DISSERTATION

RESEARCH ON EVALUATING MICRO SIZE PARTICLE MATERIAL OF JATROPHA AS FLUID LOSS REDUCER IN SYNTHETIC HTHP DRILLING FLUIDS

by

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Dissertation submitted in partial fulfillment of
the requirements for the
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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS
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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.

MUHAMMAD ZUL'AIZAT MOHD ZULKIFLI

ABSTRACT

This paper would be focusing on the potential of the plant seed called jatropha which is abundance in Malaysia to be used as fluid loss reducer agent. The Jatropha material would be mixed in drilling mud and then will be tested under high pressure and high temperature (HPHT) condition. The better concentration of Jatropha material for better drilling fluid stability would be found and then investigation on the rheology of the drilling fluid which includes the fluid loss would be done.

However, there is this new phenomenon that involves researching on potentials of Jatropha materials to be used in oil and gas industries. Jatropha is a plant that produces high oil content in its seed. Other than its use in biodiesel, it also has value in other area such as medicine and pharmaceutical. However, the potential use of the Jatropha in oil and gas industries only has reached the drilling fluid such that the oil from the seed is substituting the traditional diesel oil based mud. On the other hand, by extracting the oil, the seed still has its remaining which is the solid part. Thus, it will be more beneficial if the remaining of the Jatropha seed can also be used as an additive in drilling mud such as fluid loss reducer.

The Jatropha seeds would be first undergo drying process in order to reduce its humidity as possible. This is done in order to make the ball milling process easier and the outcome would be as fine powder. If the seeds are wet, the outcome would be sticky and hard to be used for mud mixing. Once the sample is used as additive in oil based mud, the mud rheology would be checked. Then, the sample will be hotrolled for 16 hours in order to make it a matured mud. After that, it will be used for filter press test which would be HPHT and LPLT filter press test. The fluid loss produced will be observed and be compared to commercial fluid loss reducer which is in this case is CONFI-TROL.

The Jatropha shells that has been ball milled for 24 hours shows the best result among the Jatropha samples. In addition, it is also better in fluid reducing compared to CONFI-TROL which is 30% less fluid loss for HPHT and 50% less fluid loss for LPLT.

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Alhamdullilah, praise be upon Allah, with His will and permission, this project had been completed successfully. The author would like to express the deepest gratitude and appreciation to the following people for their support, patience and guidance. Without them, this project would not have been made possible.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The first oil well in human history in 1859 which is Drake well in Pennsylvania has created an overturn in energy industry players[1]. From inefficient, hazardous to health coal, investors turned their interest to oil and gas related industries. Since then, world has prospered from this energy saving, easy to store oil based products in various ways starting from transportation, plastic, dermatology products, health to even food. Nevertheless, for the past 20 years, growth in energy related technology has been remarkable and still there are urgencies to satisfy the world's need for fossil fuel in order to meet the requirement and demands nowadays. From then, people starts to go drilling down to deeper and harsher condition for meeting this unconditionally coming demand[1].

Of all the wells that are drilled in USA, less than 1% has penetrated below 15,000 feet. However from this 1% number of well, total production of oil from these well totals up to 7%.This shows those deep wells have high capabilities to produce high quality, abundance source of oil much more than average depth well. Even the National Petroleum Council designed a plan to increase the drill operation of deep well up to 12 percent by 2010 in order to meet the increasing US demand. However the bad side of this plan is that the construction cost can sum up to 12 times the average lower deep well (6,000 feet)[2].

Oil and gas players have done deep drilling for years, but there is no specific way to classify the type of the well whether it is a deep well or just average well. In an attempt to clarify the issue, Schlumberger uses a guideline that organizes HPHT well into three categories according to the **figure 1** below[1].

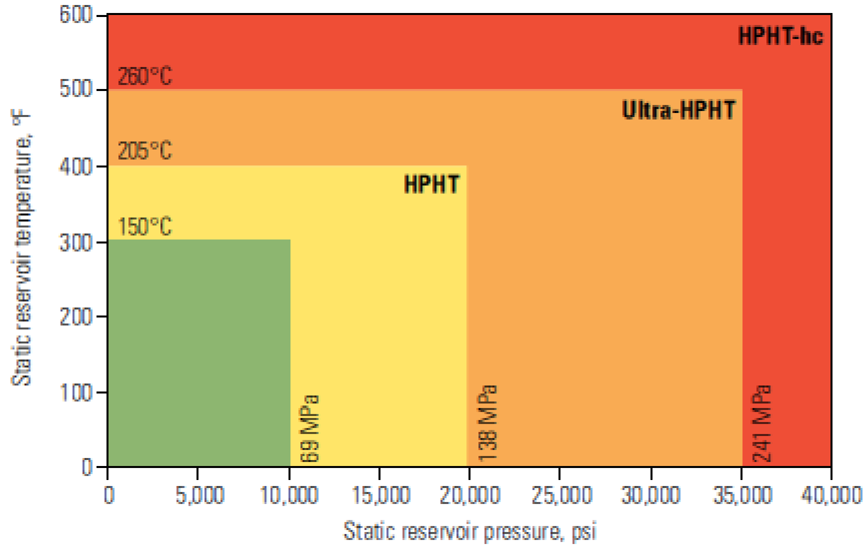


Figure 1: HPHT Well Classification

Figure 1 show that HPHT well where bottom-hole pressure is above 10 000 psi and temperature above 300^{0}F (~150^{0}C). Ultra HPHT well however is the next in line where it is in zone where the bottom hole pressure is above 20 000 psi and temperature above 350^{0}F(~205^{0}C). Finally Extreme HPHT (xHPHT) has bottom hole pressure above 35 000 psi and temperature is above 400^{0}F (~260^{0}C).

Oilfield Review last reviewed the HPHT domain in 1998. Since then the number of HPHT projects has grown. HPHT well has been drilled all around the world under various oil companies. Hot spots for these productions are mostly in deepwater Gulf of Mexico, North Sea, Norwegian Sea, Thailand and Indonesia. The distribution is shown on the figure below. The number of HPHT production has grown steadily since then. For example, a survey by Welling and Company on the direction of subsea system and services reported that 11% of well to be drilled in the next three to five years are expected to have BHTs exceeding 177^{0}C (350^{0}F). In addition, 26% of respondents expect BHPs between 69 and 103 MPa (10000 and 15000 psi) and 5% predict even higher pressure[1].

Figure 2: HPHT Projects All Around The World

In drilling a well, a particular, specific to the well drilling fluid is needed. Drilling fluid is a fluid which may contain any liquid, gas or gasified liquid circulating continuum substance that was used in the rotary drilling process to perform any or all of the various functions required in order to successfully drill a usable wellbore at the lowest overall well cost.

The selection of drill fluid must possess some of the required functionalities such as [3]:

- Ability to maintain the integrity of weak rock
- Ability to minimize fluid loss into permeable rock
- Ability to provide stable well control
- Ability to efficiently transfer hydraulic power
- Ability to move cuttings to the surface
- Provide steel/steel and steel/rock lubricity
- Provide protection against all forms of corrosion
- Allow formation evaluation
- Pose little or no hazard to rig personnel

- Have little or no adverse effect on the environment
- Have little or no adverse effect on elastomer

On the other hand, the drill fluid selection for HPHT well is more complicated and need to be really specific. HPHT well has bottom hole pressure and temperature above the normal zone well. In this harsh and more challenging zone, the drill fluid tends to lose its rheology and undergoes degradation and will damage the formation and increase the skin. Plus with the high temperature, the drill fluid will dehydrate and increase the rate of fluid loss.

1.2 Problem Statement

Conventional or regular drilling fluid used all around the world has limitation when it is been used in HPHT well. The usage of barite in conventional drilling fluid will tend to create high frictional pressure losses during circulation in long sections,(since HPHT well is much deeper and longer) leading to high ECDs (Equivalent Circulating Densities) in narrow drilling windows[3]. High bottomhole temperature would degrade the drill fluid quality in many ways such as its capability to carry the solids or drill cuts to the surface. This will lead to loss of well control especially in high-angle well where the solids cannot be taken out. Other than that, an inhibitor which contain sulphur which help in reducing the rate of corrosion in halide brines are proved to decompose to H2S at high temperatures and raise the risk of corrosion higher[3]. The drill fluid would also tend to dehydrate in high temperature and increase the chance of fluid impairment. With the problems combined, the drill fluid will tend to make the well under constant risk of hydrostatic pressure imbalance and lead to fluid loss. Thus, a detail, specially made drilling fluid must be made according to the well specification and in this case is a HPHT well. In this paper, the discussion would be on the study on fluid loss reduction in HPHT well with various based mud under different concentration of Jatropha based material.

1.3 Objective and Scope of Study

The objectives of this study are:

- To do research on Jatropha material as fluid loss reducer in drilling fluid under HPHT condition.
- To figure out the most efficient and economical way to reduce fluid loss by using jatropha.
- To study the better concentration for better stability under different pressure and temperature.

The scope of study includes:

- Produce the Jatropha material from jatropha plant seed.
- Plan a lab methodology to study the Jatropha material best concentration in getting the best fluid stability.

1.4 Relevancy of the Project

This project is directly related to Petroleum Engineering course since the main point focus on the fluid loss reducer based from Jatropha material. Fluid loss reducer is really important in term of Petroleum Engineering since it will lead to drilling fluid loss reduction. This will prevent formation damage and thus increase the potentiality of the formation to produce more.

1.5 Feasibility of the Project

The project can be done in the allocated time for the FYP duration. The experiment would not take much time, plus all the equipment are all readily available in UTP and no need to go out far. The Jatropha material making would not take more than 3 hours per samples and mud preparation for each sample would only take about 1 full day.

CHAPTER 2

LITERATURE REVIEW

2.0 Literature Review

2.1 Drilling Fluid Loss

Lost in drilling usually happened when there is uncontrolled flow of whole mud into the formation[4]. Any kind of formation that have a channel for the fluid to pass through can induced to fluid loss especially in permeable beds or formation that been fractured hydraulically or mechanically.

2.1.1 Induced Lost Circulation

Induced lost circulation is happened when the formation is not capable to withstand the pressure load from the excessive overbalanced condition due to the drilling fluid. The drilling fluid density or weight is the main reason for the fluid loss. The reason behind this scenario is always related to poor management of drilling fluid program and inaccurate well planning. Poor circulation system may also be one part of the fluid loss. Human error that resulted from mechanical part may too lead to the fluid loss such as pump surges, bit and stabilizer bailing, poor hole cleaning, abnormally high pump flow rates, poorly designed hole geometry and poor fluid properties, making it difficult to break circulation after the fluid has been static[4].

2.2 Fluid Loss Reducer

One of the ways to prevent the fluid loss is to have a good well planning program. The borehole pressure must be controlled in static and dynamic pressures and at all time, the pressure is below the fracture limit of the rock that is being drilled. Next is to closely monitor the drilling fluid properties and the additives that have been used. The aspect that should be prioritized is the solid controls, then the rheology and hydraulics in order to minimize the fluid gel strength, annular pressure losses, and equivalent circulating density (ECD)[4].

2.2.1 Fluid Lost Reduction Materials

Lost circulation has become a serious problem since the kick-off of drilling campaign. Varieties of materials have been identified in order to tackle the problem and eventually cure the lost circulation. Till this day, the lost circulation materials have been divided into four categories [4]:

1. Fibrous materials: such as shredded sugar cane stalks (bagasse), cotton fibers, hoghair, shredded automobile tires, wood fibers, sawdust, and paper pulp. These materials have relatively little rigidity, and tend to be forced into large openings. If large amounts of mud containing a high concentration of fibrous material are pumped into the formation, sufficient frictional resistance may develop to affect a seal. If the openings are too small for the fibers to enter, a bulky external filter cake forms on the walls of the hole and is knocked off when the well is cleaned out.

2. Flaky materials: such as shredded cellophane, mica flakes, plastic laminate and wood chips. These materials are believed to lie flat across the face of the formation and thereby cover the openings. If strong enough to withstand the mud pressure, they form a compact external filter cake. If not strong enough, they are

forced into the openings, and their sealing action is then similar to that of fibrous materials.

3. Granular materials: such as ground nutshells or vitrified, expanded shale particles. The latter are made by firing ground shales at temperatures up to 1800^0F (982^0C). These materials have strength and rigidity and when the correct size range is used, seal by jamming just inside the openings. Experiments by Howard and Scott shoed that the greater the concentration of particles in the mud, the larger the opening bridged, and that strong granular materials, such as nutshells, bridged larger openings than did fibrous or flaky materials, however, weak granular materials such as expanded perlite did not.

4. Slurries whose strength increases with time after placement, such as hydraulic cement, diesel oil-bentonite-mud mixes, and high-filter-loss muds. Neat cement is usually used only for squeezes at the casing shoe. If used to plug a channel in open hole, the strength of the cement may be reduced by contamination with mud. Also, a danger of sidetracking exists when drilling out cement left in the hole.

Material	Type	Description	Concentration (lb/bbl)
Nutshell	Granular	50% - 3/16+ 10 mesh 50% - 10+ 100 mesh	20
Plastic	Granular	50% - 3/16+ 10 mesh 50% - 10+ 100 mesh	20
Limestone	Granular	50% - 3/16+ 10 mesh 50% - 10+ 100 mesh	40
Sulphur	Granular	50% - 3/16+ 10 mesh 50% - 10+ 100 mesh	120
Nutshell	Granular	50% - 10+ 16 mesh 50% - 30+ 100 mesh	20
Expanded Percite	Granular	50% - 3/16+ 10 mesh 50% - 10+ 100 mesh	60
Cellophane	Laminated	3/4-in flakes	8
Sawdust	Fibrous	1/4-in particles	10
Prairie Hay	Fibrous	1/2-in particles	10
Bark	Fibrous	3/8-in particles	10
Cottonseed Hulls	Granular	Fine	10
Prairie Hay	Fibrous	3/8-in particles	12
Cellophane	Laminated	1/2-in flakes	8
Shredded Wood	Fibrous	1/4 in fibers	8
Sawdust	Fibrous	1/16 in particles	20

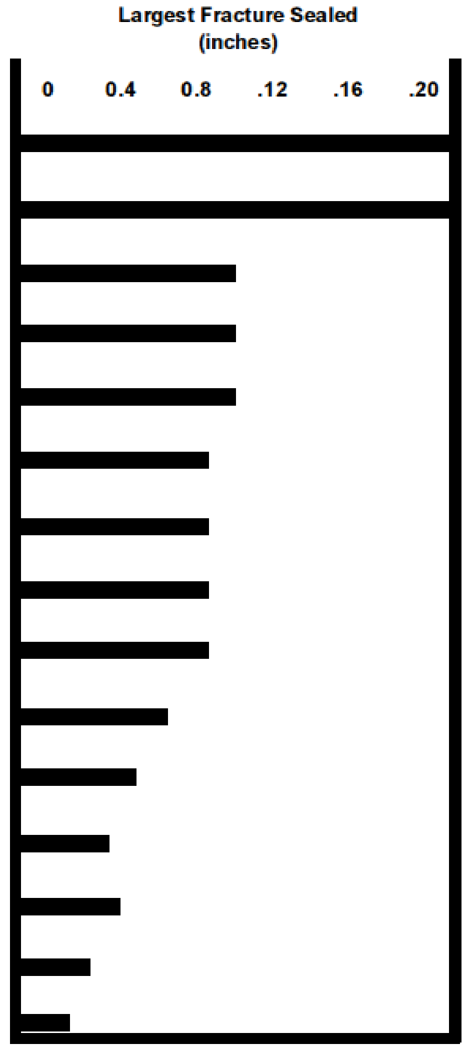


Figure 3: Fluid Loss Reducer Materials

2.3 Jatropha

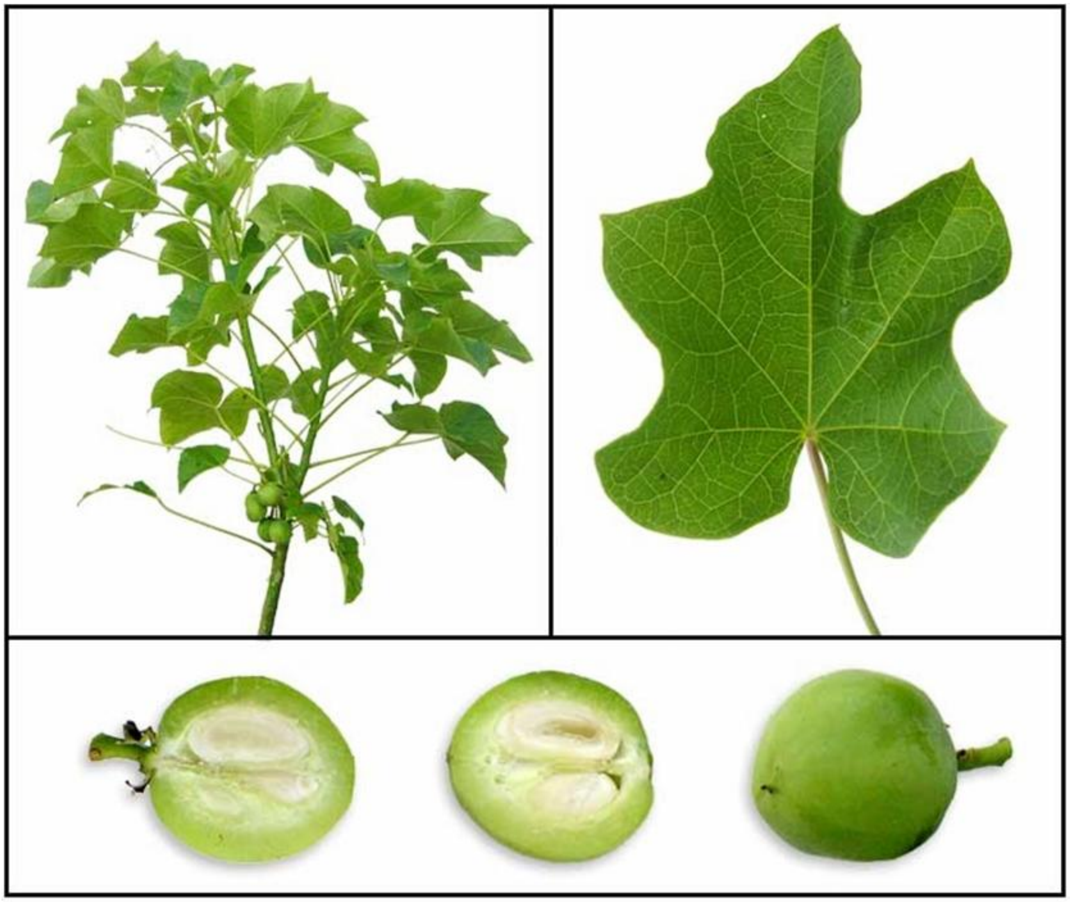


Figure 4:Picture showing Jatropha plant and seed.

2.3.1 Overview

2.3.1.1 Definition and Origin

Jatropha is a type of plant that come from the family Euphorbiaceae. The name Jatropha comes from Greek which is "Jatras" that means Doctor and "Trophe" that means Nutrition [5]. First it is a native plant in Central America and now it has been produced in other subtropical areas such as India, Africa and North America. Originally the Jatropha comes from Caribbean, however Portugese traders have brought the Jatropha out to Africa and Asia[5]. The Jatropha has a really advantageous point where it is resistant to drought and pests and has seeds that contain up to 30-40 % of oil content. The seed is usually crushed in order to extract the oil to be used as biodiesel and the remaining will be used as biomass for powering electricity[5].

2.3.1.2 Production

Jatropha can grow in many types of soil such as gravelly, sandy and saline soils[5]. It also can grow on low fertility soils. However it still need fertilizer in order to increase the yield. Jatropha still can survive in drought season by dropping its leaves, but in order to make the jatropha produce the seed, it needs at least 600 mm of rain annually[5]. Jatropha grows up to 3 metres high and can be harvested 2 to 3 times per year. The plant can produce seeds until 30 years[5].

2.3.2 Usage of Jatropha

2.3.2.1 Medicine

Jatropha uses in the whole world have been established a long time ago and earn the name of "physics nut" by local folk[6]. It can help in malaria treatment, windbreak for animals and many more. However, the plant sap is irritant to the skin.

In few years back, many studies have been done on its medical commercial value. Extracts from bark of *Jatropha macarantha* are being sold as raw drugs which been used as male sexual stimulant[6]. It also contains characteristics such as antitumor, cytotoxic, anti inflammatory, molluscicidal and fungicidal properties[6].

2.3.2.2 Pharmaceutical

There is also research on the potential of the Jatropha to be used in pharmaceutical area. It claims that it can be used as serum, hair cleanser, body wash, body scrub, bar soap, shampoo, foam, mousse cream, lotion and pomade[7].

2.3.2.3 Oil and Gas Industry

2.3.2.3.1 Biodiesel/biofuel

There is extensive research on oil from Jatropha seed in India.The seed contains high number of oil which is about 33-60%. The oil extracted from mechanical ways is processed and can be used as biodiesel for running diesel engine. The government of India has fixed the bio diesel price as Rs 25 per litre[8].

2.3.2.3.1 Alternative Oil Based Mud

There is a study from Covenant University, Nigeria which focused on environmental safe drilling mud using this plant seed called jatropha [9]. The oil was extracted from the jatropha seed and added to mud samples to study its stability for drilling operation as well as its toxicity, filtration, pH, viscosity, density and degree of safety to the environment. The jatropha oil mud was compared with conventional oil base mud.

The result shows that jatropha based mud has lower viscosity, that it has less resistance to flow and lower pressure losses[9]. The toxicity test also shows that it is safer and less harmful to the aquaculture and soil micro organism than the conventional oil base mud[9].

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

One of the main resources of the research will be from OnePetro website and drilling lab manual from UTP lab. It contains variety of research paper suitable for literature review and finding the basic knowledge of the research topic. Other than that, IRC does provide petroleum engineering handbooks that are available to be borrowed anytime. Last but not least, the supervisor for this research does help a lot in guiding to the right path and the valuable knowledge shared with the writer.

This part will show how to prepare the jatropha for the material to be used in the drilling fluid as fluid loss reducer. The methodology sequence would be as follows:

1. The dried version of jatropha seeds are bought from BIONAS company.
2. The seeds are crushed first by using mortar in order to reduce their sizes.
3. Then, the seeds are prepared to be inserted into oven in order to dry the seeds much more. The temperature was set to 60°C for 5 hours.
4. Before inserting the jatropha into ball milling, the ball mill jar was divided to three parts. One part was for ball mills, one part for sample and another part was for ball milling space.
5. The material will be milled for about 2 hours.
6. Step 4 and 5 were repeated for 4 and 6 hours.
7. Another jatropha seeds were taken in order to take their shells for another ball milling process.
8. The shells were crushed first by using mortar in order to reduce their sizes.
9. Then, the shells were inserted into oven by setting the temperature to 60°C for 5 hours.
10. The shells were also ball milled with 1:3 ratio.

11. The material will be milled for about 8 hours.
12. Step 10 and 11 were repeated for 16 and 24 hours.
13. The characterization of the material was analysed with XRD application.
14. The result will be recorded in the table.

Mud Mixing

1. The following materials were prepared first.

Materials	Amount (Gram)
Saraline 185 V	147.13
CONFI-MUL P	5.00
CONFI-MUL S	7.00
CONFI-GEL	7.00
CONFI-TROL/Jatropha Sample	8.00
Lime	8.00
Fresh water	81.05
Calcium chloride	29.36
Barite	190.66

2. Calcium chloride and fresh water were mixed first with magnetic stirrer for 10 minutes.
3. Saraline, Confi-Mul P and S were mixed in mud mixer for 4 minutes.
4. Confi-Gel was added slowly and mixed for another 5 minutes.
5. Confi-Trol was added slowly and mixed for another 2 minutes.
6. Lime was added slowly and mixed for another 2 minutes.
7. Brine was added slowly and mixed for another 15 minutes.
8. Barite was added slowly and mixed for another 33 minutes.
9. Step 1 to 8 were repeated but by using different Jatropha samples.

Mud Weight or Density Test

1. The mud weight test will be using typical mud balance.
2. The lid from the cup is removed and completely fills the cup with the mud to be tested.

3. The lid is replaced and rotated until firmly seated, making sure some mud is expelled through the hole in the cup.
4. The mud is washed or wiped from outside the cup.
5. The balance arm is placed on the base, with knife edge resting on the fulcrum.
6. The rider is moved until the graduated arm is level, as indicated by the level vial on the beam.
7. At the left hand edge of the rider, the density is read on either side of the lever in all desired units without disturbing the rider.
8. Mud temperature is noted down corresponding to density.

Mud Viscosity

1. The mud viscosity will be tested using Marsh Funnel.
2. With the funnel in an upright position, the orifice is covered using a finger and the mud is poured through the screen into a clean, dry funnel until the fluid level reaches the bottom of screen.
3. The finger is immediately removed from the outlet and the time required is measured for the mud to fill the receiving vessel to the 1 quart level.
4. The result is recorded into the table.

pH

1. The pH will be measured using pH meter.

Viscosity

1. Viscosity will be measured using FANN Viscometer.
2. A recently agitated sample in the cup is placed, tilted back the upper housing of the viscometer, locate the cup under the sleeve and lower the upper housing to its normal position.
3. The knurled knob is turned between the rear support posts to raise or lower the rotor sleeve until it is immersed in the sample to the scribed line.
4. Stir the sample for about 5 seconds at 600 rpm, and then select the RPM desired for the best.
5. Wait for the dial reading to stabilized.
6. Record the dial reading and RPM.

Gel Strength

1. Stir the sample at 600 rpm for about 15 seconds.
2. Turn the RPM knob to stop position.
3. Wait the desired time rest time.
4. Switch the RPM knob to GEL position.
5. Record the maximum deflection recorded on the dial.

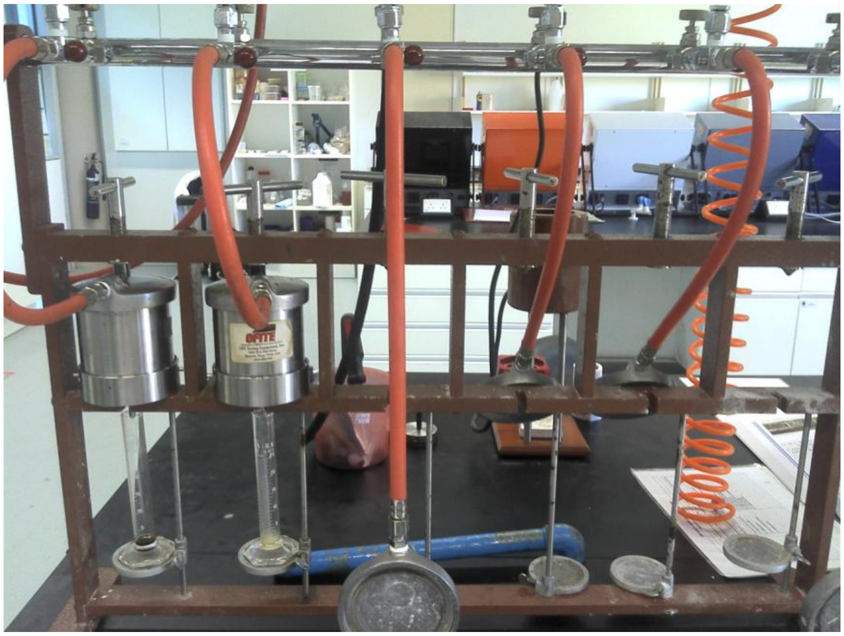


Figure 5: LPLT Filter Press

LPLT Filtration

1. Filtration test would be using Filter Press.
2. Detach the mud cell from filter press frame.
3. Remove bottom of filter cell, place right size filter paper in the bottom of the cell.
4. Introduce mud to be tested into cup assembly, putting filter paper and screen on top of mud tighten screw clamp.
5. With the air pressure is set to 1 atm, clamp the mud cup assembly to the frame while holding the filtrate outlet end finger tight.
6. Place a graduated cylinder underneath to collect filtrate.
7. Open air pressure valve and start timing at the same time.
8. Report cc of filtrate collected for specified intervals up to 40 minutes.
9. Record the data in table.

Mud Cake Build

1. The mud cake thickness will be measured using vernier caliper.

Figure 6: HPHT Filter Press

HPHT Filtration

1. The mud sample was first hotrolled for 16 hours under 120°C and 100 psi.
2. After the mud sample was taken out from the hotroller and cooled down, the mud will be poured into the HPHT cup.
3. The HPHT cup will be tightly sealed.
4. The temperature was set to 250°F. Once the temperature is achieved, the cup is inserted.
5. 100 psi of pressure was introduced into the HPHT cup.
6. Once the cup's temperature achieved 250°F, the pressure applied is increased to 250 psi.

7. Place a graduated cylinder underneath to collect filtrate.
8. Open air pressure valve and start timing at the same time.
9. Report cc of filtrate collected for specified intervals up to 40 minutes.
10. Record the data in table.

Mud Properties

Sample No.	Mud Weight			Marsh Funnel Viscosity	pH
	ppg	Psi/1000	Pp ft^3	sec	pH
1					
2					
3					

Mud Rheology

Sample No.	Viscosity CP	Gel Strength
1		
2		
3		

Filtration

Sample	Pressure (psi)	Mud Thickness (mm)	Fluid Loss (ml)
1	14.7		
	250		
2	14.7		
	250		
3	14.7		
	250		

3.2 Project Work

Figure 4 shows the process flow of the Final Year Project :

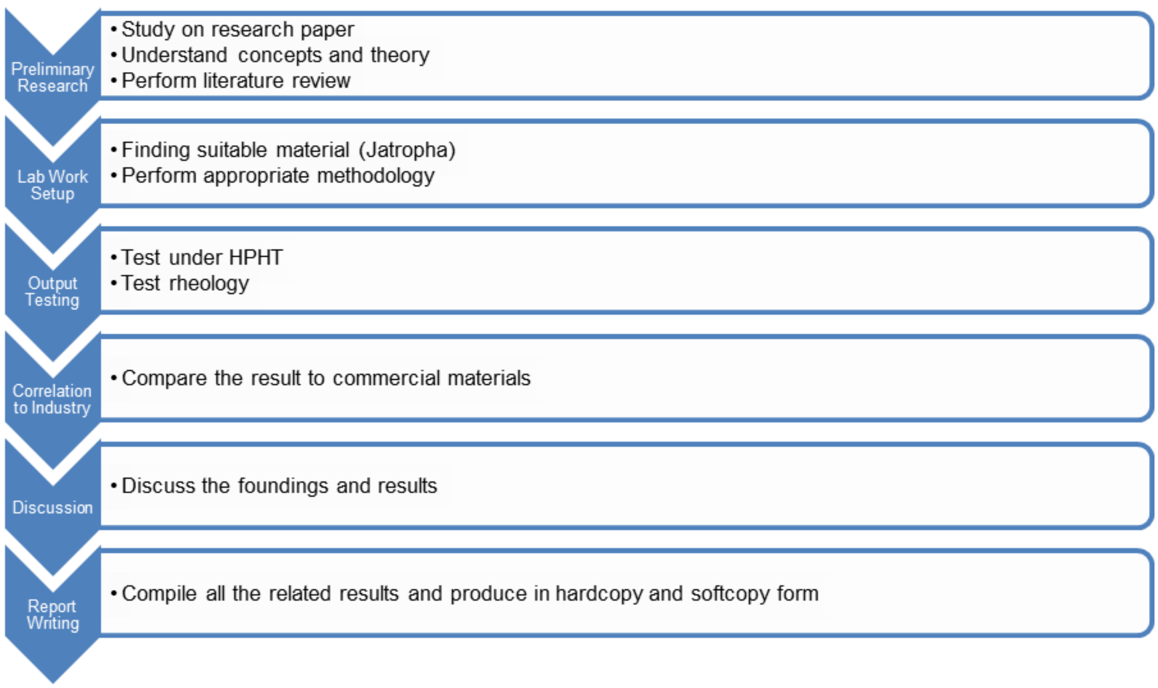


Figure 7: Process flow of work

3.3 Gantt Chart and Key milestones

Table 1 shows the Gantt chart to schedule the implementation of Project:

No	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
1	Topic Selection/Proposal																														
2	Preliminary Research Work																														
3	Submission of Proposal Defense Report																														
4	Proposal Defense																														
5	Project Work Continues																														
6	Submission of Interim Draft Report																														
7	Submission fo Interim Report							S														S									
8	Acquisition of Jatropha seeds							E														E									
9	Briefing of FYP 2							M														M									
10	Lab Booking																														
11	Crushing and Drying							B														B									
12	Ball Milling							R														R									
13	Acquisition of Mud Additives							E														E									
14	Mud Mixing & Rheology Check							A														A									
15	Submission of FYP2 Progress Report							K														K									
16	HPHT & LPLT Filter Press																														
17	Pre-Sedex Poster and Presentation																														
18	Submission of Dissertation (soft bound)&Technical Paper																														
19	XRD & SEM																														
20	Oral Presentation																														
21	Submission of Hard Bound Dissertation																														

Legend

Task

Key milestone

Table 1: Gantt chart and key milestones for the project implementation

3.4 Tools

1. Ball milling equipment in Building 17.
2. Scanning Electron Microscope analyser.
3. XRD Characretization application.
4. Typical mud balance.
5. Viscometer.
6. LPLT and HPHT Filter press equipment.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Jatropha Seeds Preparation

The jatropha seeds were first being dried in an oven for 5 hours in 60^{o}C in order to keep the seeds are dried as possible in order to be prepared for it to undergo ball mill process.

Figure 8: Oven temperature was set to 60^{0} C.

The sample was first being put inside a metal container that can stand the allocated temperature which is 60^{o}C for a period of time (5 hours). Picture below shows the jatropha seeds being prepared in the metal container before put inside the oven.

Figure 9: Jatropha seeds prepared in metal container before being put inside the oven for drying.

For every one hour passed drying, the sample was taken out for observations in order to ensure the sample is thoroughly dried and stirred with spoon for the sample to dry evenly.

After 1 hour drying.	After 2 hours drying.
	

After 3 hours drying.	After 4 hours drying.
	
After 5 hours drying. 	

After the sample is dried for 5 hours in 60°C, the sample is then taken out and left to be cooled down. Then, the sample is securely kept in plastic container for processing with ball mill equipment.

4.2 Jatropha Seeds Ready for Ball Mill

Figure 10: Glass container which will be used for ball milling process. One part is filled with jatropha sample, one part with ceramic balls and another part for ball milling space.

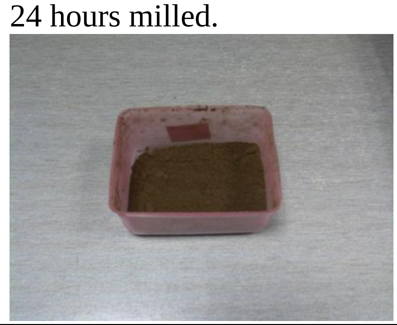
For ball milling process, the sample is kept in a glass container. One third is filled with sample, one third is filled with ceramic balls as crusher and other one third is the space for the milling process.

Figure 11: Ball milling in process. The speed was set to 100 rpm.

Ball milling in process. During this process, the equipment is set to 100 rpm for 2 hours, 4 hours and 6 hours.

2 hours milled.	4 hours milled.	6 hours milled.
		

Ball milling is also done with jatropha sample but only with its shells. Since it is in really dried form, an extended time can be done for ball milling for 8 hours, 16 hours and 24 hours.

8 hours milled.	16 hours milled.	24 hours milled.
		

4.3 Jatropha seeds samples sent for XRD scan

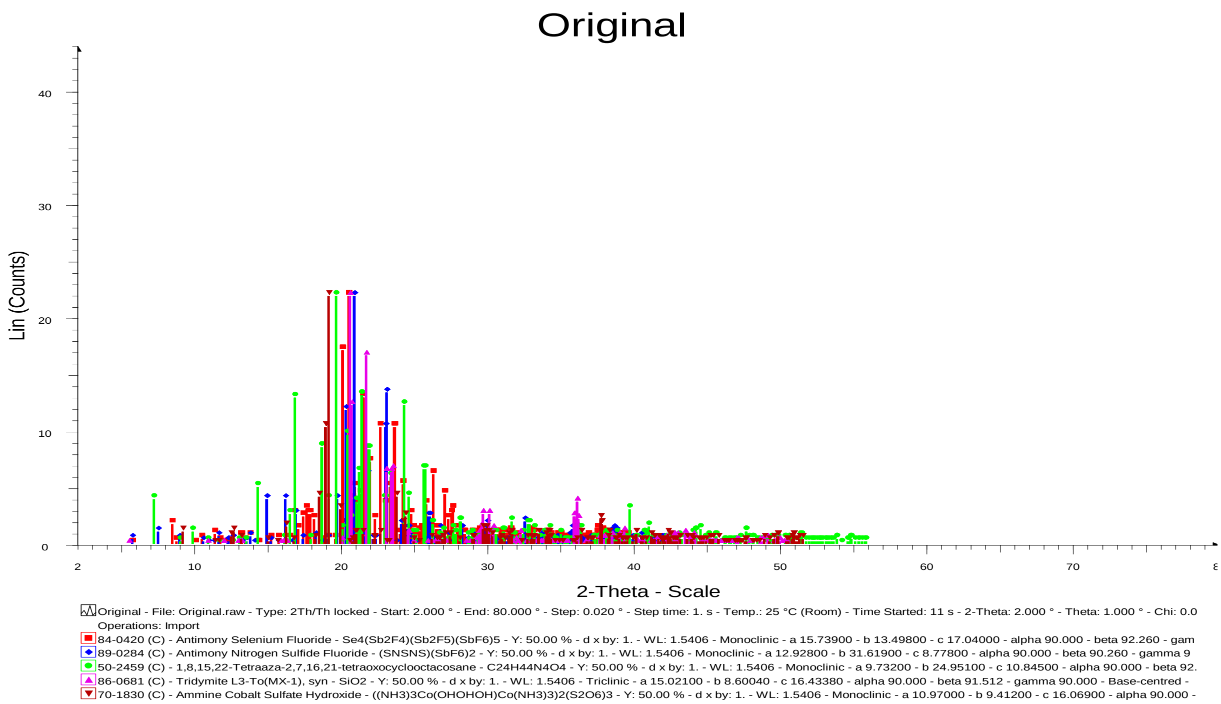


Figure 12: Original sample XRD scan.

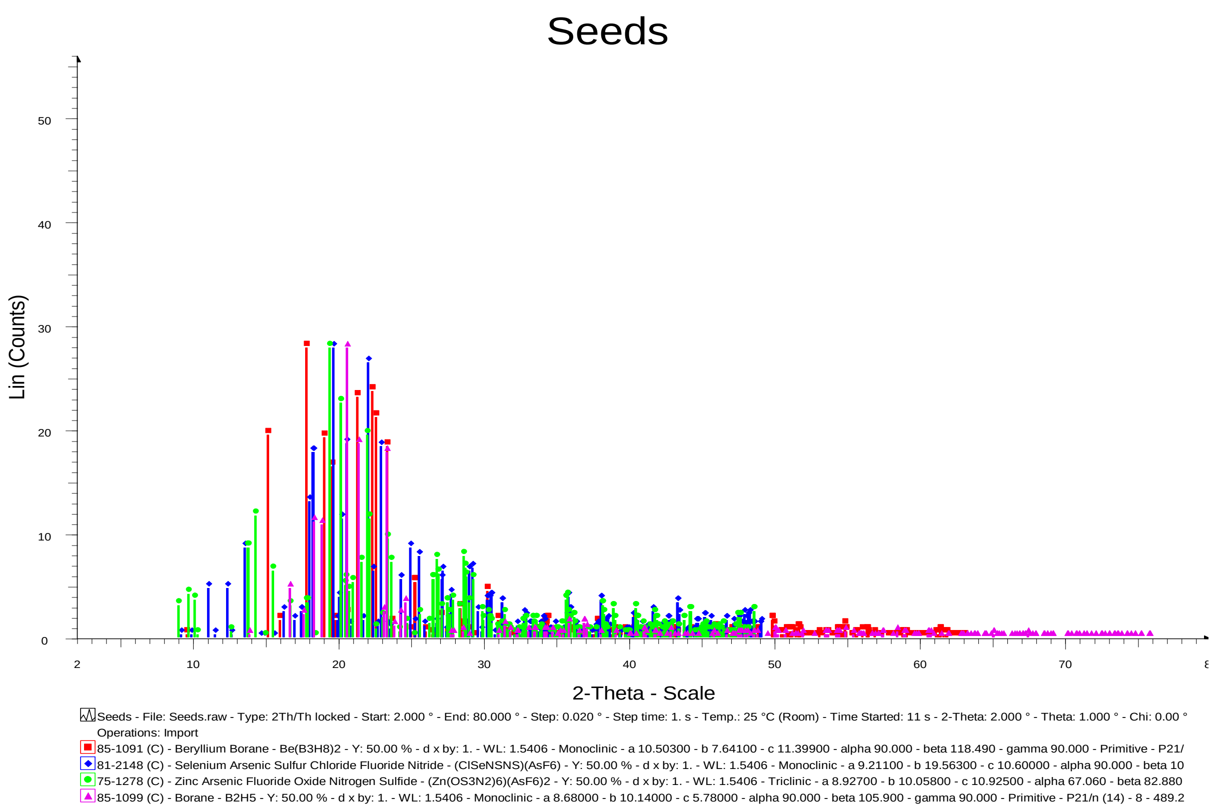


Figure 13: Jatropha seeds XRD scan.

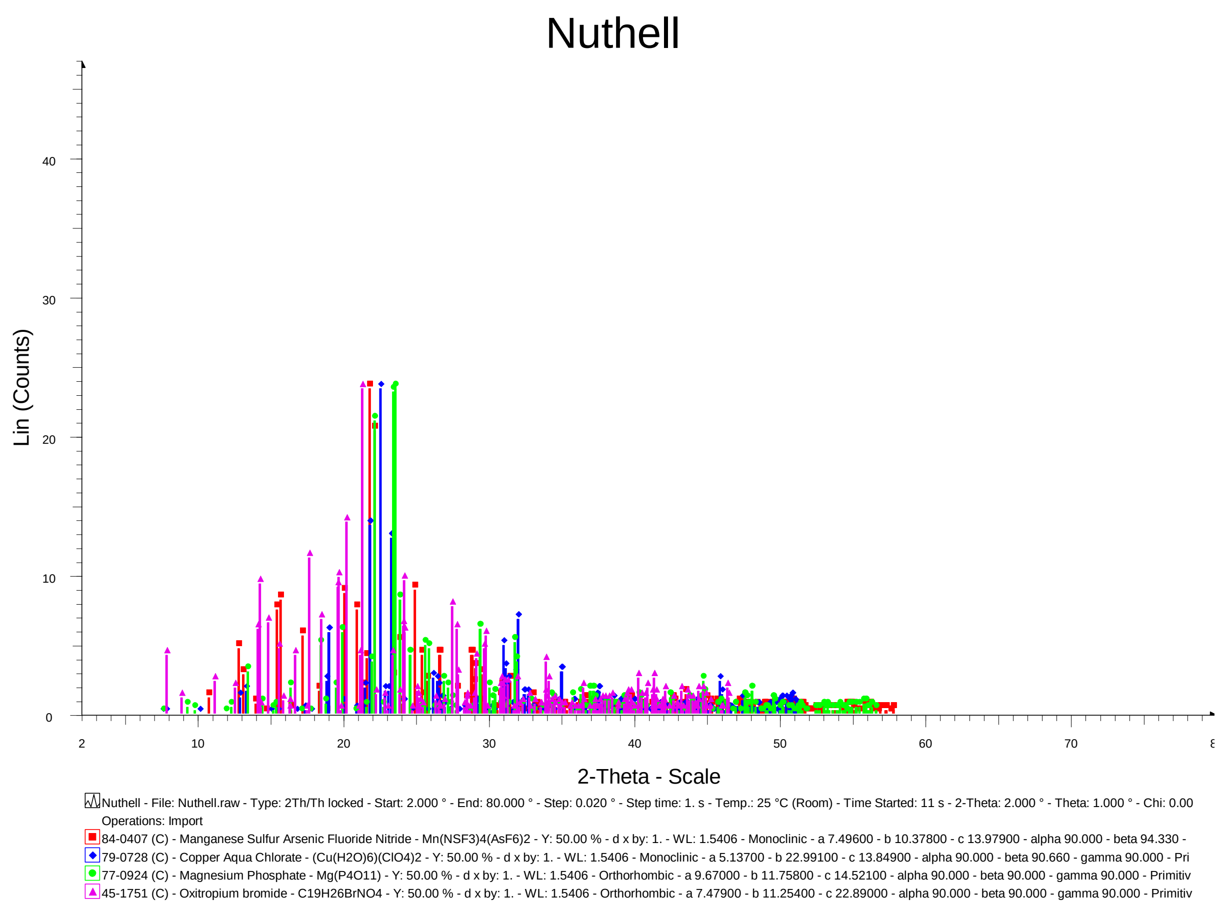
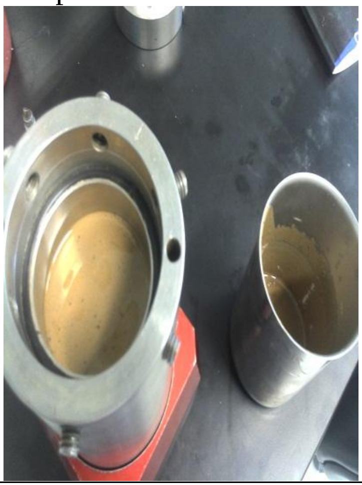


Figure 14: Jatropha nutshell XRD scan.

4.4 Jatropha Sample Used As Fluid Loss Reducer Additive

Then, the sample is used in mixing the mud as fluid loss reducer additive.

Jatropha 2 hours mud sample. 	Jatropha 4 hours mud sample. 	Jatropha 6 hours mud sample. 
Jatropha 8 hours mud sample. 	Jatropha 16 hours mud sample. 	Jatropha 24 hours mud sample. 

After it is hot roll for 16 hours, the mud is used for HPHT test. The result for mud rheology and the HPHT is tabulated as below.

Mud Rheology Test Result Before Hotroll.

Sample No	Viscosity (cp)					Gel Strength	
	600 rpm	300 rpm	Plastic	Apparent	Yield Point	10 seconds	10 minutes
Control Mud	80	44	36	40	8	8	12
Jatropha 2 hours	74	44	30	37	14	9	13
Jatropha 4 hours	80	45	35	40	10	10	15
Jatropha 6 hours	82	50	32	41	18	11	16
Jatropha 8 hours	69	39	30	34.5	9	11	18
Jatropha 16 hours	73	40	33	36.5	7	16	16
Jatropha 24 hours	71	40	31	35.5	9	8	12

Figure 15: Mud rheology test result before hotroll.

Mud Rheology Test Result After 16 Hours After Hotroll

Sample No	Viscosity (cp)					Gel Strength	
	600 rpm	300 rpm	Plastic	Apparent	Yield Point	10 seconds	10 minutes
Control Mud	125	71	54	62.5	17	9	25
Jatropha 2 hours	90	60	30	45	30	10	16
Jatropha 4 hours	65	38	27	32.5	11	8	10
Jatropha 6 hours	117	68	49	58.5	19	10	20
Jatropha 8 hours	95	50	45	47.5	5	10	22
Jatropha 16 hours	77	45	32	38.5	13	12	12
Jatropha 24 hours	97	57	40	48.5	17	8	14

Figure 16: Mud rheology test result after 16 hours hotroll.

HPHT Filter Press

	Sample						
Time (minute)	Control Mud	Jatropha 2 hours	Jatropha 4 hours	Jatropha 6 hours	Jatropha 8 hours	Jatropha 16 hours	Jatropha 24 hours
1	2	1	2	2	1	1	1
2	2.5	2	3	3	2	1	1
3	2.5	2	3.5	4	2.5	1.5	1.5
4	2.5	2.5	4	4.5	2.5	2	1.5
5	2.5	3	5	5	2.5	2	2
10	3.0	4	7	6.5	4	2.5	2.5
20	4.0	5	10.5	9	5.5	3	2.5
30	4.5	6	10.5	10	7	3.5	3
40	4.8	6.5	11	10	8	4	3.5

Figure 17: HPHT filter press fluid loss result.

Mud Cake Thickness

Sample	Thickness (mm)	Picture
Control Mud	2.3	
Jatropha 2 hours	2.4	
Jatropha 4 hours	2.3	

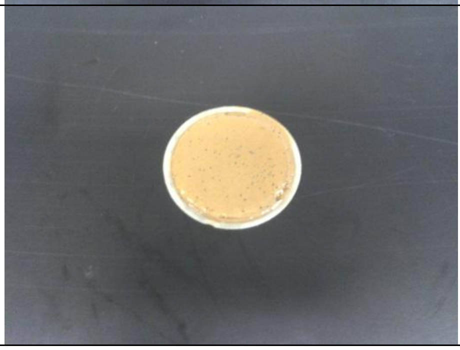
Jatropha 6 hours	2.3	
Jatropha 8 hours	2.4	
Jatropha 16 hours	2.4	
Jatropha 24 hours	2.3	

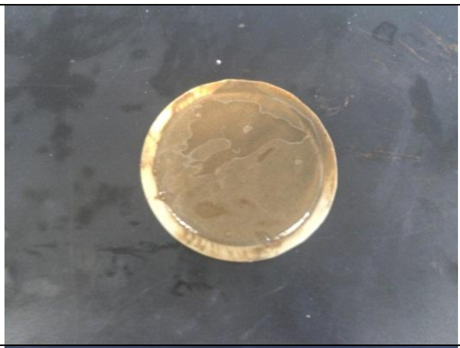
Figure 18: Mud cake thickness formed after HPHT filter press.

LPLT Filter Press

	Sample						
Time (minute)	Control Mud	Jatropha 2 hours	Jatropha 4 hours	Jatropha 6 hours	Jatropha 8 hours	Jatropha 16 hours	Jatropha 24 hours
1	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0
3	0	0.5	0.5	0	0	0	0
4	0	1.0	1.0	0.5	0	0	0
5	0	1.5	1.0	1.0	0	0	0
10	0.5	2.5	1.5	1.5	0.5	0	0
20	1.0	3.0	2.0	2.0	1.0	0.5	0.5
30	1.5	3.5	2.5	2.5	1.0	1.0	0.5
40	2.0	4.0	3.0	3.0	1.5	1.5	1.0

Figure 19: LPLT Filter Press result

Mud Cake Thickness

Sample	Thickness (mm)	Picture
Control Mud	0.9	
Jatropha 2 hours	1.5	
Jatropha 4 hours	1.3	

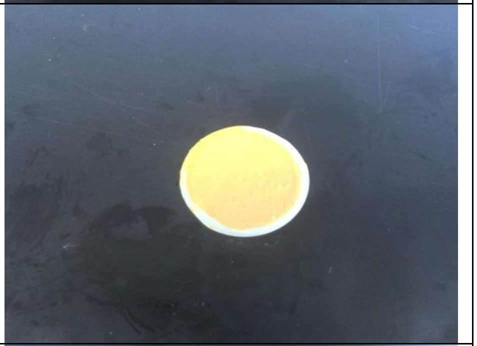
Jatropha 6 hours	1.4	
Jatropha 8 hours	1.1	
Jatropha 16 hours	1.2	
Jatropha 24 hours	1.2	

Figure 20: Mud Cake Thickness for LPLT Filter Press

4.5 Discussion

The XRD results are based on the type of samples that are collected. First sample would be the original sample which is straight from the Jatropha seeds without any altering of its physical properties. It shows various chemical compounds for example Serenium Floride, Nitrogen Sulphide and Cobalt Sulphate Hydroxide. This would be the original chemical compounds that existed in the Jatropha seeds. For the second sample, the Jatropha seeds are crushed by using ball mills. There are some impurities due to the contact of the ball mills which shows the existence of Zinc Arsenic Floride and Berillium Borane. The reason behind it would be the ball mill itself which while crushing the seeds, may also crushed some of the balls themselves. Other than that would be the cleanliness of balls which the balls have been used so many times with other projects too although the cleaning process of the balls have been attempted before the start of the experiment. For the last sample, the dry nutshell of the Jatropha is used. There are existence of chemical compounds such as Manganese Sulfur and Magnesium Phosphate. This is also due to the impurities from the ball milling process.

Although there are impurities inside the samples, the main structure of the Jatropha seeds is still remained. The impurities do not really affect the capability of the Jatropha as fluid loss reducer since the impurities are in small value and almost invisible to naked eye.

The reason for the difference in time allocated for each sample ball milling is its content. For the first 3 samples which are being ball milled for 2 hours, 4 hours and 6 hours are totally made from full jatropha seeds, the shell including the inside. For the first three samples, the HPHT filter press showed that the result is totally a failure since it surpassed the control mud fluid loss from 4.8 ml to 10.5 ml which is almost 2 times the fluid loss. After all, the sample cannot be ball milled any time longer than 6 hours because when the jatropha seeds are ball milled for 6 hours, the outcome is totally wet and sticky. When this happened, the ceramic ball cannot crush the sample effectively because the sample is sticky. This is because of the high oil content inside the seeds. Thus, the size reduction is really bad and cannot act as good fluid loss reducer which needs to be in small size to fill the void in the mud cake. This void is important to be filled because it will prevent the fluid to flow through.

Thus, another attempt to make a good fluid loss reducer is by using the jatropha shell alone. The jatropha shell shows great result after being ball milled for up to 8 hours. It is still dry and shows real improvement in size reduction. It managed to be ball milled until powder form. Thus, in order to get the best size reduction, the jatropha shell is ball milled for another 16 hours and 24 hours. After HPHT filter press, jatropha shell that are being ball milled for 16 hours and 24 hours shows lesser fluid loss than control mud which contain commercial fluid loss reducer. They managed to make it 4 ml and 3.5 ml for 16 hours and 24 hours each compared to control mud which fluid loss is 4.8 ml.

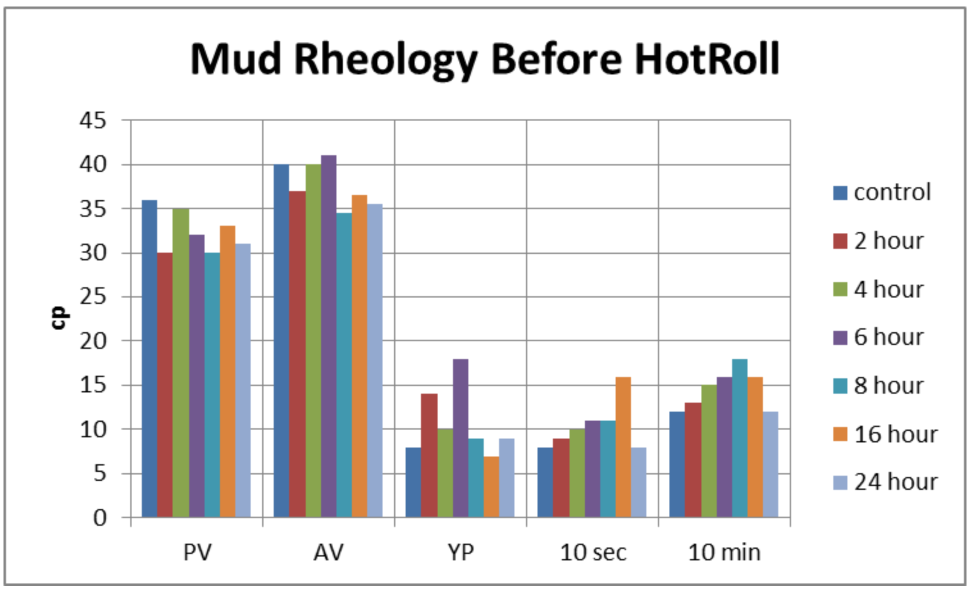


Figure 21: Mud rheology before hotroll.

After the muds are hotroll, there are some changes in the mud rheology. It may be because of the long exposure to high temperature which is 250°F and pressure for 100 psig. The hotroll is done was because to apply with API standard on mud. Before doing experiment about mud in HPHT condition, it must be undergoing aging condition by hotrolling for 16 hours.

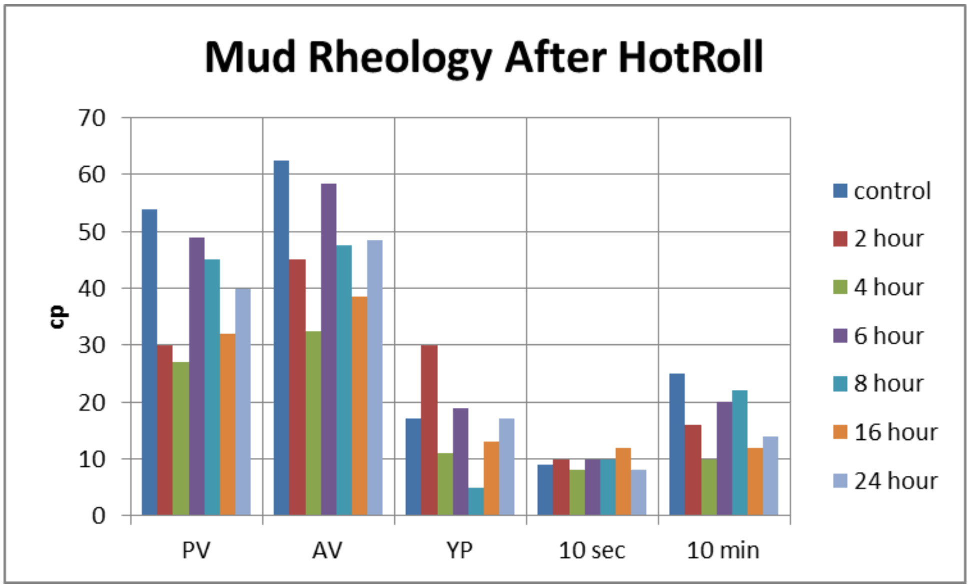


Figure 22: Mud rheology after hotroll.

From above mud rheology graph, the plastic viscosity for control mud is the highest for about 52 followed by 6 hours, 8 hours and 24 hours. For 2 hours, 4 hours and 16 hours, it shows low plastic viscosity. Plastic viscosity is important since it will indicate how easy or hard for the bit to drill. Low plastic viscosity will make the drill bit to drill easier and faster compared to high plastic viscosity.

The yield point for 2 hours shows the highest for about 30 followed by control mud, 6 hours and 24 hours. Yield point for 4 hours, 8 hours and 16 hours show low yield point value. The high yield point for 2 hours mud must be because of its unstable mixture which contains large particles of jatropha seeds. That is why it shows really different value compared to other samples. High yield point value shows that it can better lift cuttings out of the annulus.

Gel strength for each samples show that it does not have high difference. For 10 seconds, the difference from the highest to lowest value is only 3. It must be due to the brief time that the mud is not yet be able to be stabilized as possible. The gel strength shows big differences when the muds are left for 10 minutes. Control mud has the highest gel strength which is about 25 while lowest is 10 by 4 hours mud. Gel strength if possible should be as low as possible because if the gel strength is high, it means that the bit would has problem to start drilling again after certain time drilling is stopped for example by tripping out. The high gel strength would make the bit difficult to rotate again.

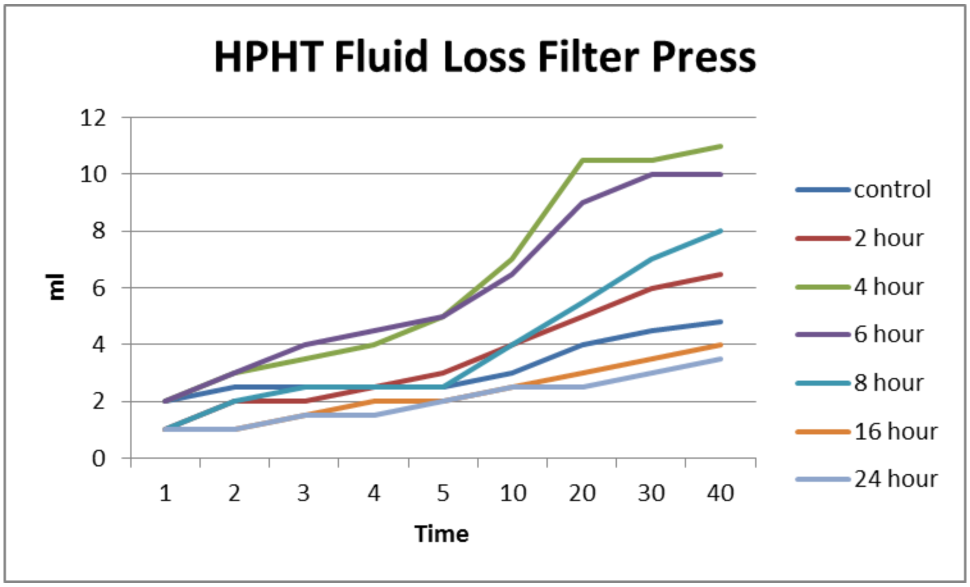
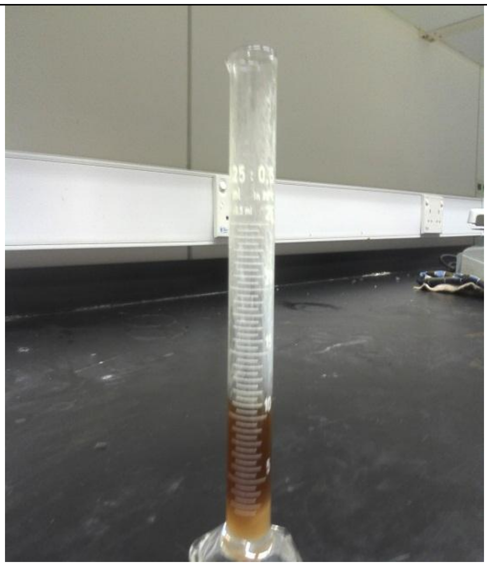
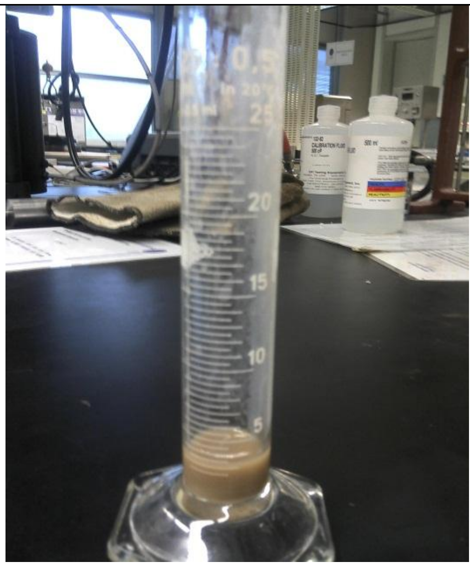


Figure 23: HPHT fluid loss filter press graph comparison.

Control mud is mixed with commercial fluid loss reducer which is Confi-Trol. The control mud shows that it can reduced fluid loss down to 4.8 ml. Other type of sample muds shows higher fluid loss value than the control mud except for 16 hours and 24 hours muds. The greatest achievement would be 24 hours mud which can reduce fluid loss down to 3.5 ml. Plus another great victory would be the 24 hours mud rheology is not really in big difference compared to control mud. Thus the 24 hours mud served its purpose to be fluid loss reducer which is better than the Confi-Trol.

	
Fluid loss from the 4 hours ball milled. (Worst fluid loss - 10.5 ml)	Fluid loss from the 24 hours ball milled. (Best fluid loss - 3.5 ml)

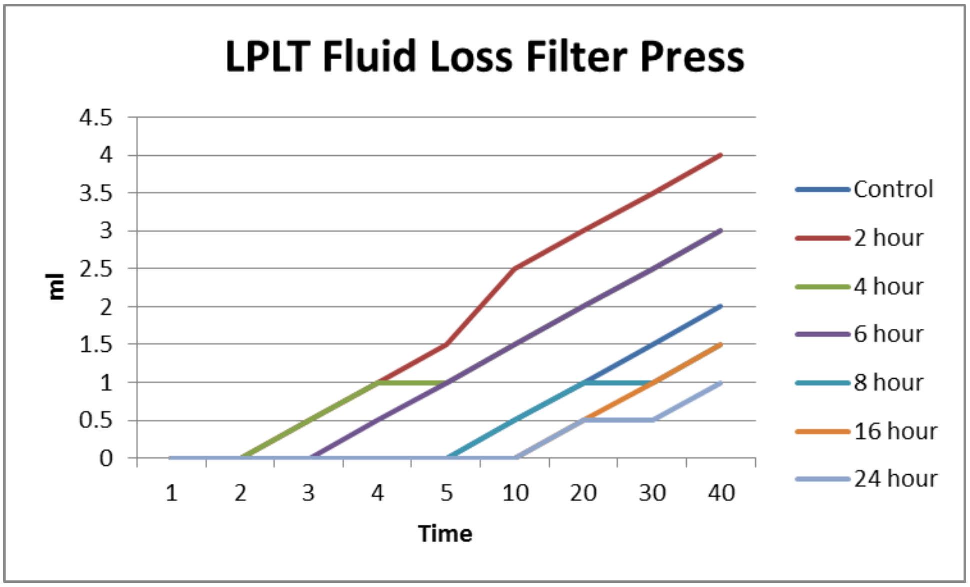
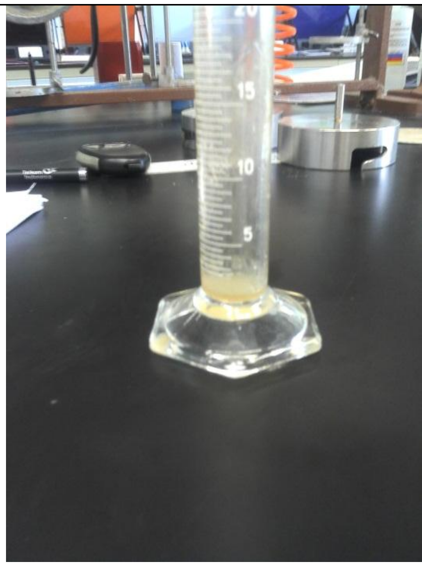


Figure 24: LPLT Fluid Loss Filter Press

Based on the graph, the worst fluid loss reducing agent would be jatropha sample for 2 hours. It has the highest fluid loss which is 4 ml. However, the comparison with the control mud shows that there are 3 jatropha samples that show better result which are Jatropha 8 hours, 16 hours and 24 hours. For the best result would be 24 hours which manage to reduce the fluid loss to 1 ml compared to control mud which is 2 ml. This shows that the sample can be an alternative for fluid loss reducer other than the commercialized one which in this case is CONFI-TROL.

Fluid loss from the control mud. (2 ml)



Fluid loss from the 24 hours ball milled. (Best fluid loss - 1 ml)

CHAPTER 5

CONCLUSION

5.1 Conclusions

Jatropha seeds shells after a special preparation which include size reduction are capable to be an alternative for commercial fluid loss reducer additive. The superb result shown from HPHT fluid loss filter press shows the ability of the Jatropha seeds shells to reduce the fluid loss even better from commercial fluid loss reducer.

Jatropha materials have high potential to become the next big phenomenon in oil and gas industry. Together with the scientists that are willing to do research to study more about the capability of the new material such as Jatropha, one day the material will create more opportunities to search oil and gas in more complex and dangerous region. In the future, the new material will profit the oil and gas industry in term of cost effective and time consumption.

5.2 Recommendation

Control of Humidity

While doing the ball mill with the jatropha seeds, the seeds do show that they contain oil content in the seeds. Thus, the ball mill needs to be in low humidity as possible in order to produce the product in powder form and not in wet and sticky form. Thus, while doing the ball mill, it is advisable to introduce some heat to increase the temperature in order to make the whole process of ball milling is in dry condition.

Further reduction in size

Based from the result above, it shows that the jatropha can help in replacing the commercial fluid loss reducer if the particle size of the sample can be reduced more. However, due to the limitation with the equipment lab, the best that can be done is only to ball mill for 24 hours (maximum 100 rpm only). If the process is made longer, there would be small significant result improved. That is why better equipment for size reduction such as planetary ball mill (can be up to 500 rpm) or equivalents is needed in order to get the maximum size reduction as possible.

HPHT Temperature Control

While doing this experiment, the temperature for HPHT filter press cannot be set accurately because the knob only shows toward a number (1 to 9) and not temperature. So, if the temperature is desired for example 250^0 F, the user gets confused which number should be referred to. Thus, it would be advisable to introduce a dial or knob that been able to set the temperature directly and not to be play around with the numbers.

LPLT Metal Filter

The metal filter is needed in order to support the filter paper while doing the filter press. Thus, in order to get a more accurate result, a clean metal filter is needed in order to make sure the filter paper be able to filter the mud and form a mud cake evenly.

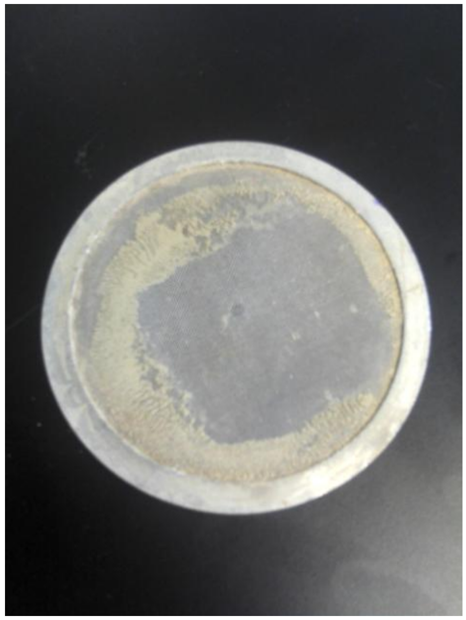


Figure 25: Dirty Metal Filter

5.2 Suggested Future Work for Expansion and Continuation

Use different concentration of Jatropha seeds inside the mud.

It would be better if the Jatropha seeds were also be compared according to the percentage or portion of the sample in the oil based mud. By comparing the result, it can show the best concentration to be used in the mud and thus will help in determining the economic value. Is it really had to be in 8 gram or could it be reduced more? If it can, there is possibility for the Jatropha for commercial value.

NOMENCLATURE

ECD	=	Equivalent Circulating Densities
HPHT	=	High Pressure High Temperature

REFERENCES

1) DeBrujin G., Skeates C. (2008). *High Pressure, High Temperature Technologies*. Oil Field Review, Schlumberger.

2) Payne M.L., Patillo P.D., Miller R.A., Johnson C.K. (2007). *Advanced Technology Solutions for Next Generation HPHT Wells*. IPTC 11463, International Petroleum Technology Conference, Dubai, U.A.E.

3) Godwin W., Ogbonna J., Boniface O. (2011). *Advances in Mud Design and Challenges in HPHT Wells*. SPE 150737, Nigeria Annual International Conference and Exhibition, Abuja, Nigeria.

4) Darley H., Gray G. (1988).*Composition and Properties of Drilling and Completion Fluids*. Woburn. Gulf Professional Publishing.

5) *Jatropha Cultivation, Production, Properties and Uses*, Fosfa Newsletter.

6) J Am Oil Chem Soc., (2011). "Jatropha Diterpenes : a Review.", *Journal of The American Oil Chemists' Society* **88(3):** 301-322.

7) Deo N. (2012). *Plant Extracts, Composition Containing Same, and Uses Thereof*. US Patent Online.

8) Mohammad Abdulla, (2008). *Morpho-Physiological and Molecular Diversity in Jatropha Curcas (L.)*, MSc., Thesis, University of Agricultural Sciences, Dharward, INDIA.

9) Adesina F., Anthony A., Gbadegesin A., Eseoghene O., Oyakhire A. (2012). *Environmental Impact Evaluation of a Safe Drilling Mud*. SPE 152865, SPE Middle East Health, Safety, Security, and Environment Conference and Exhibition, Abu Dhabi, U.A.E..

10) Arias A. (1975). *Chemical Reactions of Metal Powders With Organic and Inorganic Liquids During Ball Milling*. NASA 44135, Lewis Research Center, Cleveland, Ohio, U.S.A..

11) Siqueira V., Resende O., Chaves H. (2012). *Determination of the Volumetric Shrinkage in Jatropha Seeds During Drying*. Ciencia e Tecnologia Goiano, Goias, BRAZIL.

12) Rule A., Fergus A., Daellenbach C. (1985). *Influence of an Organic Polymer in Ball Mill Grinding of Quartz, Dolomite and Copper Ore*. RI 8964, Bureau of Mines Report of Investigation, U.S.A..

13) Stolle A., Szuppa T., Leonhardt S., Ondruschka B. (2011). *Ball Milling in Organic Synthesis: Solution and Challenges*. Institute for Technical Chemistry and Environmental Chemistry, Friedrich-Schiller University Jena, GERMANY.