMs is that the base fluid consist of high level of toxicity poses an environmental hazard if it is released into the ocean either through spill or cuttings removal.

These days, synthetic-based muds are designed to combine the advantageous operating qualities of OBMs with the lower toxicity and environmental impact qualities of WBMs. SBMs have drilling and operational properties similar to those of OBM systems and are used where OBMs are commonly used, such as in difficult drilling situations where the properties of WBMs would limit performance.

All types of mud use emulsifying agents to help them achieve stability in emulsion between their phases.

CHAPTER 3: METHODOLOGY

[3.1] **Research Methodology**



The assessment of this research is to test if emulsifying agents synthesised from palm oil fatty acids can compete with the commercial emulsifying agents and achieve stability of the water-in-oil emulsion of oil based mud. Besides functionality, we also hope to achieve mud properties which have less toxification agents and overall environmentally friendly drilling fluids.

1. The first step in any project is to find and analyze results and findings from previous researchers regarding the topic. My topic is based on producing emulsifying agents from palm oil fatty acids for drilling fluids. Therefore the research papers that i refer to must have information to help me understand in depth of the characteristics of an emulsifier.
2. The initial step in this research is to obtain the fatty acids from the palm oil as the fatty acids act as the main component that we are to test in this research
3. Then the fatty acids will undergo some modifications so that it could act as an emulsifier for the drilling fluid
4. Next, is to oxidise the fatty acids using a special equipment
5. After the oxidising process, these fatty acids will be synthesized by reacting with DETA. Catalyst is only added to the emulsifier if required
6. The final step will be by testing the emulsifier produced with several types of based mud and comparison will be made to conclude the best result. Further studies is solely based on the results obtain from the experiment.

[3.2] Project Activities

[3.2.1] Emulsifier Formation Test

Objective: To produce a new emulsifier from palm oil based fatty acids for drilling fluids

Theory: Emulsifier is a chemical substance that stabilizes the emulsion property of two immiscible
phase (oil phase and water phase) by increasing its kinetic stability

Procedure:

1. *Place some extracted amount of fatty acid into the reactor*
2. *oxidize the fatty acid by inducing heat*
3. *Alter the temperature of the reactor to obtain various results*
4. *React the oxidation of fatty acid with diethylenetriamine (DETA)*
5. *Catalyst can be added to improve formation*

[3.2.2] Mud Density Test

Objective: To determine the density of the drilling fluid

Theory: The density of the drilling fluid must be controlled to provide adequate hydrostatic head to prevent influx formation

Procedure:

1. The Instrument base must be set at flat level surface
2. Next measure and record the mud temperature
3. Then, fill the mud cup with the mud that is to be tested and gently tap the cup to allow any trapped gas to break out
4. Replace the cap and rotate until its firmly seated, ensuring some of the mud is expelled though the hole on the top
5. Holding the cap firmly, wipe the outside of the cup until its clean
6. Finally place the beam on the base support and balance it by using the rider along the graduated scale. Balance is achieved when the bubble is directly below the centre line

[3.2.3] Funnel Viscosity Test

Objective: To measure the plastic viscosity, gel strength and yield point of the drilling fluids

Theory: The viscosity is defined as the resistance of fluid to flow, and it is important because in the drilling operation it influenced several factor such as mud density, pumping rate and pressure system.

Procedure:

1. cover the orifice with a finger and pour a freshly agitated fluid sample through the screen into the clean, dry and upright funnel until the liquid level reaches the bottom of the screen
2. next quickly remove the finger and measure the time required for the fluid to fill the vessel to the one quart
3. Finally report the result to the nearest second as Marsh Funnel viscosity and the temperature to the nearest degree.

[3.2.4] Rheology Test

Objective: To determine single or multi-point viscosity

Theory: The parameters used to measure viscosity is the shearing stress to the rate of shearing strain. There are two types of fluid characterized which are Newtonian and non-Newtonian fluid.

Procedure:

1. First we need to stir the sample at 600 rpm while the sample is heating or cooling to 120 degrees F.
2. Next ensure that the Dial Reading has been stabilized at this speed before noting the result and proceeding to 300, 200, 100, 6 and 3 RPM.
3. Having taken the 3 RPM reading stir the sample at 600 RPM for 30 seconds before taking the 10 second gel at 3RPM
4. Re-stir the sample at 600 RPM for 30 seconds and leave it undisturbed for 10 minutes, ensuring the temperature stays at 120 degrees F. Take the 10 minute gel reading at 3 RPM.

[3.2.5] Retort Analysis Test

Objective: To determine the quantity of solid and liquid of mud from evaporation of weighted sample.

Theory: Knowledge of the liquid and solid content of the mud is essential because to ensure good control properties of the mud. By this test we can identify poor performing mud and proper treatment can be used to solve the problem.

Procedure:

1. Retort assembly should be dry and clean. Remove all traces from previously used with a steel wool. Failure to change the steel wool can result in inaccurate measurements.
2. weigh and clean the dry retort cup and lid on the triple beam balance
3. Add the mud which has been allowed to cool to ambient temperature to retort the cup gently tap the cup to remove air bubbles and place the lid with a rotational movement to obtain a proper fit. Be sure an excess fluid flows out of the hole in the lid.
4. Carefully clean the cup and lid of excess fluid and reweigh on the triple beam balance . The retort mud weight SG is determined as the difference between the empty and full wweights in grams divided by 50 (the volume of mud)
5. Pack the retort body with a new steel wool, apply Never-Seez , to the threads and assemble the top and bottom parts. Ensure that the two parts are fully screwed together. If it is not possible to fully screw together the two parts, it will be necessary to clean the threads and repeat the steps above.
6. Attach the condenser and place The retort assembly in the heating jacket and close the insulating lid
7. Place the clean, dry liquid condenser on the outlet and turn on the heating jacket
8. The temperature should be adjusted so that the retort cell glows dull red at the end of the distillation and the final drops coming out of the retort should be observed to be black and sink through the clear oil to the meniscus. Ultimately smoke will emerge from the retort and distillation is only complete when the smoke stops.

[3.2.6] HTHP Filtration Test

Objective: To testing drilling fluids at elevated pressure and temperature

Theory: Filtration control is essential to prevent differential sticking in the formation which minimizes formation damage

Procedure:

1. To standardise this test backpressure is applied during the test to avoid filtration evaporation
2. Allow the heating jacket to reach the required temperature
3. Check all “O” rings on the HPHT bomb and lid and change out any damaged ring. The rings that should be checked are the four small stem “O” rings , one in the lid and one in the cell. The large “O” rings should be rounded profile and free from dirt.

4. Tighten the bottom valve stem, taking care not to over tighten, and fill the cell to about 0.5 inch from the rim
5. Place a filter paper on the rim and put the lid on the cell. Ensure the lid stem is open while doing this to avoid damaging the filter paper
6. Tighten the six studs in the bomb and close the lid stem
7. Place the bomb in the heating jacket with the lid facing downwards. Rotate the bomb until it seats on the locking pin.
8. Place a CO2 cartridge in each regulator and tighten up the retainers
9. Place the top regulator on the stem and engage the locking pin. Close the bleed off valve and turn the regulator clockwise until 100 psi is shown on the gauge.
10. Repeat the process with the bottom regulator
11. Turn the top valve stem ¼ to ½ turn anticlockwise to pressure up the cell to 100 psi
12. when the cell reaches the required test temperature open the bottom stem (½ Turn) and increase the pressure on the top regulator to 600 psi over +/- 20 seconds
13. Commence the test. The test should be carried out as soon as the bomb reaches the test temperature. Leaving the cell for long periods at high temperature will provide undesirable results
14. If the pressure on the bottom regulator increases significantly above 100 psi bleed off som of the filtrate into the graduated cylinder
15. After 30 minutes , close the top and bottom valve stems. Slack off the regulator on the bottom collection vessel. Bleed off the filtrate into the graduated cylinder. Disconnect bottom collection vessel, fully open the bleed off valve and tip any residual filtrate into the graduated cylinder.
16. Bleed the pressure off the top regulator
17. Disconnect the top regulator and remove the bomb from the heating jacket
18. When the bomb has cooled bleed off the trapped pressure by slowly opening the top valve with the bomb in an upright position. With the residual pressure bled off invert the cell, loosen the six studs and remove the lid
19. Examine the filtrate paper and report the thickness of 32^{nd} of an inch. Comments about the quality of the cake should be noted in the comments section of the mud report
20. Thoroughly clean the bomb and stems in preparation of the next test
21. Do not Preheat the bomb by resting on the heating jacket

[3.2.7] PH Test

Objective: To measure the potential difference and directly by dial reading the PH of the sample

Theory: PH excess acidity and alkalinity of an aqueous solution. It is used for improve performance of mud additives and minimize pipe corrosion.

Procedure:

1. The PH meter must be calibrated, as per the manufacturer's instructions, on a regular basis. Buffer solutions used must be within their use by date. Do not re-cycle the buffer solutions used to calibrate the meter. Throw them away each time and use fresh buffer solutions when the meters are calibrated
2. For accurate PH measurements the test fluid, buffer solutions and reference electrode must all be at the same temperature ambient temperature
3. Insert the electrode into the fluid contained in a small glass flask and swirl gently
4. Measure the fluid PH according to the directions supplied with the instrument. When the meter reading becomes constant, record the PH nearest 0.1 of a unit.

[3.2.8] Emulsion test

Objective: To indicate the stability and the type of emulsion present in the mud

Procedure:

1. Test the meter in air, the reading should go off scale and show the display start flashing. If the meter does not go off scale, it is an indication that the probe is shorting out due to an accumulation of detrtius between the two prongs. It is clear that the probe can short out before the end point of the mud is reached and erroneous reading will result. The probe should be carefully cleaned and retested in air to ensure that it now goes off scale before testing
2. Place the clean checked probe in the sample at 120 degrees F and use it to stir the fluid to homogeneity. Position the probe so it does not touch the bottom or the sides of the heated cup, ensuring the tip of the electrode is completely immersed
3. Press the button to initiate the voltage ramp, holding the probe still until the end point is reached and a steady reading is seen in the Digital Display. Note the reading
4. Repeat the test. the two ES values should be within 5 % and anything greater would indicate a problem
5. The result is the average of the two reading.

[3.3] Gantt Chart

NO.	Project activities	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Preparation of sample														
2	Lab sessions and data acquirement														
3	Analysis on the mud performance														
4	Progress Report submission														
5	Studies About Drilling Fluids and Additives														
6	Poster Preparation														
7	Pre-SEDEX & draft final report submission														
8	Technical paper submission														
9	Submission of soft bound														
10	Submission of Hard bound copy														
11	Final Presentation to external examiner														

CHAPTER 4: RESULTS & DISCUSSION

This experiment main purpose is to synthesize and prepare a new sample of emulsifier. The sample will be prepared once all the materials required have arrived. The sample will be prepared by reacting the fatty acids with the Diethylene Triamine (DETA) in order to produce a new type of emulsifier. For this experiment also we try to obtain a longer chain fatty acid (C18-C22) but it seems that the only longest available carbon chain at this moment is C18. The fatty acids will act as a raw material and DETA will be used as an additive agent for the emulsifier.

The procedure is to add 500ml of fatty acids with 50 ml of DETA into the reactor container. An approval has already been obtained to use the reactor at UPM. Next the container will be heated according to the heating temperature required for complete reaction and the liquid is stirred constantly to make the reaction process consistent. Later air is permitted into the reactor and the temperature is made constant and the liquid will be always stirred. The process is continued for 30 minutes.

The obtained emulsifier will then be tested into mud and the objective is to evaluate the performance of the drilling fluid using this emulsifier. The performance of this drilling mud will be compared to the commercially used emulsifier products of SCOMI.

The main test is by comparing it with CONFIMUL-P and CONFIMUL-S which is a product of which SCOMI use to formulate drilling fluids. The emulsifiers have been tested in high temperature and high pressure conditions. The following shows the chemical that will be used for this experiment:

Material Used	Function
SARALINE 18SV, SARAPAR 147 ESCAID	Base Oil
CONFI-GEL HT	High temperature viscofer
CONFIMUL P	Primary Emulsion
CONFIMUL S	Secondary Emulsion
CONFI-TROL XHT	Very high temperature fluid loss control additive
Lime	Alkalinity buffer
Barite	Weighting agent

PART 1- EMULSION FORMATION TEST

The first experiment involves the emulsion formation test which is the process of synthesizing the new type of emulsifier required. The emulsion formation test requires the following equipments and materials:

1. **DETA**
2. **C 18-98 Stearic Acid**
3. **2L Bio-Reactor**

The experiments that follow are in order to determine how to obtain the most optimum type of emulsifier. First the **500 g** of Stearic Acid is measured and added into the reactor and the stearic acid is heated until it completely melts into liquid. Once all the stearic acid has melted the second experiment is done by first measuring **50 ml** of **DETA** and adding it to the liquid stearic acid and the reaction is recorded. The time is varied in each experiment to see how long does it take for complete reaction. The mixture is observed constantly.

Experiment 1: to test the time required for the stearic acid to completely melt under different temperatures.

Experiment	Manipulative: temperature where the 500g stearic acid is heated (degrees Celcius)	Responsive: time required for the 500g Stearic acid to melt completely (mins)	Observation
1	64	Not feasible	The stearic acid does not melt
2	80	240 mins	The stearic acid melts but in a slow order
3	100	120 mins	The stearic acid turns completely into liquid

Note that for the 64 degrees celcius the stearic acid does not melt though the melting point for the stearic acid as stated in its MSDS is **64 degrees celcius**. This may due to the amount of fatty acid used and the method of heating practiced as the reactor uses the heat from boiling water to heat up the stearic acid.

Experiment 2: Testing the effect of rotation spin on the melting time required for stearic acid which is maintained at 100 degrees celcius to react with DETA:

Experiment	Manipulative: the rotation speed (RPM)	Time required for complete reaction (mins)	Observation
1	100	60	A yellowish solution forms after 1 hr
2	200	30	A yellowish solution forms after 30 mins

In both experiments the yellowish solution forms though the time required differs according to respective speed used. The yellowish solution solidifies after the solution is cooled down to room temperature

Figure 4.1: The used 2L Bio-Reactor from UPM which uses water as its heating agent

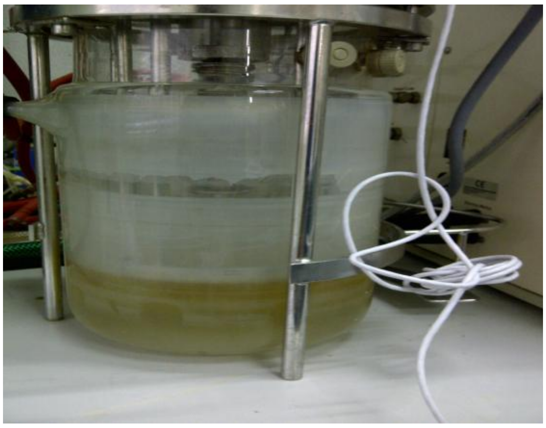


Figure 4.2: The Stearic Acid is heated with the reactor at 100 degrees celcius and forms a viscous solution

Figure 4.3: The melted stearic acid is added with DETA and the time required for the reaction is recorded using a stopwatch.

Figure 4.4: The mixture of Stearic acid and DETA solidifies as it cools down to room temperature (23-24 degrees Celcius)

Through the experiments conducted the new type of palm oil based emulsifier has been produced though the produced emulsifier did not follow entirely the specifications as suggested earlier in this project. Initially the emulsifier produced is determined to have the same type of properties as the commercially used emulsifier especially in terms of its parameters as such as the carbon chain used. but due to some restrictions and unavailability to obtain a bigger carbon chain (C22-C38) some modifications had to be made upon the experiments. We had to settle for the C18-98 Stearic acid (a type of fatty acid), smaller chain fatty acid to be used for the experiments.

The emulsification test process also was modified due to some constrictions along the way. the unavailability of the chemical reactor in UTP caused us to borrow the reactor in UPM and the only available reactor there was a 2L Bio-Reactor. This bio reactor could only support a heat of 100 degrees Celcius due to the reactor using water as its heating medium. Thus even though a complete reaction could not be achieved the experiment was continued under 100 degrees Celcius.

There are also many uncertainties involved with this experiment. For example the final result of the formation test where the new emulsifier solidified under room temperature was unexpected as earlier it was assumed that the emulsifier will be in liquid form and the result did not vary with all the experiments that were conducted. Though, the result was accepted and will be tested out with the commercial emulsifier.

PART 2-TESTING THE NEW EMULSIFYER ON THE PROPERTIES OF DRILLING MUD

the next part for the experiment is to test the new type of emulsifier with drilling mud under various condition to see if the drilling mud is still able to retain its properties and if possible the functionality of the drilling mud is able to overcome the base sample. The mud will be tested for under high temperature and high pressure condition (300 degrees F and 500 psi), plastic viscosity, emulsion stability , gel strength and yield point . The mud density that tested will be 11.5lb/gallon and the emulsifier will be added according to the formulation listed below.

Products	Sample 1	Sample 2	Sample 3	Base Sample
SARALINE 18SV, SARAPAR 147 ESCAID(g)	154.84	155.01	154.69	155.21
CONFIMUL P(g)	0.00	3.00	0.00	3.00
CONFIMUL S(g)	9.00	0.00	0.00	9.00
Palm oil Based fatty acid emulsifier(g)	3.00	9.00	12.00	0.00
CONFI-GEL XHT(g)	8.50	8.50	8.50	8.50
CONFI-TROL XHT(g)	8.00	8.00	8.00	8.00
Lime(g)	8.00	8.00	8.00	8.00
Drill water(g)	67.32	66.78	66.98	66.62
CaCl(g)	26.88	27.04	27.34	26.31
DRILL-BAR barite(g)	199.23	196.20	198.43	195.32

A sample of using CONFIMUL-P and CONFIMUL-S only has been prepared as a base sample that will be referred to the mud tested with the new type of emulsifier. Below shows the specification that are required to be achieved in this experiment:

Fluid Properties	**Specification**
Mud Density, SG	**11.5 ppg**
6pm dial reading	**8-16**
Yield point	**15-25**

10 sec gel strength	**8-18**
10 min gel strength	**15-30**
HTHP @ 300 degrees F, 500 psi	**4<**
Electrical stability	**>400**

The experiment is then continued and another 3 samples are prepared with different proportions of palm oil based emulsifier in order to determine which proportion will give out the optimum properties for the drilling mud. Oil and synthetic based additives such as CONFI-GEL XHT and CONFI-TROL XHT are also added to the mud in order to retain the emulsifier's properties.

After hot rolling, 50 C for 16 hr	**Sample 1**	**Sample 2**	**Sample 3**	**Base Sample**
Mud density , lb/gal	11.5	11.5	11.5	**11.5**
Rheological Properties	**120 ^{o}F**	**120 ^{o}F**	**120 ^{o}F**	**120 ^{o}F**
600 RPM	97	363	343	**81**
300 RPM	68	321	315	**60**
200 RPM	53	280	265	**45**
100 RPM	36	278	261	**28**
6 RPM	25	155	143	**19**
3 RPM	18	119	106	**15**
Plastic Viscosity, cP	29	42	28	**21**
Yield Point, lb/100 ft sq	39	279	287	**39**
Gel 10 Sec, lb/ft sq	21	108	114	**16**
Gel 10 Min, lb/ ft sq	30	121	127	**19**
HTHP,cc/30min at 30 degrees F	3.2	3.8	4.3	**4**
ES, volts at 120 ^{o}F	634	758	789	**936**

The above shows the results obtained after hot rolling each sample. The result obtained for each parameter is an average value obtained after 3 trials of each parameter. The gel strength test had 3 trials to it so that the most accurate value of gel strength for each sample can be obtained. The most important test is the emulsion stability of the mud. The importance of this test is to determine if the new type of emulsifier is able to stabilize the emulsion of the drilling mud and the result obtained is as shown below:

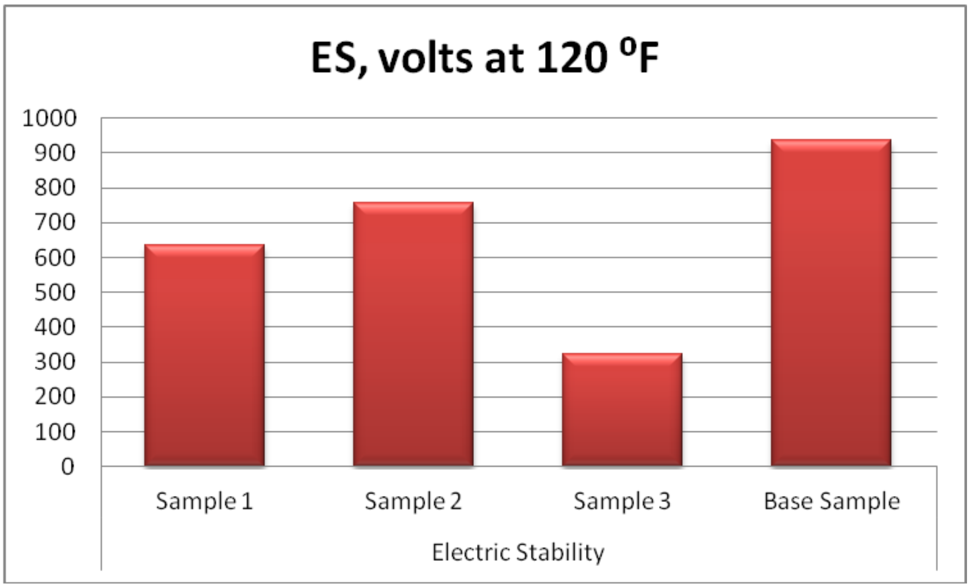


Figure 4.5: Shows the Emulsion Stability recorded for each Sample

The result shows that the base sample has the highest ability to retain the emulsion in the drilling mud with more than 900 volts. Sample 3 shows the lowest recorded emulsion stability which is the sample that has only the new type of emulsifier. The overall results show that Sample 1 and Sample 2 passed the requirement for emulsion stability test. Then the rheology and fluid loss filtration test was conducted on the mud samples.

Next the mud was tested to obtain its plastic viscosity (PV) and Yield point (YP). The plastic viscosity (PV) and Yield Point (YP) for the base sample and other samples will be calculated using the following formulas:

Plastic Viscosity, PV= Reading (600rpm) – Reading (300rpm)

Yield Point, YP = Reading (300rpm) – Plastic Viscosity

The result obtained for PV and YP, it shows that the new type of emulsifier are too high and is not suitable to be used as a replacement for commercial emulsifier except for Sample 1. Though the amount of the new type of emulsifier used was not significantly high in Sample 1 and thus the result obtained may only signify the usage of the commercial emulsifier. Therefore some modification was made on the mud formulation in order to obtain better result by using the new emulsifier.

Products	Sample 4
SARALINE 18SV, SARAPAR 147 ESCAID(g)	155.65
CONFIMUL P(g)	6.00
CONFIMUL S(g)	3.00
Palm oil Based fatty acid emulsifier(g)	3.00
CONFI-GEL XHT(g)	8.50
CONFI-TROL XHT(g)	8.00
Lime(g)	8.00
Drill water(g)	68.45
CaCl(g)	27.68
DRILL-BAR barite(g)	198.34

Figure 4.6: The suggested changes that have been made on the mud formulation

The parameters that were evaluated from the emulsion produced from the palm oil based fatty acids are the:

1. ***Emulsion Stability***
2. ***Plastic Viscosity***
3. ***Yield Point***
4. ***Gel Strength (10 sec and 10 min)***
5. ***The ability of the emulsion to withstand under High Pressure and High Temperature condition***

Condition 1: Emulsion Stability

The importance of investigating the ES is to evaluate the stability of emulsion. The higher the reading, the better the emulsion stability. The different types of samples tested are required to have this property/ have high emulsion stability so that it could be used in the field.

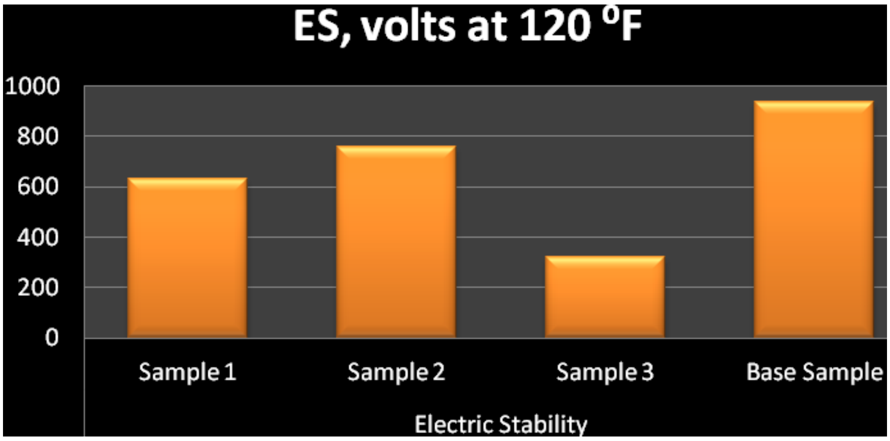


Figure 4.7:Emulsion stability for the samples

The result from the emulsion stability test shows that sample 3 failed to meet the requirement as it has the lowest emulsion stability and is unable to retain the emulsion in the drilling mud. The Sample 3 is made up of 100% new type of emulsifier. Though Sample 1 and Sample 2 shows promises as it met the requirement for emulsion stability.

Condition 2: Plastic Viscosity

PV is the slope of the shear stress/shear rate line above the yield point. PV represents the viscosity of a mud when extrapolated to infinite shear rate on the basis of the mathematics of the Bingham model. (Yield point, YP, is the other parameter of that model.) 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 of the mud. Plastic viscosity is important in order to determine if the mud can act as a good drilling fluid. High PV is normally due to viscous base mud.

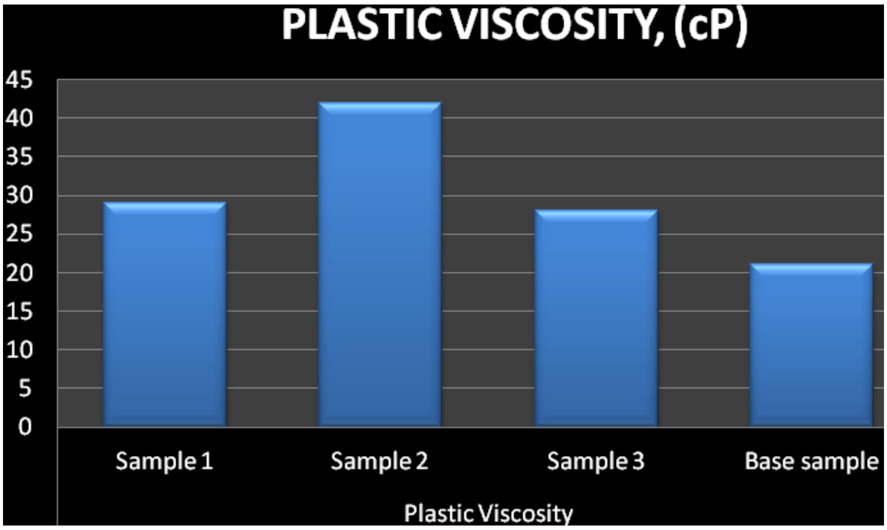


Figure 4.8: Plastic viscosity recorded for the samples

The base sample recorded the lowest PV value among all the tested sample. Except for Sample 1, all the tested samples failed the PV test and thus some modification was made on the mud formulation and this modified mud was tested for PV and the result shows improvement and is compared to the base sample.

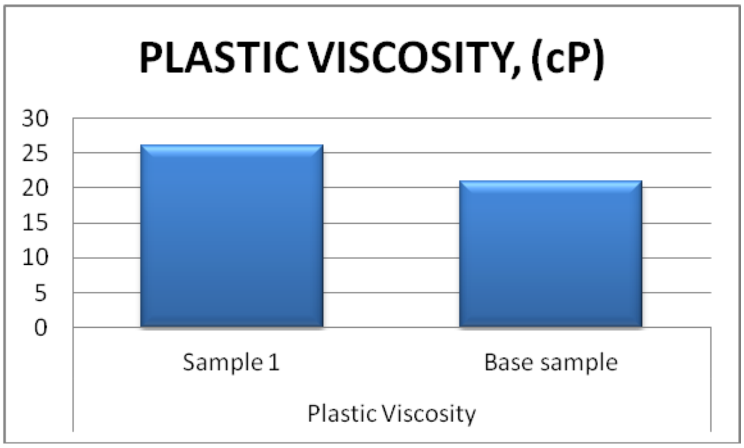


Figure 4.9: Plastic viscosity recorded for the modified mud sample when compared to the Base sample

Condition 3: Yield Point

YP is the yield stress extrapolated to a shear rate of zero. YP is used to evaluate the ability of amud to lift cuttings out of the annulus. A high YP implies a non-Newtonian fluid, one that carries cuttings better than a fluid of similar density but lower YP. YP is lowered by adding deflocculant to a clay-based mud and increased by adding freshly dispersed clay or a flocculant, such as lime.

The yield point recorded for the samples tested is as shown below:

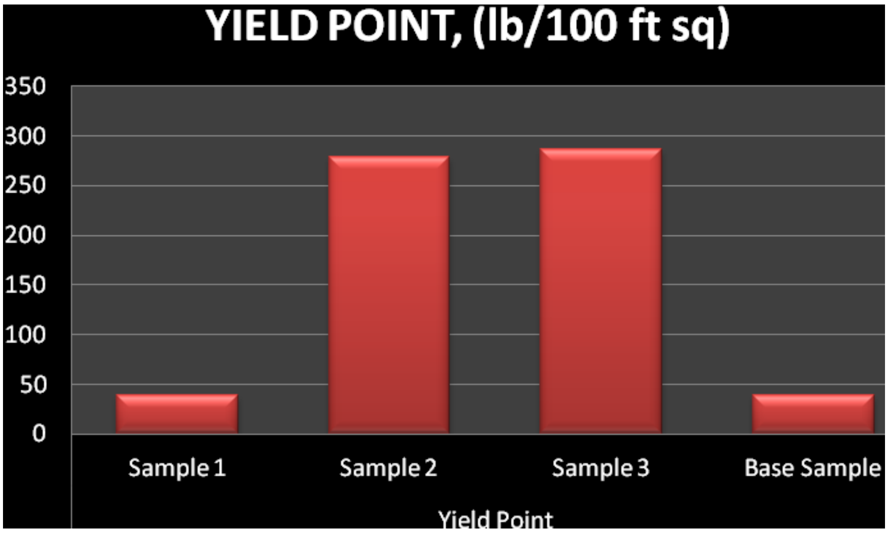


Figure 4.10: Shows the yield point recorded for the 4 samples tested

The yield point recorded for the modified sample is as shown below:

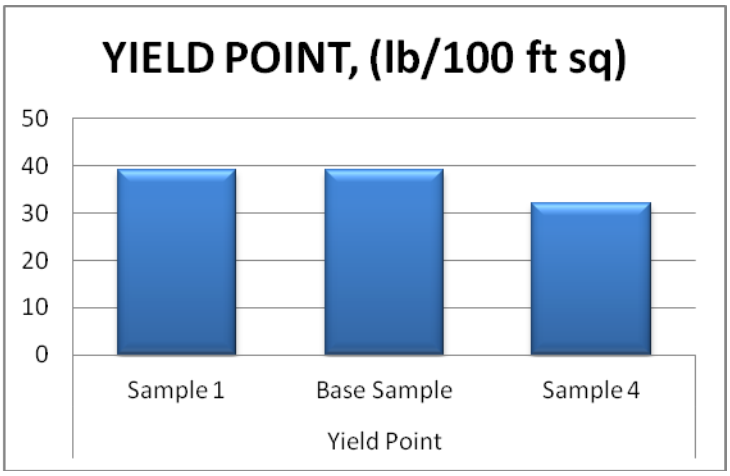


Figure 4.11: Shows The yield point for the modified mud sample compared to Base sample and sample 1

Condition 4: Gel Strength (10s and 10 min)

The shear stress measured at low shear rate after a mud has set quiescently for a period of time (10 seconds and 10 minutes in the standard API procedure, although measurements after 30 minutes or 16 hours may also be made). The base sample exhibits good gel strength.

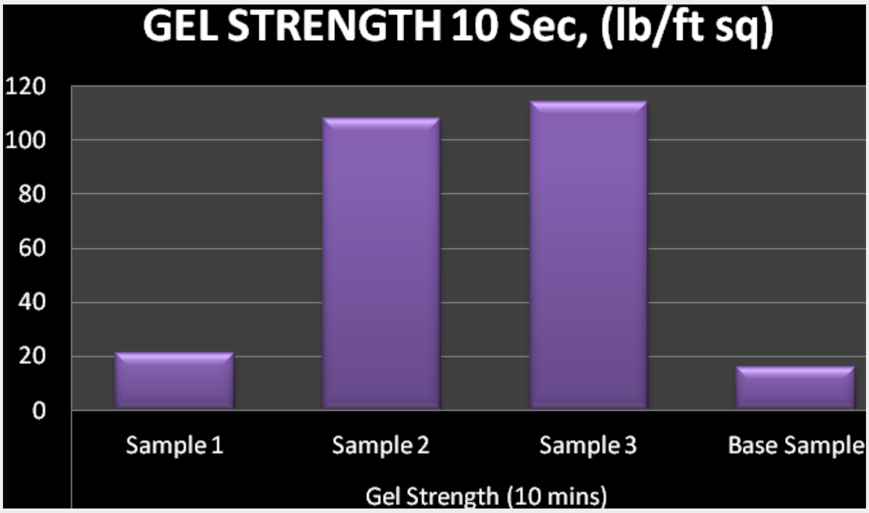


Figure 4.12: 10 sec gel strength for all samples

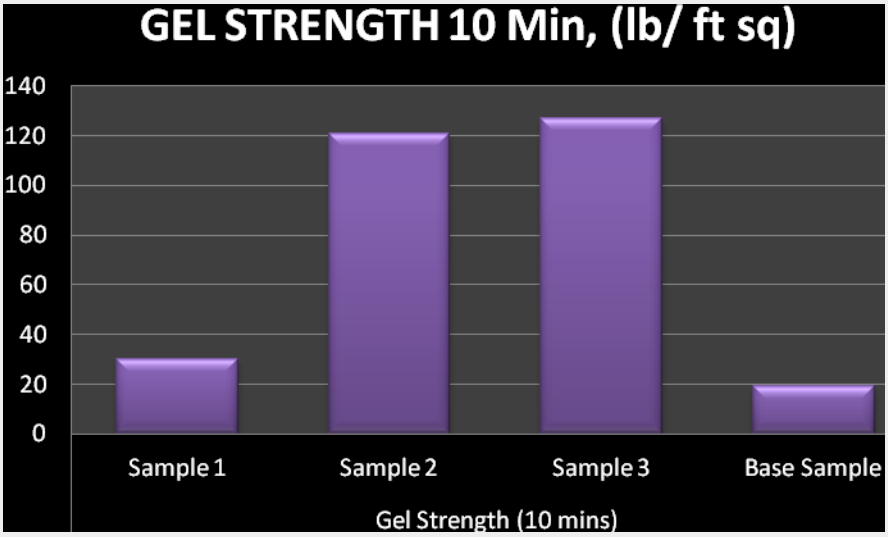


Figure 4.13: 10 min gel Strength for all samples tested

Condition 5: HTHP Test

In order to evaluate the amount of fluid loss from the mud due to high temperature and high pressure conditions. Base sample exhibited low fluid loss under these conditions and less quantity of fluid gained the better the result.

Basically 4 more extra samples excluding the base sample will be prepared and the manipulative variable for this experiment will be the quantity of CONFIMUL P, CONFIMUL S, and fatty acids based emulsion used. The result obtained will then be compared to the values of the base sample for the parameters mentioned above.

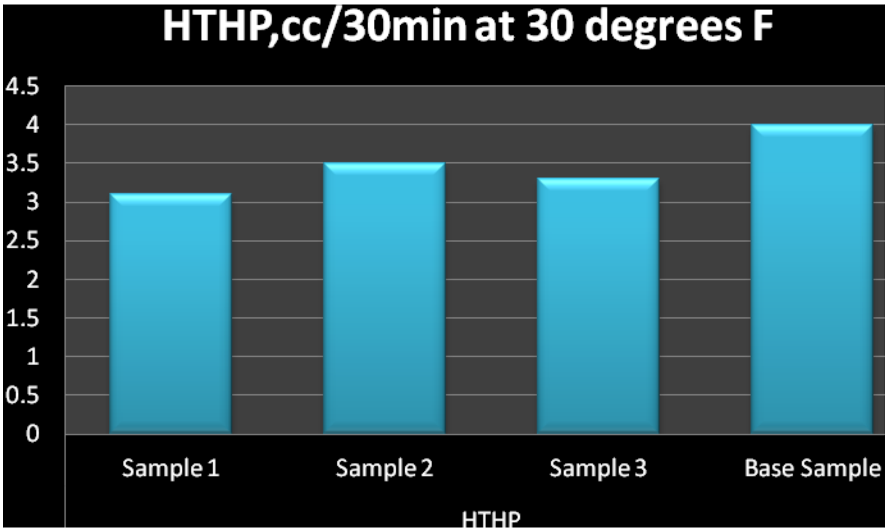


Figure 4.14: HTHP recorded for each Sample

Based on all the result obatained above Sample 1 shows high promise that it could act as a replacement for the commercial used emulsifier. Sample 4 which had some modification made to its mud formulation also shows promise though it is important to always consider uncertainties in this type of experiment and we need to conduct further research so that we could improve the functionality of this new type of emulsifier.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

[4.1] Conclusion

The aim of this research is to identify the effectiveness of emulsifying agent produced from palm oil based fatty acids to be used in drilling fluids to achieve a stable emulsion. Emulsifying agents reduce interfacial tension between two immiscible phases example oil phase and water phase and help these two(or more) phases to achieve a stable emulsion that does not losses its physical properties when tested in high temperature and high pressure. Therefore it is essential that these palm oil based fatty acid emulsifying agent is able to perform this functionality.

Based on the result obtained on the emulsifier produced from palm oil, it shows that the new emulsifier is suitable to function as an emulsifier for drilling fluid if some modification is made on the formulation. Though the results obtained is still not as good as the commercial emulsifier but it shows promise that it might act as a suitable replacement for the commercial emulsifier in the future. Hence, i believe if further studies were to be conducted we will be able to obtain better result on the performance of the emulsifier with lower cost and has less impact on the environment.

Palm oil is also an essential and abundant resource in Malaysia and has many functionality and this industry should be exploited further because it holds many potential. Not only that, but palm oil based emulsifiers are also non-toxic and environmentally friendly agents and are biodegradable in anaerobic environment. Thus this overall reduces the impact caused by drilling fluids when there is a loss circulation or when removing drilling cuttings from the well.

Therefore, I believe that palm oil based fatty acid emulsifying agents could be a significant replacement to all commercial type additive agents which are usually produced from diesel and ground nut oil. This is because diesel type products are less environmentally friendly compared to palm oil based products. We will be also able to promote in the economic production of Malaysia as it has an abundant resource of palm oil available to be exploited.

[4.2] Recommendation

The usage of palm oil based fatty acids as a drilling fluid emulsifier is still a new technology and is not yet as popular as the usage of commercialised emulsifying agents. Therefore more promotions and information should be provided by organizations such as Society of Petroleum Engineers (SPE) regarding the benefits of using palm oil based fatty acids as an emulsifier. Other than that, there must also be initialization from the government by providing funds for research on this type of emulsifier as it does not only enable the achievement of great heights in the oil and gas industry but at the same time will promote the economy of the country by usage of our abundant resource of palm.

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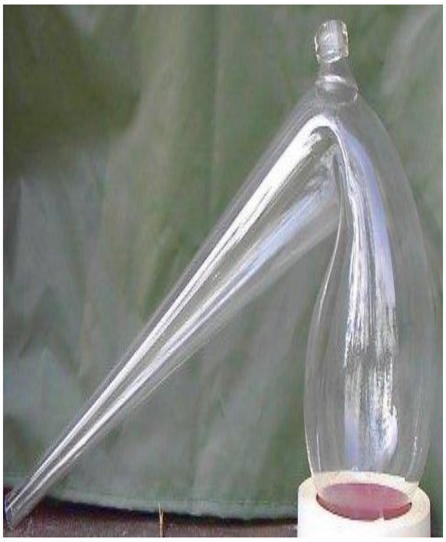
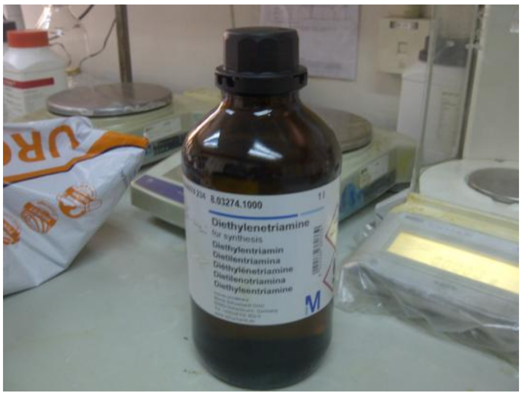
APPENDIX

Tools & Materials to be Use:

Below shows the materials that have been used for this experiment and also the equipments in general used:

	**Figure 1: Viscometer**
	**Figure 2: HTHP Filter Press**

<table>
<tr><td></td><td>Figure 3: Retort</td></tr>
<tr><td></td><td>Figure 4: Pressurized Mud Balance</td></tr>
<tr><td>
</td><td>Figure 5: Diethylene Triamine

C14H13N3
1 L=0.95 Kg
M: 103.16 g/mol

Specification
Assay(CG area %) >98%
Density(d20 °C/4°C)</td></tr>
</table>

Figure 6: Flaky C18 98 Stearic Acid

Physical & chemical properties:

Designation	Value	Testing for
Physical State	Solid	
Odour	Fatty	
Colour	White	
Boiling point	Approx. 214˚C	
Solidification range	Approx. 67°C	
Flash point	Approx.205°C	Din ISO 2592
Density	Approx.0.85g/ml	
Solubility	insoluble	

Figure 7: Modified 2L Bio-Reactor