

**Study of the Rheology of Oil – Based Drilling Mud (OBM) Subjected
to Green Weighting Agent Substitution**

by

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16390

Dissertation submitted in partial fulfillment of

the requirement for the

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(Chemical)

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CERTIFICATION OF APPROVAL

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Approved by,

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January 2015

CERTIFICATE 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 specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or person

FAKHRUL RAZI NASARUDIN

ABSTRACT

The aim of this study is to investigate the rheology properties of oil-based mud system using calcium carbonate from cockle shell as the weighting agent. This study will conduct experiments focused on the rheology characteristics and electrical stability to compare the properties commercial mud system using commercial calcium carbonate and cockle shell's calcium carbonate. The scope of this study will cover range of 9 lb/gal mud to 13 lb/gal mud. The results of the studied properties will be compared with the acceptable range provided by the Standard Petroleum & Gas Hand book. Weighting agent is one of the main components that are needed to be included in the basic mud formulation. It used to weight up the mud to counter the wellbore formation pressure. Barite and calcium carbonate is the common weighting agent in mud system. In this study is focussing on comparing the efficiency of rheology calcium carbonate from cockle shell to commercial calcium carbonate from limestone. This study will answer the possibility of replacing weighting agent in the current mud system with green weighting agent as mention above to save the cost and to reduce the pollution to the environment caused by the quarrying to harvest the commercial weighting agent.

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Chapter 1

Introduction

Background Study

With the oil and natural gas are being the primary source of energy for the world the producers are always keep advancing their technologies to maximize the production of these resources. Currently, the drilling operation which is the only method for us to extract the oil from the earth crusts costs millions to produce a single well that yield oil. In oil and gas industries, cost, environment, health and safety impact from drilling have been the vital issues in this century. In the past time companies that involved in this industry did not consider health and environment matters seriously which causes the area of drilling in the deprivation of pollution affected by the usage of chemicals in drilling fluids^[1].

In this study, the application of green-weighting agent is used to replace the current weighting agent in drilling fluids. Drilling fluid is a fluid that aids the drilling operation of the borehole down to the earth crusts. It has several important role in drilling operation that includes [2]:

- Transporting cuttings to the surface.
- Suspending the cuttings when circulation is stopped.
- Cooling the bit and lessening drill string friction.
- Consolidating the tectonic pressure.
- Preventing inflows of formation fluids into the well.
- Acting as a drilling parameter.
- Transmitting power to a downhole motor.
- Providing geological information.

Weighting agent in drilling fluids is one of the components to accomplish the function in consolidating the tectonic pressure. In conventional drilling most of the pressure downhole can reach more than 10,000 psi (equivalent to 680.5 atm). Therefore, the method used is to provide much denser drilling fluid to comply with

the operating pressure; by which weighting agent is utilized to densify the fluids. Currently, most of the oil-based mud uses barite, i.e. is a mineral consisting of barium sulfate with a density of 4.2 ppg [3], as the weighting agent. Despite being the perfect weighting agent for oil-based mud to reach the required density Roger, Leuterman [1] mentioned that barites ore sources are inspected meticulously as the substance also contains traces of heavy metals, particularly cadmium and mercury impurities. Environmentalist discovered that these materials are hazardous to the marine life, thus decided to restrict the amount of barite used in drilling fluids.

Calcium carbonate is also another type of weighting agent used in oil-based mud formulation. This material is believed to have several advantages such as;

- Reduce pollution that might affect marine life.
- Lower the cost for mud development as it is cheaper than barite.
- Suitable for the formations that does not have extreme conditions requirement.
- Provide much less caustic formulation and less abrasive characteristics while drilling in payzone.

Calcium carbonates that are commonly used in the industry are harvested from three types of calcium carbonate rock, which are limestone, chalk and dolomite [4]. In the current drilling fluids industry, limestone is the main source for calcium carbonate. The definition of green-weighting agent in this study is to substitute the conventional calcium carbonate harvested through limestone to the one that is more sustainable and environmental-friendly. The high potential that has been identified is the waste cockle shell as Malaysia is in abundance for this resource. Based on the statistics by the Malaysian Fisheries Department, between the year 2012 to 2013, the country has constantly produced over 40,000 tonnes of cockle shells for food industry, totalling an area of culture of 10,314 hectares. Through this number, the waste resulting from the discarded shells could be projected to be highly abundant that signify its sustainable attributes towards the future usage.

This study is to investigate the feasibility and viability of waste cockle shell that could replace the current calcium carbonate source used as weighting agent in the drilling fluids, in the effort to support biodiversity in Malaysia and reduce the impact to the environment caused by limestone mining.

Problem Statement

By using natural harvested calcium carbonate, we can save a lot of costs on weighting agent for drilling fluids. This is deduced by estimating the specific gravity (SG) of both compound barite and calcium carbonate is at 4.2 and 2.2, respectively. Although Malaysia is rich with lime and dolomite resources, the mass quarrying activities will cause the source to deplete and thus affecting the environment from the perspective of biodiversity and air pollution subjected from the quarry mining activities.

For this matter, this study would like to proof that drilling fluids will be one of the biggest consumer for the calcium carbonate. Replacing the commercial calcium carbonate with the green weighting agent in this study subjected to calcium carbonate that is harvested from cockle shell will utilise the excess natural resources shell to avoid environmental effect from industry activities.

Other than that, the cost reduction would be a question on this study. How much will it reduce the cost of a barrel of oil-based mud that uses calcium carbonate as its main weighting agent? For this specific question the calcium carbonate used will be harvested from the waste cockles shell. Comparison will be made by using the industrial calcium carbonate and cockles shell in economic wise.

Despite the use of calcium carbonate has long been introduced in drilling fluids, the utilization of waste cockles shell as the alternative source for calcium carbonate is still an untested area that opens for exploration. This study is hoped to give insight on the performance of the waste cockle shell-based calcium carbonate to the overall formulation of the new oil-based mud.

Objectives

The main objective of this study is to compare the viability of using calcium carbonate that is harvested from cockle shells to be utilized as the weighting agent for oil-based mud. Its aim is to create other functions to the cockle shells waste by introducing it into the drilling fluids industry.

Scope of Study

This study will focus on mud rheology based on the weighting agent substituted from calcium carbonate sourced from limestone and dolomite to calcium carbonate of waste cockle shells. This specific mud tested will be with 10 ppg and cannot be used for deep well bore which have extreme condition which requires higher mud weight. This is because calcium carbonate will be effective to be weighting agent for the mud weight less than 14 ppg. This mud however will be tested in low temperature and low pressure condition for which suitable to imitate the top and possibly the intermediate section condition in well bore i.e. 250°F and below 500 psi.

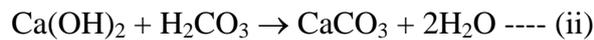
Other than that, the type of cockles will be used is *Anadara granosa* or locally-known as blood cockles. It is commonly found in the Indo-Pacific region such as the western coastal areas in Malaysia, and is considered cheap food sources prepared in many Malaysian local dishes [5]. The drilling fluids used will be limited to synthetic oil based fluids which commonly used in Malaysia offshore operation to suit the demand of mud in Malaysia.

Chapter 2

Literature Review

2.1 Calcium Carbonate & Cockle Shell ashes

Calcium carbonate is based on three elements which are also the basis of all other elements on the earth: carbon, oxygen and calcium. Calcium carbonate is commonly found on earth in three types of source rocks; limestone, dolomite and chalk. It is a simple salt which results from the reacted from burnt carbon dioxide (i) or lime that is been slaked (ii) according to formulas below:



The chemical formula for calcium carbonate is responded to the mass ratio which consist of calcium oxide at 56.03% to carbon dioxide at 43.97%, or varies at 40.04% of calcium to 59.96% carbonate [6].

Calcium carbonate has been applied widely in the many industries worldwide due to the cheap source of the material. Some of the field that involve calcium carbonate applications are:

- Paper : filler, coating pigment
- Plastics: fillers and reinforcing agents.
- Surface coatings systems
- Agriculture: lime treatment on the soil
- Oil and Gas: weighting agent, alkalisation & bridging agent

However there are differences between calcium carbonate that are from limestone and of cockle shells. According to Islam et al. [7], polymorphs that exist in cockle shells is aragonite type, whereas in the commercial-grade calcium carbonate is a calcite. Aragonite and calcite have the same chemical formula, CaCO_3 but their atoms are configured in different configurations. Aragonite was designed to have orthorhombic structure while calcite is in a trigonal arrangement [8].

According to Alden [8], generally calcite is more stable compared to aragonite in temperature wise where aragonite cannot withstand temperature above 400°C for a long period.

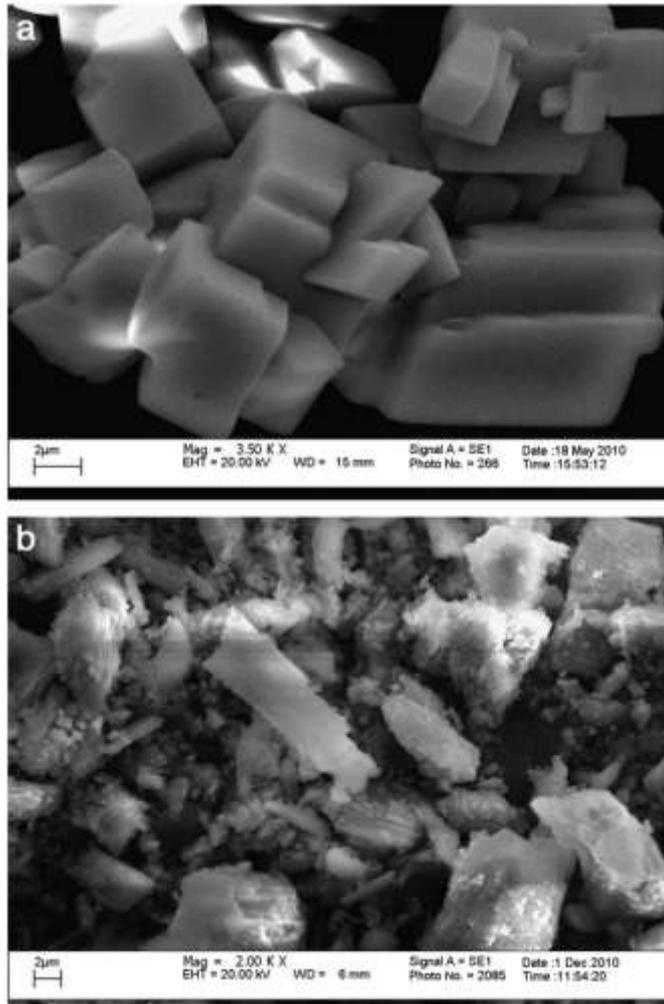


Figure 1: Surface of Morphology

The surface morphology of (a) commercial calcium carbonate powder and (b) the cockle shells powder were studied using VPSEM. Aggregated cubic-like calcite crystals were observed in the commercial calcium carbonate, whereas rod-like aragonite crystals were found in the cockle shells powder. Courtesy of Islam, Bakar^[7]

In Islam et al. [7] research, the authors prepared a guidelines to characterise calcium carbonate and identify the polymorphs structure in cockle shells. The study stated that the method used was by using a variable pressure scanning electron microscope (SEM), where the powder needs to be coated with gold. The study also used TEM to observe the crystal shape but first the powders have to be dispersed in

absolute alcohol, dropped onto carbon-cover copper grids placed on a filter paper and dried at room temperature.

It was mentioned earlier in the background of study the abundance of cockle shell wastes in Malaysia. Therefore the need to study for existence of calcium carbonate in the cockle shell is done. Base on the study from Mustakimah et al. [5], chemical analysis using XRF conducted on cockle shells showed that it has about the same characteristics as seashells where it main element is Calcium refer to the results below.

Table 1: Chemical analysis of cockle shells by XRF, courtesy of Mustakimah. M, Suzanah. Y & Saikat. M

Oxide	(%wt)
CaO	97.93
MgO	0.85
SiO	0.17
Fe ₂ O ₃	0.04
Others	<1.00

In another study conducted by Othman. H [9], it is shown that the content of calcium carbonate in cockle shells ranges in between 95% - 99% by weight, which could be considered as high and equal to limestone. Consequently in this study it tested the feasibility by using different proportion of cockle shells ashes to be filler material in concrete. The results shown, that the high content of calcium oxide in the cockle shell ash will cause slow hydration process that will reduce the strength of concrete in the early age of curing. The strength of concrete with cockle shell ash mixture is not as strong as normal concrete. Nevertheless, for certain period of time (up to 90) days the percentage of strength increased compared to normal concrete. This can be the benchmark for the strength of cockle shell ash to be in heavy industry including oil and gas.

From a study carried out by M.Nemati [10], the biological formation of calcium carbonate, catalysed by purified urease enzyme or urease produced by a bacterium isolated from a Canadian oil field, were studied in batch systems. In this study, the calcium carbonate efficiency formed by this method in plugging porous media in enhanced oil recovery model was studied in model core systems. The data

obtained plugging by biomass is not permanent and degradation of bacterial cells will eventually lead to increased permeability. However, in this result shown that it is plausible that further study can be conducted if the calcium carbonate used from stronger resources and can hold the plugging in the core thus reduce the permeability. In conclusion, other sources of calcium carbonate made other than dolomite and limestone are used in oil and gas industry.

2.2 Drilling Fluids

Drilling fluids are defined as the fluids that aid the drilling process for oil and gas operation. In the beginning the function of drilling fluids is as the transportation to carry the cuttings from the well bore but now the functions are varies to suit the bore hole conditions. In rotary drilling the functions performed by the drilling mud are as below list ^[2]:

1. Transport cutting from drill bit to the surface
2. Cool and cleanse the drill bit
3. Act as lubricant to reduce friction between the drill string and the formation wall
4. Maintain the stability of formations
5. Prevent flow from the formations to the well bore. i.e.: gas, oil or water
6. Forming thin permeable filter cake that seals crack and other openings in formations
7. Assists in the collection and interpretation of data available from drilling waste, cores and electric logs.

In current trend of well drilled, they have become more challenging and taking longer time to drill due to the distance from the shore and type of reservoir condition that not only needs advance technology in hardware part as well as the drilling fluid to withstand the high pressure and high temperature well.

Nevertheless, environment restriction also can be a challenge for drilling fluid company to produce fluids that are less polluted to the surrounding area. As an example in the North Sea, despite the extreme well condition, the government banned the usage of oil based mud to lower the pollution caused by drilling operation.

However, oil based mud can be recycled and that is why it is used in the close loop circulation in certain rigs.

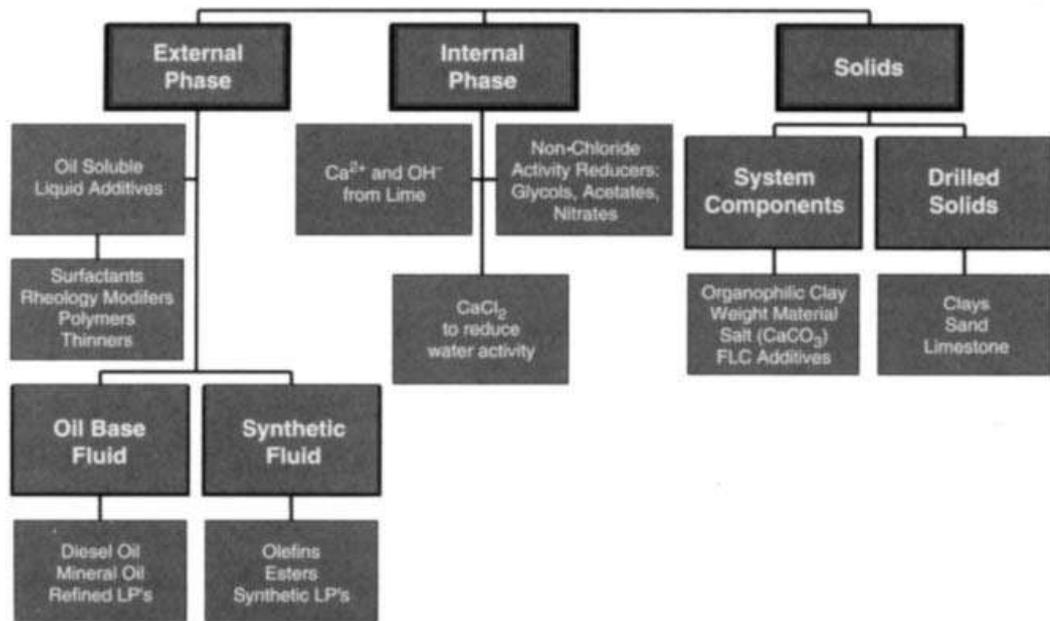


Figure 2: Type of Invert Emulsion Muds courtesy of MI-Swaco

2.2.1 Oil Base Mud

There are three types of drilling fluids compositions, all of them are depending on their bases. Water-based muds, oil-based muds, and gas. For this study it will be focussing on oil-based mud which is used widely in oil and gas companies in Malaysia. However, from environmental point of view water-based mud is better compared to OBM, but in the long term OBM will be more cost effective. Oil-based muds are evolving based on their base oil usage. In the previous years, OBM used diesel and mineral oils as its base. After the advent of the Clean Water Act environmental rules and regulations, these base oils are restricted to be used in offshore operation because of the toxic effect to the marine organisms [2]. As the result, Synthetic Base Mud were introduced which used synthetic oil as the base such as Sarapar 147 and Saraline 185V which are made from paraffin and olefins.

2.2.2 Advantages of Oil Base Mud

Oil based mud could be the most effective alternative for problematic water sensitive shale formation, corrosive gases and water soluble salts. The high cost of oil base mud can be tackled by proper handling and storage of the mud. Following are the significant advantages using oil base mud.

i. **Coring and Completion.**

Oil based mud has its own special characteristic such as low water content, does not hydrate the clays. With the help of special additives, the oil base mud can prevent the liquid phase to escape into the formation as filtration loss. These properties can reduce the damage to the producing formations and reduce the changes of the rock in the core.

ii. **Hole Stability and Corrosion.**

The existing water in the wellbore may cause the slough off of the hole wall formation. Therefore, oil based mud is very important in order to maintain the stability of the wall formation. Combination of corrosive gas with water in water base mud will form acids thus impose hazard to the operation. Without the combination of corrosive gas and water the oil base mud can reduce the corrosion of bottom hole assembly and drill string as well as tubing and casing.

iii. **High Temperature Drilling**

It is known that oil base mud can withstand high temperature condition in drilling operation. In drilling high temperature and high pressure well oil base mud would be the ideal choice with the capability of functioning at temperature more than 400°C and formation pressure more than 20,000 psi (1088 atm).

2.2.3 Component in Oil Base Mud

Oil-based drilling fluids have been developed to overcome certain unwanted characteristic that exists in water-based muds. The defects are mainly because of the water properties, naturally able to dissolve salts, hence, it interfere the flow of oil and gas through porous rocks. In addition, water also promotes the dispersion of clays and affect the corrosion of iron since water and corrosive gas will mixed together to form acids. To counter these deficiencies oil muds offer potential advantages over water-based mud thus the technology is continuously developed including the development of the additives for its component to be used in drilling in such extreme condition well. The table below shows the main components used in making up oil-based mud however it should be remembered that these are the standard examples. If there was a case of selecting specific type of fluid and components in a certain well often a more complex formulation and treatment is needed.

Table of Component and Functions of Oil Based Mud

Component	Functions
Base Oil	Type of solvent, the main component in oil-based mud that dissolves additives to be mixed and become certain mud systems. Previous base oil used are diesel and mineral oil but the industry are switching to synthetic fluid such as olefins and esters.
Primary Emulsifier	It is used to allow oil and water to be mixed and act as a homogenous mixture either in oil-in-water or water-in-oil emulsion. Type of calcium soap made from the reaction of the lime and fatty acids in the mud.
Secondary Emulsifier	Secondary emulsifier is an oil-wetting chemical extracted from wet solid prior to emulsion before the emulsions are formed. Function as to prevent any water intrusion.
Emulsifier Activator	Lime is commonly used to act as the activator for the primary emulsifier to form the calcium soap

	emulsion. Other function of lime is to neutralize the corrosion gases as examples CO ₂ and H ₂ S; hence it is advisable to be added in excess quantity.
Viscosifier	It improves the drilling fluid's ability to remove cuttings from the wellbore and to suspend cuttings and weight materials during periods of no circulation. Clays and natural or synthetic polymers are the materials most commonly used as viscosifiers.
Weighting Agent	Weighting materials (densifiers) are compounds that are dissolved or suspended in drilling fluid to increase its density. They are used to control formation pressures and to help combat the effects of sloughing or heaving shales that maybe countered in stressed areas
Brine	Use to form the water phase in the water-in-oil emulsion. The high salinity water phase helps to prevent shales from hydrating, swelling and sloughing into the wellbore. Most conventional oil-based mud systems are formulated with calcium chloride brine.
Filtration Control Agent	Filtration control agents reduce the amount of filtrate lost from the drilling fluid into a subsurface formation. Organophilic lignite is an amine-treated lignite commonly used for filtration control in oil-base muds and synthetic base muds.
Lost Circulation Materials	It can be defined to include any material that seals or bridges against permeable or fractured formations to inhibit the loss of whole drilling fluid.

2.2.3 Weighting Agent

Weighting agent in the drilling fluids function is to increase the density of the mud to match the formation pressure of the well bore. The reason why it needs to have specific weight is to prevent the formation from collapsing and also disallow the formation liquid to flow into the mud which will cause contamination to the mud. There are many weighting materials that has been used in drilling fluids such as [2]:

1. Barite (most common agent)
2. Ilemenite
3. Calcium carbonate
4. Zinc oxide
5. Manganese tetroxide
6. Hollow glass microspheres (Use for underbalance drilling)

Calcium carbonate has already been used in mud for a while. However the type of calcium carbonate is the commercial grade, which has calcite polymorphs as mentioned earlier. There has been new discovery that listed some of the disadvantages in using barite as the main weighting agent. According to Mohamed et al. [11] they claimed that they found new kind of ilemenite that has an attributes between barite and high-end specialty weighting agents. These weighting agents however are the type that will be needed to drill in an extreme condition. On the contrary, calcium carbonate is focused more towards top section of the well with low pressure and low temperature that does not need high density mud.

Al-Bagoury also mentioned that micronized ilmenite (FeTiO_3) can be introduced as intermediate between barite and the high end specialty weighting agent. It was stated that ilmenite was first used in drilling fluids in late 70's in North Sea area, however its abrasiveness due to the concentration of coarse material and para-magnetic properties, the usage was put on hold. In this paper, it described the new micronized ilmenite, weighting agent, suitable for use in drilling and completion fluids, which offers significant advantages in the control of equivalent circulation density (ECD), sag and formation damage.

Another research by Badrul et al. [12] suggested that dolomite is an alternative weighting agent that can be used efficiently in Malaysia. It was compared to barite which contains toxic materials thus makes it a potential environmental polluter. When barite is used in water-based mud, commonly the mud will be dumped into the sea after drilling. In the study conducted, it focused more on the mud weight that is suitable for the dolomite to produce good mud characteristic in term of rheology, loss filtration and high pressure and high temperature tests compared to barite. The results shown indicate the addition of both barite and dolomite increase the density of the mud, hence both materials could be used for weighting agents. Slurry with barite mixture usually exhibit higher plastic viscosity compared to slurry with dolomite mixture. With this conclusion we can test the viability if using cockle shell calcium carbonate to be mixed as the mud weighting agent and observe how much it affect the mud rheology and mud weight.

2.2.4 Rheology of Drilling Fluids

Drilling fluids have progressed over the years, from simple clay suspensions to highly complex substances both rheological and chemical. Mud is behaving as non-Newtonian fluids because there is no single basic equation to relate between shear stress and shear rate over all ranges of shear rates. Therefore The American Petroleum Institute (API) has set of standards for the rheological determination of drilling fluids [13]. API BUL 13D and a second edition were published in 1985 to comprise everything from basic rheology concept to analysis and acquiring data. Based on Clark [13], drilling muds rheology that is related to range of shear rates are categorised to two categories which are:

Bingham Plastic:

$$\tau = \tau_o + \mu_p + \gamma \dots\dots\dots (2.1)$$

And

Power Law

$$\tau = K\gamma^n \dots\dots\dots (2.2)$$

Where τ is the shear stress, τ_o is the yield stress, μ_p is the plastic viscosity, K is the power law consistency index and n is the power law flow behaviour index. Another perfect model to simulate drilling fluids is Herchel-Bulkley Model

$$\tau = \tau_o + K\dot{\gamma}^n \dots\dots\dots (2.3)$$

To explain the Herchel Bulkley fluid model, when n is assume as one, K will be considered to become plastic viscosity and the model reduces to Bingham plastic model or when assuming $\tau_o = 0$ the model reduce to be power law.

Specifically stated in Baker Hughes Fluid Facts by using Bingham Plastic model is the common model to describe the rheological properties of drilling fluids. Base on API 13A assumed that shear stress is a linear function of shear rate once a specific shear stress has been exceeded.

The reason behind this is because this model is considering data of shear rates of 500 to 1000 sec^{-1} where it is excellent to categorize fluid at higher shear rates. Plastic Viscosity and Yield Point are directly determined from conventional viscometer date taken at 600 and 300 rpm with following equations.

$$PV = \theta_{600} - \theta_{300} \dots\dots\dots (2.4)$$

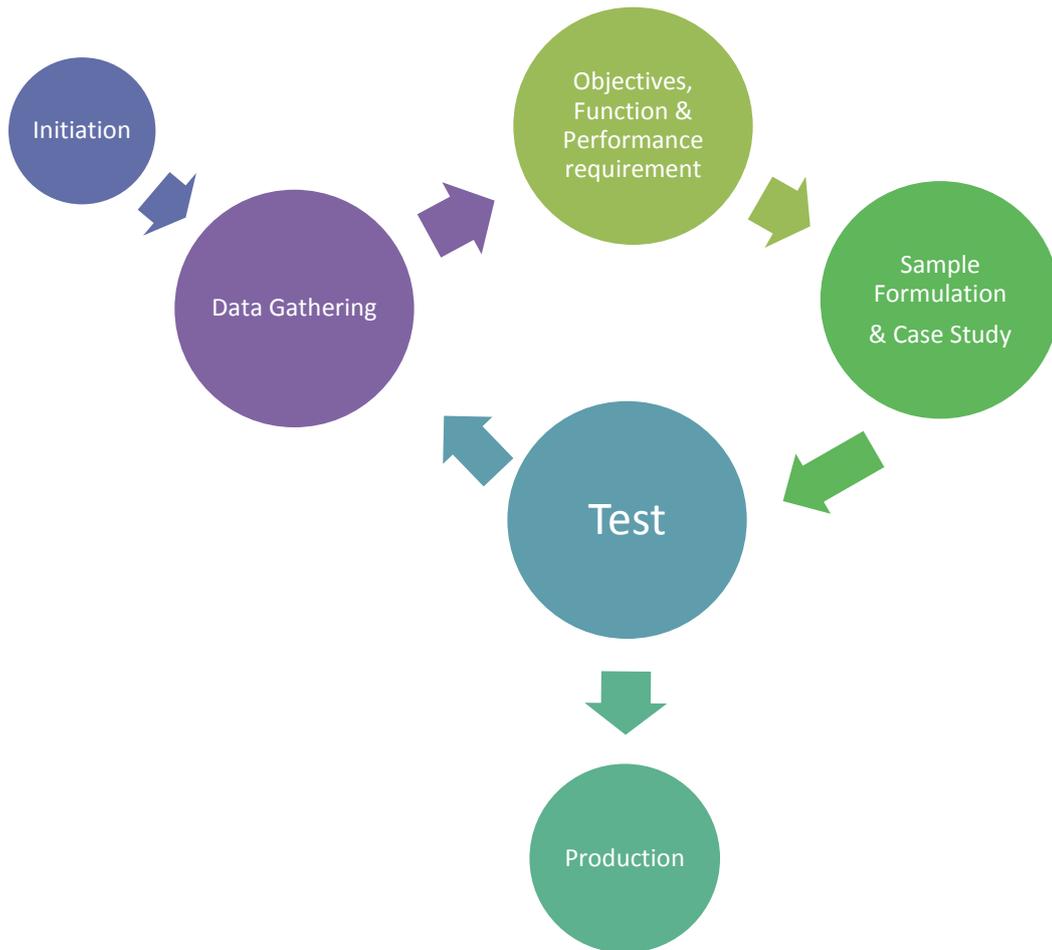
Where:

- PV = Plastic Viscosity
- θ_{600} = 600 rpm dial reading
- θ_{300} = 300 rpm dial reading
- YP = Yield point = $\theta_{300} - PV$

Chapter 3

Methodology

For this study, the author decided to use modified throwaway prototyping model to suit the research methodology.



3.1 Mud Formulation

In this project the mud formulation used will be based on the Confi-Drill mud system from Scomi Oil Tools. The densities of the mud are ranged from 9 ppg to 13 ppg. The formulations are as follow:

Sequence	Materials	Function	Concentration(ppb)
1	Sarapar 147	Solvent	156.31
2	Confi-Mul S	Secondary Emulsifier	9.00
3	Confi-Mul P	Primary Emulsifier	5.00
4	Lime	Activator for Primary Emulsifier	6.00
5	ADAPTA	Filtration Control Agent	2.00
6	CaCl ₂	Brine (Preventing Shale Hydration)	32.19
	Water		91.43
7	Confi-Gel	Viscosifier	3.50
8	Calcium Carbonate	Weighting agent	78.06

Table 2: Formulation for 9 ppg oil base mud formulation

Sequence	Materials	Function	Concentration(ppb)
1	Sarapar 147	Solvent	144.41
2	Confi-Mul S	Secondary Emulsifier	10
3	Confi-Mul P	Primary Emulsifier	6.00
4	Lime	Activator for Primary Emulsifier	6.0
5	ADAPTA	Filtration Control Agent	3.00

6	CaCl ₂	Brine (Preventing Shale Hydration)	24.54
	Water		69.68
7	Confi-Gel	Viscosifier	3.50
8	Calcium Carbonate	Weighting agent	146.38

Table 3: 10 ppg oil base mud formulation

Sequence	Materials	Function	Concentration(ppb)
1	Sarapar 147	Solvent	141.40
2	Confi-Mul S	Secondary Emulsifier	10.00
3	Confi-Mul P	Primary Emulsifier	6.00
4	Lime	Activator for Primary Emulsifier	6.00
5	ADAPTA	Filtration Control Agent	3.0
6	CaCl ₂	Brine (Preventing Shale Hydration)	17.77
	Water		50.47
7	Confi-Gel	Viscosifier	4.5
8	Calcium Carbonate	Weighting agent	213.86

Table 4: 11 ppg oil base mud formulation

Sequence	Materials	Function	Concentration(ppb)
1	Sarapar 147	Solvent	141.40
2	Confi-Mul S	Secondary Emulsifier	10.00
3	Confi-Mul P	Primary Emulsifier	6.00
4	Lime	Activator for Primary Emulsifier	6.00
5	ADAPTA	Filtration Control Agent	3.0
6	CaCl ₂	Brine (Preventing Shale Hydration)	17.77
	Water		50.47
7	Confi-Gel	Viscosifier	4.5
8	Calcium Carbonate	Weighting agent	213.86

Table 5: 13 ppg Oil base mud formulation

3.2 Obtaining Calcium Carbonate from Cockle Shell Procedure

3.2.1 Grinding Calcium Carbonate^[7]

1. 3kg cockle shells was washed and scrub to remove the dirt, boiled for 10 minutes and to be cooled at room temperature after boiled.
2. Thoroughly washed using distilled water and dried in oven for 7 days at temperature of 50oC
3. Cockle shells were grounded by using blender/grinder
4. The powders were sieved using stainless laboratory test sieve with varies apertures to get size variation in micron

5. The powders were dried again in oven for 10 hours to prepare it for grinding process
6. Reduced the powders diameter by grounded the powder using mortar and pestle. The powders were dried again in the oven for 7 days in 50oC temperature in polyethylene plastic bag

Target sizes for Calcium Carbonate.

Range: 50 μ m to 200 μ m.

3.2.2 Determining Terminal Velocity and the Stokes Diameter of the Powder in Base oils and Water

1. Three different type of base oils have been prepared (Sarapar 146, Saraline 185V & Water)
2. The viscosity if each solvent is specified
3. The solvent was poured into a measuring cylinder
4. The powder was poured into the measuring cylinder and the time for the first particle to reach the bottom marks was recorded
5. Terminal velocity was determined and by using Stokes law and using equation in [14] calculate the diameter of the particle



Figure 3: Sieve with various sizes for Grinder



Figure 4: Grinder MF 10 IKA



Figure 5: Oven for drying powder BINDER

3.3 Preparation of Mud Sample

The procedure of preparing mud sample is following the *American Petroleum Institute Standard 13B-2: Recommended Practice for Testing Oil-Based Drilling Fluid*. The procedure needed and their functions are as listed below:

No.	Procedure	Function
1	Mud Formulation (9ppg-13ppg)	Deciding mud system to be used
2	Mud Weight Test	To measure the amount of commercial/cockle shell calcium carbonate achieve the target mud weight
3	Electrical Stability Test	To measure stability of emulsion
6	Viscometer Test	To measure rheology, viscosity, yield point and gel strength
7	Hot Rolling	To simulate downhole and dynamic condition

Table 6: Experimental Procedure and Function

3.3.1 Procedures Preparing Mud Sample

No.	Equipment
1	Fann 9B Multimixer
2	Electronic Balance
3	Stopwatch
4	Thermometer
5	1 lab barrel mud cup

Table 7: Equipment Table

Oil base mud components are mixed using Fann 9B multimixer, the additives needed to be mixed in sequence. The mixing time will be 60 minutes and the volume of the mud produced will be in 350ml. Following are the procedures (for 10 ppg mud):



Figure 6: Fann 9B Multimixer

1. 144.41 grams of Base oil (Sarapar 147) is added in the mud cup.
2. 9.5 grams of secondary emulsifier is added to the cup.
3. 6 grams of primary emulsifier is added into the mixture and stirred for 5 minutes.
4. 6 grams of lime is added into the mixture and stirred for 2 minutes.
5. 3 grams of ADAPTA is added and to be stirred for 5 minutes.
6. Added 24.54 grams of Calcium Chloride brine in to the mixture and stirred for 10 minutes.
7. 4 grams of viscosifier is added and stirred for 5 minutes.
8. 146.38 grams of Calcium Carbonate is added and stirred until the end of the hour.

3.3.2 Mud Weight Test Procedure

Equipment: Mud Balance



Figure 7: Conventional Mud Balance

Procedure:

1. The instrument base is placed on a flat surface
2. The mud temperature was measured
3. The clean was filled, dried the cup and firmly seated. Some of the mud was insured to expel l through the hole in the cup in order to free any trapped air or gas
4. Cap was held firmly on mud cup and cleaned the outside of the cup till dry
5. The beam was placed on the base support and it was balanced by the rider moved along the rider scale. Balance was achieved when the bubble was at the centre of the line
6. Mud weight results were read at the edge of the rider towards the mud cup.

3.3.3 Rheology Test Procedure

Equipment: Fann 35 Viscometer, Stopwatch



Figure 8: Fann 35 Viscometer

Procedure:

1. The sample was stirred at 600 rpm at room temperature
2. The reading was recorded at 600, 300, 200, 100, 6, and 3 rpm speeds. The dial was stabilized before noting the value
3. The sample was stirred at 600 rpm for 30 seconds before taking 10-second gel reading. The motor was stopped for 10 seconds before it was initiated with 3 rpm speed and the highest reading was taken.

4. Mud weight results were read at the edge of the rider towards the mud cup.

3.3.4 Emulsion Stability Test Procedure

Equipment: Electrical Stability Kit



Figure 9: Electrical Stability Kit

Procedures:

1. The probe was placed in the sample and stirred to ensure homogeneity
2. The probe was completely immersed with avoiding it touching the sides of the cup for better results
3. Initiated the voltage ramp and the probe was held still until the end point and steady reading was shown
4. Reading was recorded and repeated 3 times for calculating average value

3.3.5 Hot Rolling Procedures

Equipment: Roller Oven, Aging Cells



Figure 10: Rolling Oven



Figure 11: Aging Cells

Procedures:

1. The oven must be preheated to the set temperature i.e: 120°C
2. The sample is stirred for 5 minutes in multimixer.
3. The, the sample is transferred into the aging cell container.
4. The aging cell is pressurized at 100 psi.
5. The aging cell is then placed in the oven and start rolling the sample for 16 hours.

3.4 Study Plan (Gantt chart)

No.	Milestones /Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
FYP I															
1	Title Proposal	■	■												
2	Consulting Supervisor (UTP) and Industry Supervisor	■	■	■	■	■	■	■	■	■	■	■	■	■	■
3	Acquiring cockle shell sample	■	■	■	■	■	■	■	■	■	■	■	■		
4	Acquiring Additives and Base Oil							■	■	■	■	■	■	■	■
5	Preparation of Cockle Shell's Powder							■	■	■	■	■	■	■	■
4	Documentation											■	■	■	
FYP II															
1	Formulation of Mud sample using Commercial Calcium Carbonate	■	■	■	■	■									
2	Sample Preparation for Commercial Calcium Carbonate	■	■	■	■	■									
3	Commercial Calcium Carbonate Mud Testing			■	■	■									
4	Cockle Shell's Mud Preparation						■	■	■	■					
5	Cockle Shell's Mud Testing						■	■	■	■					
6	Data Gathering	■	■	■	■	■	■	■	■	■	■				
7	Consultation with Supervisor and Industry Supervisor	■	■	■	■	■	■	■	■	■	■	■	■	■	■
8	Documentation											■	■	■	

Chapter 4

Result and Discussion

4.1 Grinded Cockle Shell Powder

Cockle shells have been ground according to the sieve sizes. There 4 sizes 25 μm , 50 μm , 100 μm , 200 μm and 300 μm . The results are as following pictures. It is also compared to commercial Calcium Carbonate side by side in to compare the physical properties.



Figure 12: Left (Cockle Shells Powder) Right (Commercial Calcium Carbonate)

The result shown that after the cockle shells powders are almost the same as commercial calcium carbonate from virtual perspective, however the cockle shells powder is finer compared to commercial as it feels slightly coarse. This might due to some difference in sizes for commercial as it is more varies in sizes.

4.2 Sedimentation Test of Commercial and Cockle Shells Calcium Carbonate Powder

Sedimentation test was conducted to determine the sedimentation rate and terminal velocity in of powders in each solvent i.e: Sarapar 147, Saraline 185V and Water.

This test utilizes gravitational sedimentation method for both powder sample with equivalent sizes of 100 μm .



Figure 13: The powder will be dropped into the measuring cylinder contains solvent and time will be taken for the powder to reach the bottom

Solvent	CaCO ₃ Powder type	Time(s)	Velocity (m/s), 10 ⁻³
Sarapar 147	Cockle Shells	34.00	9.26
	Commercial	22.00	14.3
Saraline 185V	Cockle Shells	27.40	11.4
	Commercial	19.45	16.2
Water	Cockle Shells	13.00	24.2
	Commercial	12.3	25.6

Table 8: Table of Velocity for the Particle to drop to the bottom of measuring cylinder

By using equation 1.7 in ^[14],

$$U_{pt} = \frac{d_t^2(\rho_p - \rho)g}{18\mu} \quad Re_t < 2 \dots\dots\dots(4.1)$$

it can determine the terminal velocity of the powder, but firstly it needed the particle Reynolds number which can be calculated using ^[15]

$$R = \frac{wds}{\nu}$$

Where,

R = Particle Reynold number,

w = Fall velocity

d_s = particle diameter

ν = kinematic viscosity of the fluid

Solvent	CaCO ₃ Powder type	Time (s)	Velocity (m/s), 10 ⁻³	Particle Reynold number, Re _t , 10 ⁻⁷
Sarapar 147	Cockle Shells	34.00	9.26	2.60
	Commercial	22.00	14.3	4.08
Saraline 185V	Cockle Shells	27.40	11.4	3.25
	Commercial	19.45	16.2	4.63
Water	Cockle Shells	13.00	24.2	6.91
	Commercial	12.3	25.6	7.31

Table 9: Particle Reynold Number

Since all the particle Reynold number is < 2, this equation is applied

$$U_{pt} = d_t^2(\rho_p - \rho)g/18\mu$$

Where,

U_{pt} = particle terminal velocity

ρ = density of fluid

ρ_p = density of particle

μ = fluid viscosity

d_t = equivalent dynamic diameter

Cockle shells powder density = 2070 kg/m³

Commercial CaCO₃ density = 2700 kg/m³

Sarapar Density = 760 kg/m³

Saraline Density = 790 kg/m³

Sarapar Dynamic Viscosity = 1.90 cP

Saraline Dynamic Viscosity = 2.77 cP

Water Dynamic Viscosity = 1.0 cP

Table 10: Particle Terminal Velocity

Solvent	CaCO ₃ Powder type	Time(s)	Velocity (m/s), 10 ⁻³	Particle Terminal Velocity, x 10 ⁻⁴ (m/s)
Sarapar 147	Cockle Shells	34.00	9.26	3.76
	Commercial	22.00	14.3	5.56
Saraline 185V	Cockle Shells	27.40	11.4	2.52
	Commercial	19.45	16.2	3.76
Water	Cockle Shells	13.00	24.2	5.83
	Commercial	12.3	25.6	9.27

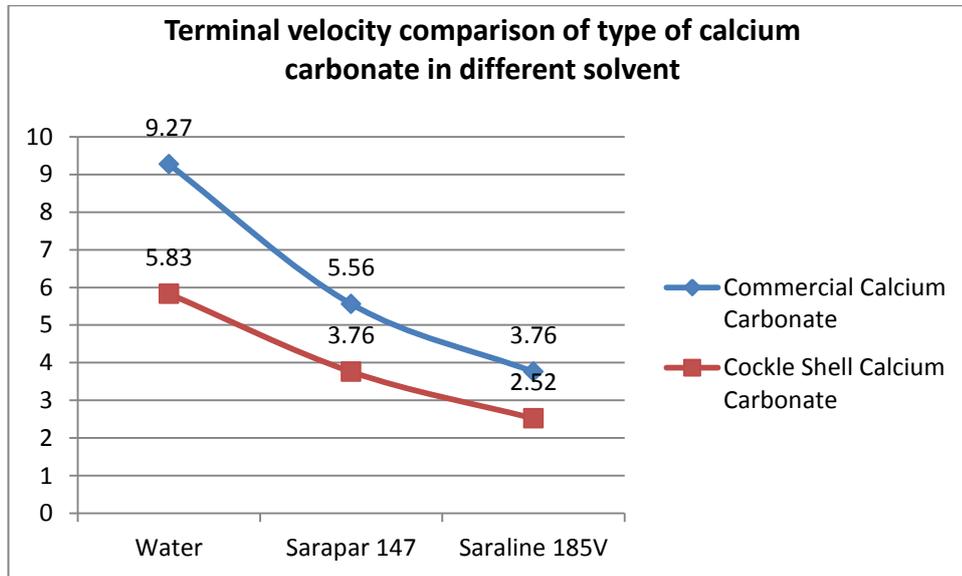


Figure 14: Graph of Terminal Velocity VS Type of Solvent (Commercial CaCO₃)

From the trend of graph we have seen that the dynamic viscosity affect the terminal velocity of the particles. It can be stated that dynamic viscosity is indirectly proportional to the terminal velocity. With the terminal velocity is decreasing for each increment in dynamic viscosity. This also can be proved that the sagging time for the powders in base oil will be shorter especially to Sarapar 147 compared to Saraline 185V as it has much lower terminal velocity.

4.3 Rheological Comparison of Oil Base Mud Using Commercial Calcium Carbonate and Cockle Shell's Calcium Carbonate

The tests were conducted in five different mud weight varying from 9 ppg to 13 ppg mud weight. The size of both calcium carbonates was determined to be as 25 μm. The composition for each mud followed the formulation provided in the methodology. The physical properties of the mud in terms of appearance are shown below, where on the left hand side is the OBM formulated from commercial calcium carbonate, while on the right hand side is the OBM of cockle shell as the weighting agent.

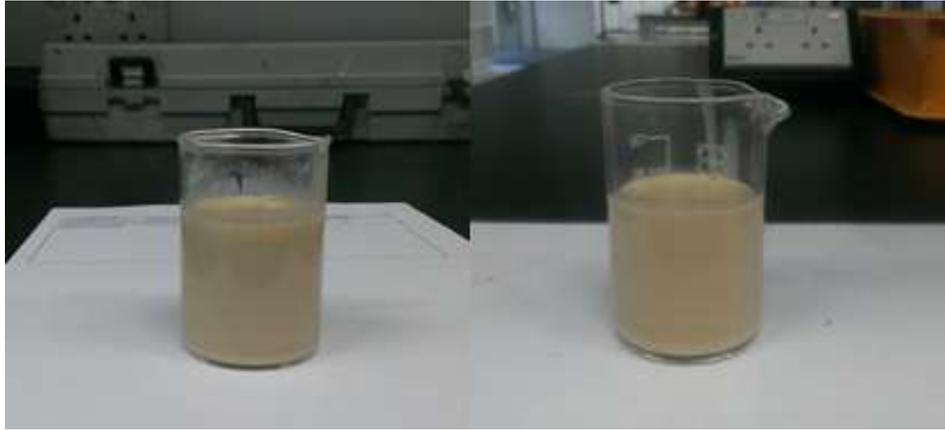


Figure 15: Oil base mud appearance comparison

4.3.1 Rheology of 9 ppg OBM Results

Based on the results shown, the rheology of the commercial and cockle shell's calcium carbonate mud before hot roll are almost the same. However in low RPM commercial calcium carbonate mud shows thick property compared to cockle shells. Both of their rheology properties are in acceptable API Standard.

However, after hot roll the both of the mud showed thinning properties as the rheology properties were decreasing after hot rolling. This property shown might be because of base oil that trapped the emulsion partially been released and cause the mud to be thinner. In this case adding more emulsion can be the solution to the problem to increase the strength of emulsion stability after hot rolling.

On the other hand, in figure 19 rheology properties of cockle shell's mud before and after hot roll are compared. The properties do not fluctuate very much, giving some flat rheology properties which are good for the mud. Especially at the low RPM rheology, the mud properties do not change. This may be because the emulsion that released is minimal and does not affect the rheology in low RPM.

Confi-Drill
 9 ppg
 OWR = 70/30
 Commercial Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Accepted Range
600 RPM	45	37	
300 RPM	25	21	
200 RPM	18	16	
100 RPM	11	9	
6 RPM	5	3	
3 RPM	3	2	
Gels (10s/10min)	3/5	3/4	
PV	20	16	15-30
YP	5	5	5-10
	BHR	AHR	
E.S, volt	288	211	200-300
API LP/LT, (ml)		0	
API LP/LT Mud cake (inch)		1/32	

Table 11: Commercial Calcium Carbonate 9ppg OBM

Confi-Drill
 9 ppg
 OWR = 70/30
 Cockle Shell Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Accepted Range
600 RPM	45	44	
300 RPM	25	24	
200 RPM	18	16	
100 RPM	10	9	
6 RPM	4	4	
3 RPM	2	2	
Gels (10s/10min)	2/3	2/3	
PV	20	20	15-30
YP	5	4	5-10
	BHR	AHR	
E.S, volt	313	280	200-300
API LP/LT, (ml)		0	
API LP/LT Mud cake (inch)		1/32	

Table 12: Cockle Shell's Calcium Carbonate 9 ppg OBM

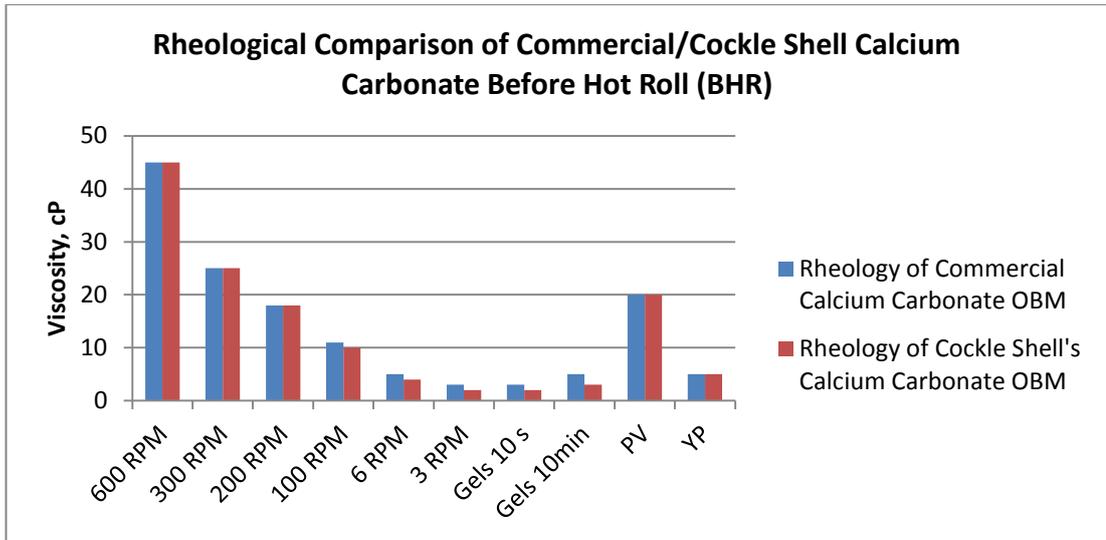


Figure 16: Rheological Comparison Graph of Commercial & Cockle Shell OBM BHR 9 ppg

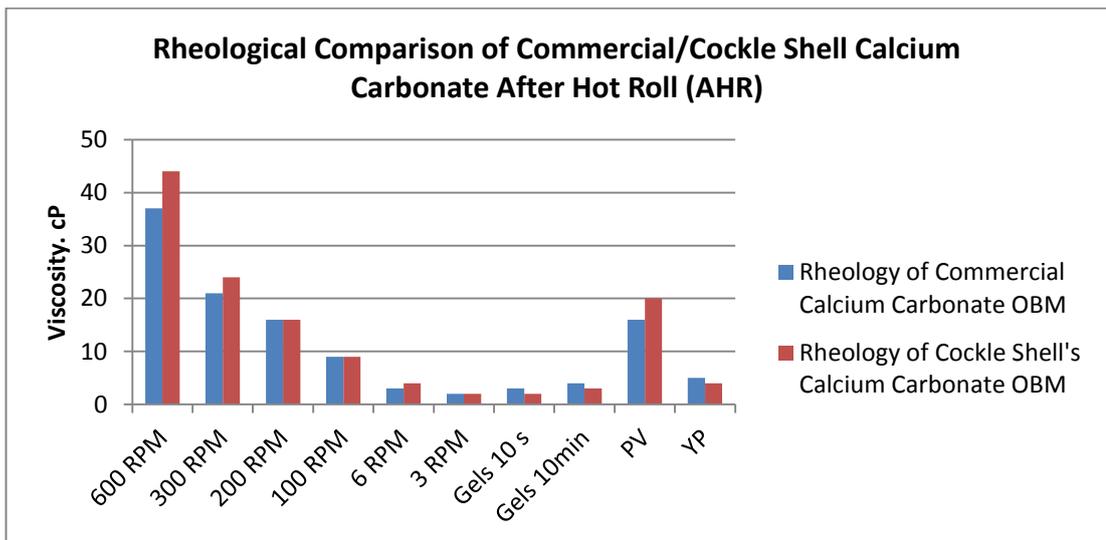


Figure 17: Rheological Comparison Graph of Commercial & Cockle Shell OBM AHR 9 ppg

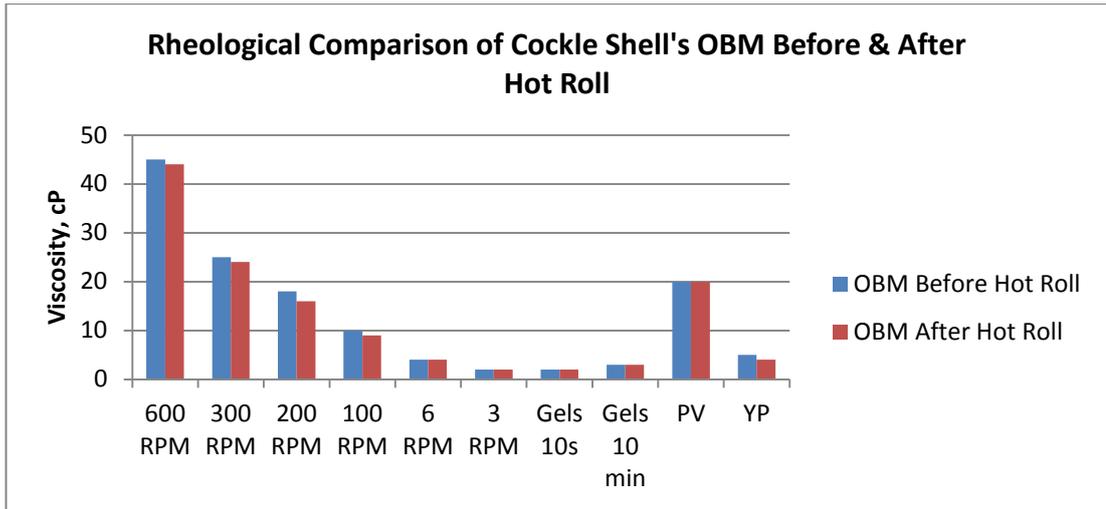


Figure 18: Rheological Comparison Graph of Cockle Shell's OBM before & after Hot Roll 9 ppg

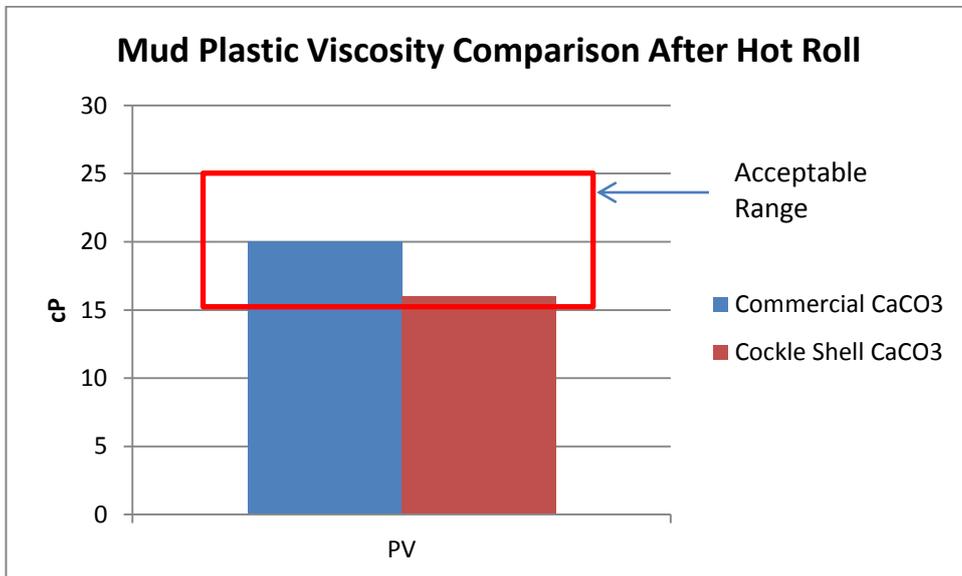


Figure 19: Plastic viscosity comparison after hot roll

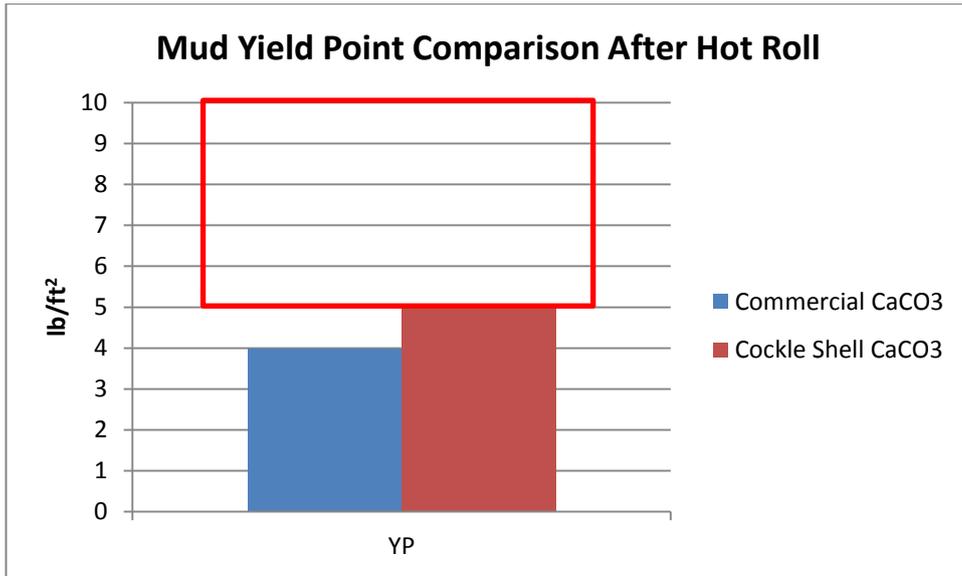


Figure 20: Yield point comparison after hot roll

4.3.2 Rheology of 10 ppg OBM Results

On Figure 20, the bar graph shows the cockle shell's mud exhibit higher rheology properties in small margin compared to commercial mud before hot roll. After hot rolling the muds, the margin increased accept for 10 min gels it gives almost same rheology value which is at 6 and 5, respectively.

For electrical stability, referring to the table 13 and 14 before hot rolling both of the mud exceed the electrical stability acceptable range. As the oil and emulsion are still intact to secure the brine therefore the stability is high. Nevertheless, it is a good property for the mud to have high E.S to ensure the emulsion is stable. After hot rolling the E.S drop significantly for commercial calcium carbonate from 520 to 380, and for cockle shell's calcium carbonate is from 500 to 211. The drop is caused by the brine that escapes from emulsion thus effect the electrical stability of the mud.

Referring to figure 21, cockle shell's oil base mud shows thickening property as the rheology properties increase slightly. This is maybe the cause of brine that has been released from inside of the emulsion causing the water to dissolve in the mud and cause mud thickening. The brine expelled might be in small amount thus cause the slight rise.

Confi-Drill

10 ppg

OWR = 75/25

Cockle Shell Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Spec. Range
600 RPM	69	71	
300 RPM	38	38	
200 RPM	28	26	
100 RPM	16	14	
6 RPM	5	3	
3 RPM	2	1	
Gels 10s/10min	3/5	2/4	
PV	31	33	20-40
YP	7	5	6-14
	BHR	AHR	
E.S volt	520	380	200-300
API LP/LT, (ml)		0	
API LP/LT Mud cake (inch)		1/32	

Table 13: Commercial Calcium Carbonate 10 ppg OBM

Confi-Drill

10 ppg

OWR = 75/325

Commercial Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Spec. Range
600 RPM	73	78	
300 RPM	41	42	
200 RPM	30	30	
100 RPM	17	12	
6 RPM	5	6	
3 RPM	3	3	
Gels 10s/10min	3/6	3/4	
PV	32	36	20-40
YP	9	6	6-14
	BHR	AHR	
E.S volt	500	211	200-300
API LP/LT, (ml)		0	
API LP/LT Mud cake (inch)		1/32	

Table 14: Cockle Shell's Calcium Carbonate 10 ppg OBM

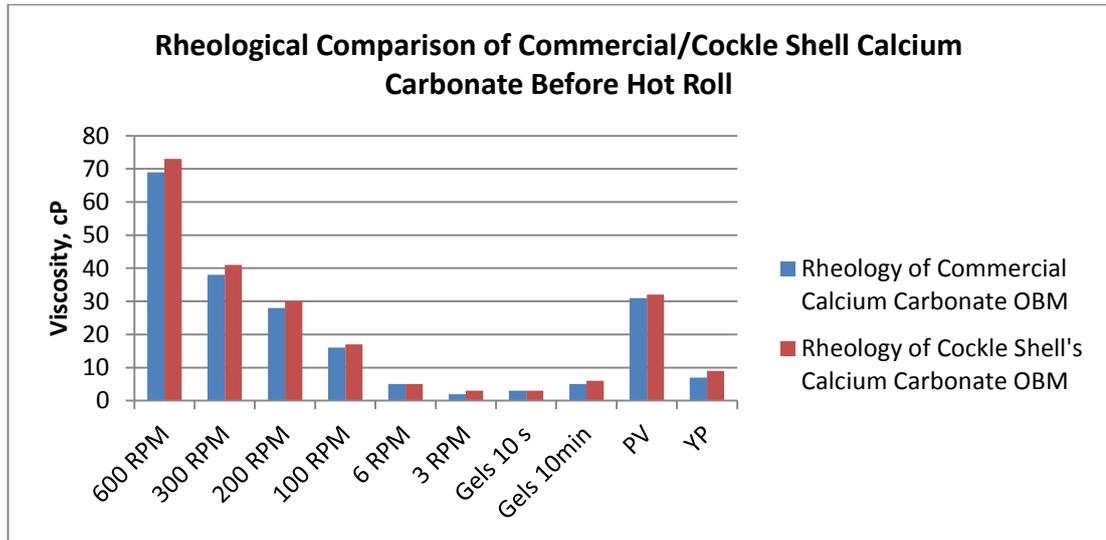


Figure 21: Rheological Comparison Graph of Commercial & Cockle Shell OBM BHR 10 ppg

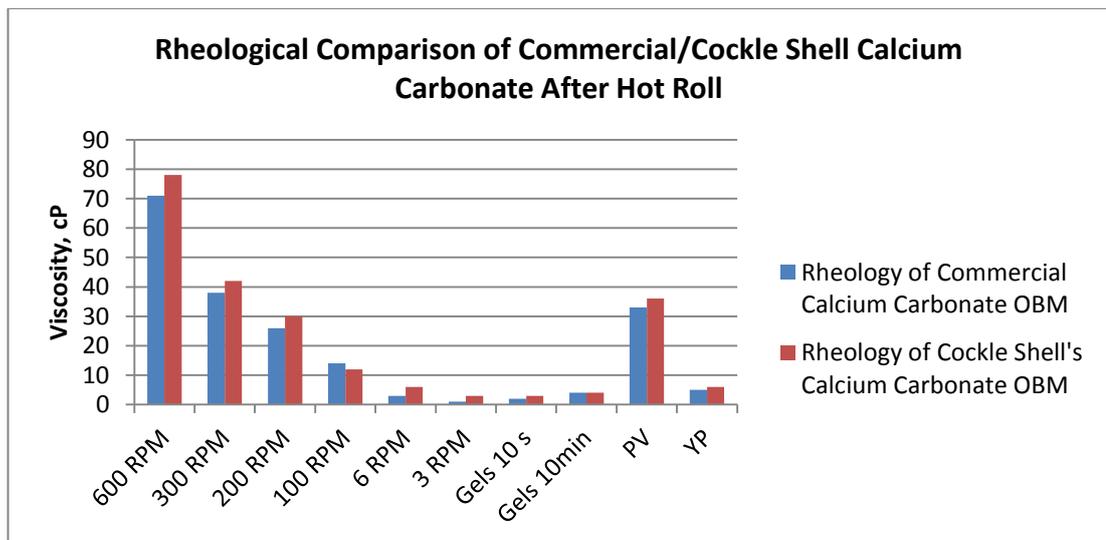


Figure 22: Rheological Comparison Graph of Commercial & Cockle Shell OBM AHR 10 ppg

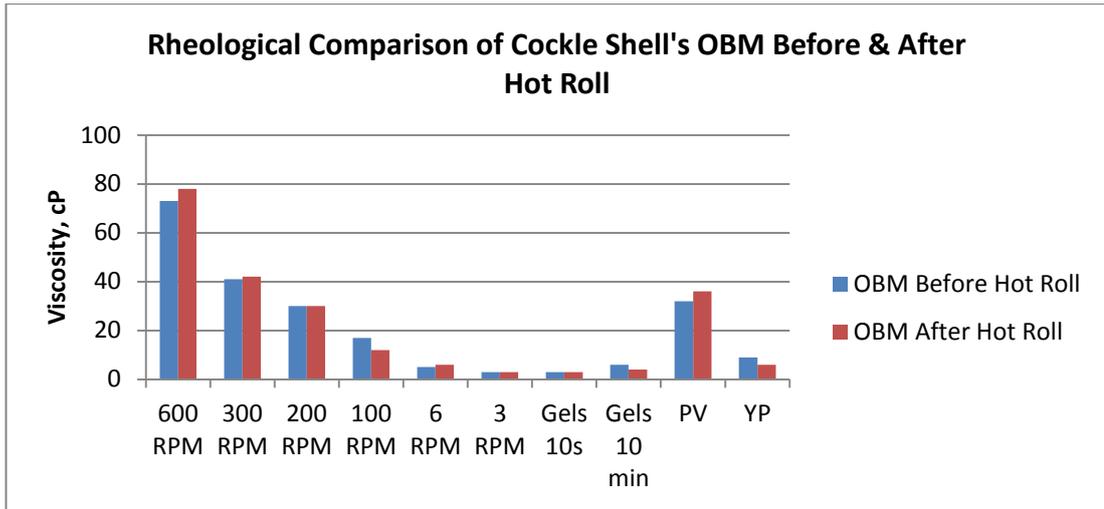


Figure 23: Rheological Comparison Graph of Cockle Shell's OBM before & after Hot Roll 10 ppg

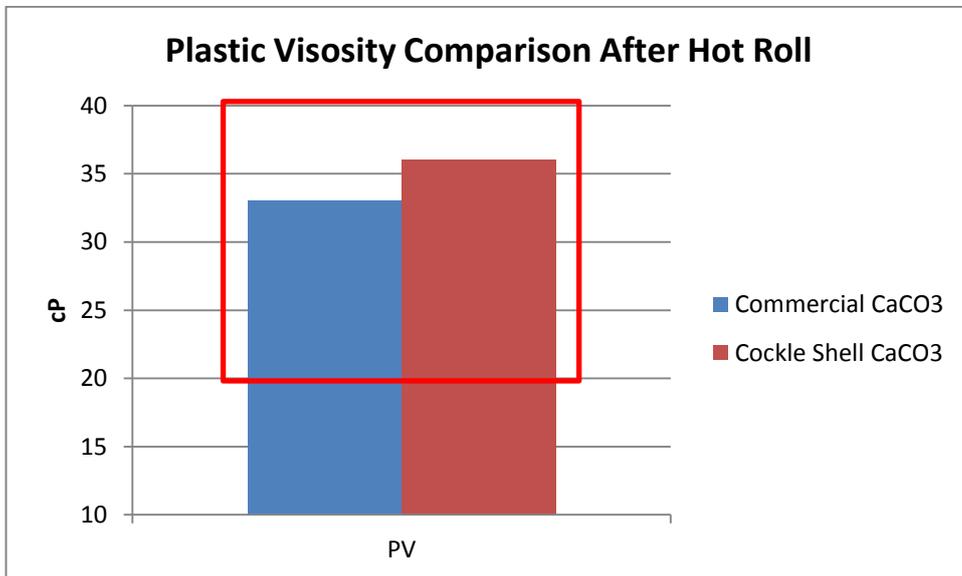


Figure 24: 10 ppg plastic viscosity comparison after hot roll

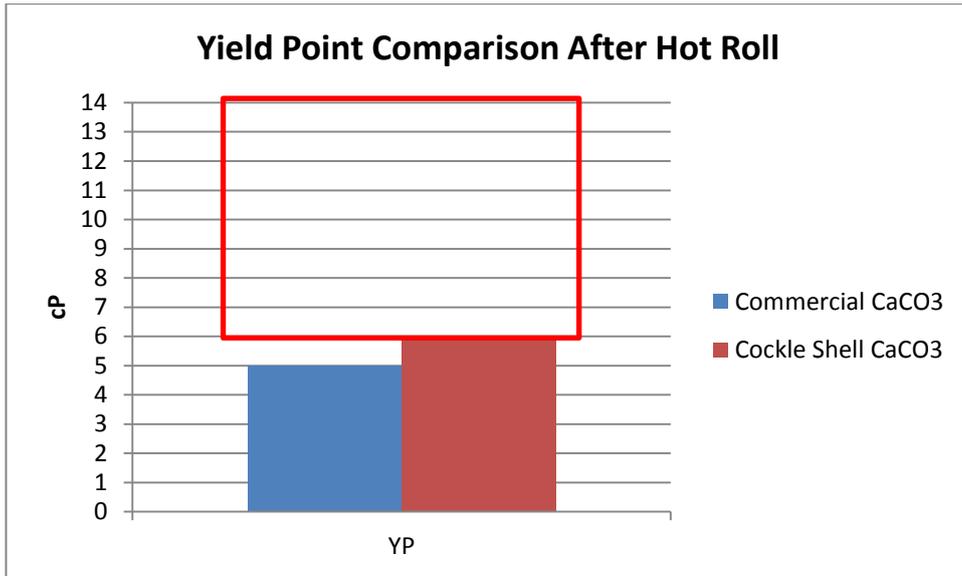


Figure 25: 10 ppg yield point comparison after hot roll

4.3.3 Rheology of 11 ppg OBM Results

In table 15 and 16, both of the muds electric stability has passed the accepted values. This show the emulsions strength can withstand the simulated bottom hole condition in the hot roll oven. The importance of having high electric stability thus it will not produce high disturbance while measurement on the formation is on-going.

Commercial and Cockle Shell's mud rheology properties did not have significant value differences before hot roll and after hot roll, only slight increase on the cockle shell's mud rheology. Since cockle shell element does not only have calcium carbonate, therefore it is believed that the small percentage of the other elements might as well affect the rheology of the mud. However, further study needed to be conducted to prove this hypothesis.

Cockle shell's OBM comparison before and after hot roll in figure 25, shows that the mud is thickening after simulated in the bottom hole condition. The reason is the same as before as some of the brine might have released out of the invert emulsion and thus contaminate the mud.

Confi-Drill

11 ppg

OWR = 80/20

Cockle Shell Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Spec. Range
600 RPM	80	90	
300 RPM	44	51	
200 RPM	36	44	
100 RPM	23	28	
6 RPM	11	7	
3 RPM	5	6	
Gels (10s/10min)	7/12	8/12	
PV	36	39	20-40
YP	8	12	6-14
	BHR	AHR	
E.S volt	811	589	300-400
API LP/LT, (ml)		0	
API LP/LT Mud cake (inch)		1/32	

Table 15: Commercial Calcium Carbonate 11 ppg OBM

Confi-Drill

11 ppg

OWR = 80/20

Commercial Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Spec. Range
600 RPM	87	100	
300 RPM	49	57	
200 RPM	39	47	
100 RPM	25	30	
6 RPM	8	9	
3 RPM	6	7	
Gels (10s/10min)	7/12	8/12	
PV	38	43	20-40
YP	11	14	6-14
	BHR	AHR	
E.S volt	1015	487	300-400
API LP/LT, (ml)		0	

API LP/LT Mud cake (inch)		1/32	
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Table 16: Cockle Shell's Calcium Carbonate 11 ppg OBM

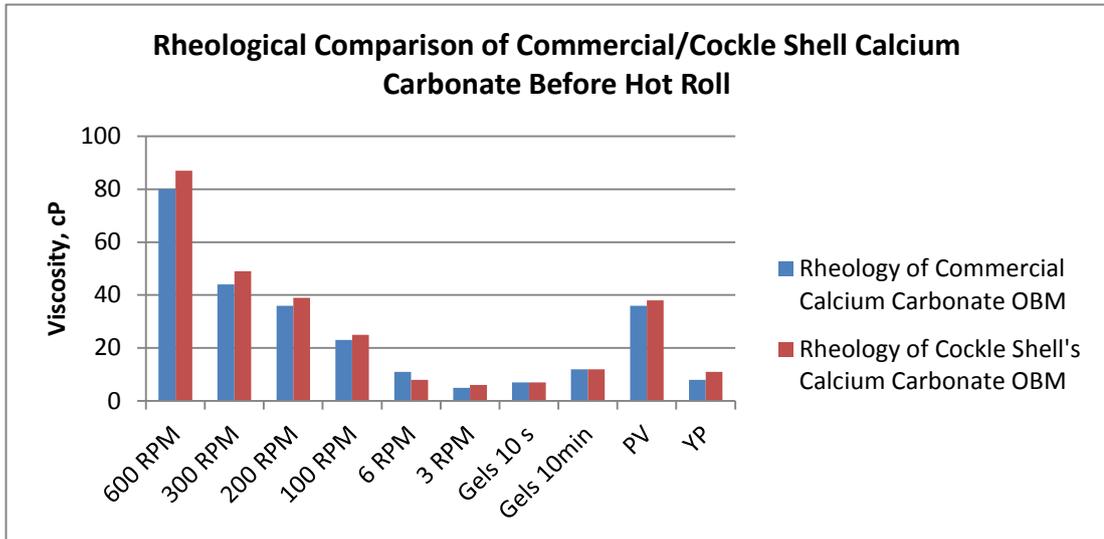


Figure 26: Rheological Comparison Graph of Commercial & Cockle Shell OBM BHR 11 ppg

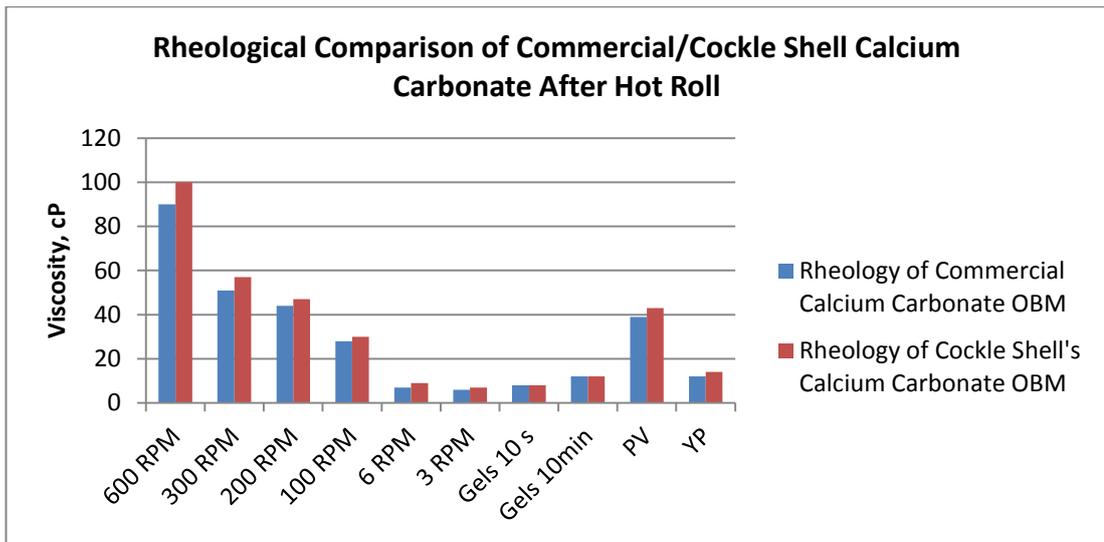


Figure 27: Rheological Comparison Graph of Commercial & Cockle Shell OBM AHR 11 ppg

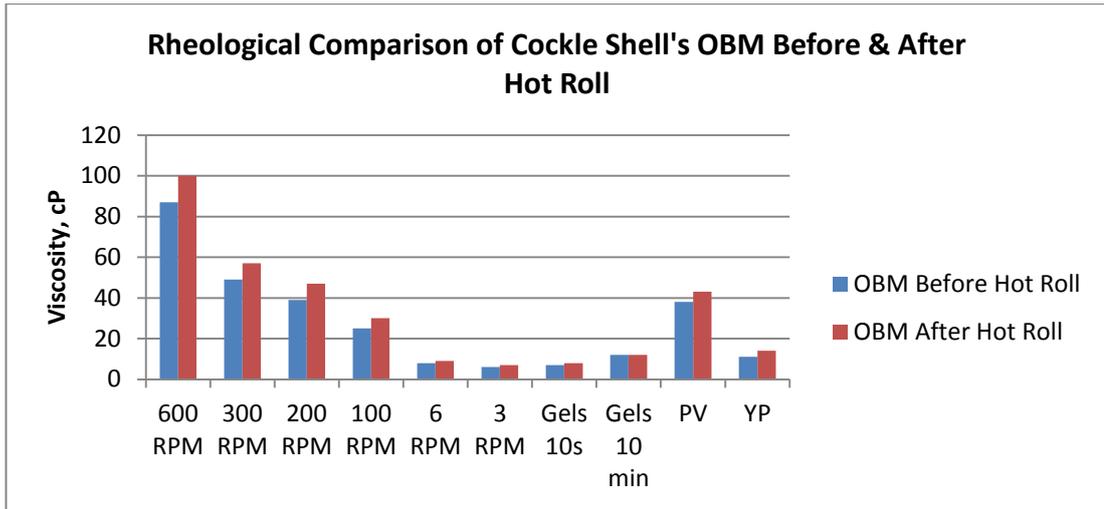


Figure 28: Rheological Comparison Graph of Cockle Shell's OBM Before & After Hot Roll 11 ppg

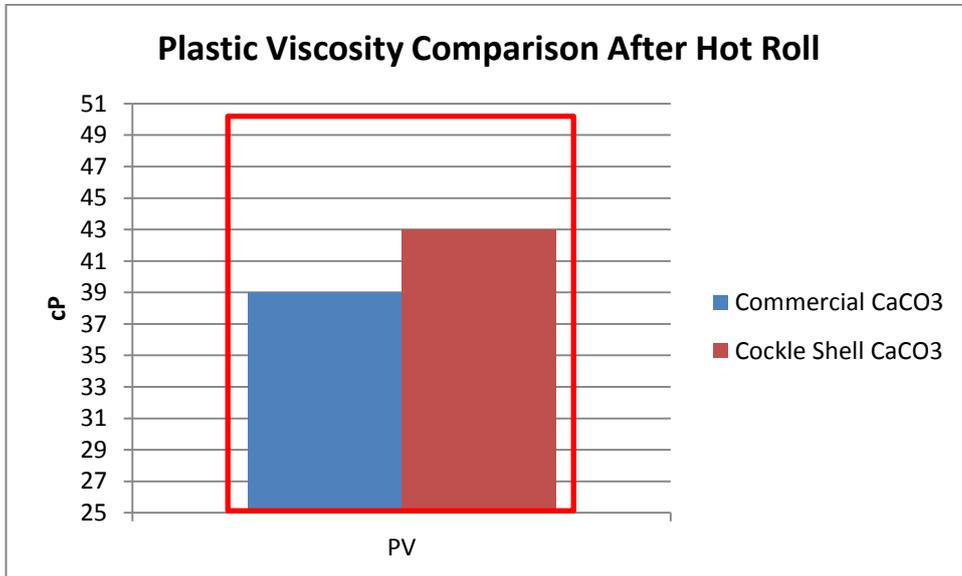


Figure 29: 11 ppg plastic viscosity comparisons after hot roll

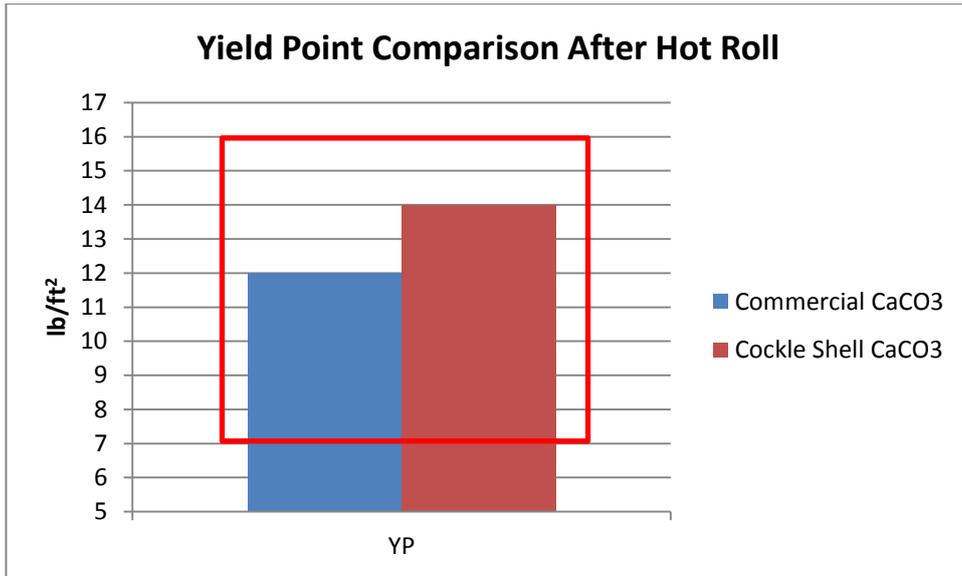


Figure 30: 11 ppg Yield Point comparisons after hot roll

4.3.4 Rheology of 12 ppg OBM Results

According to the table 17 and 18, both muds show high PV and YP value which exceed the acceptable range. The addition of calcium carbonate in both muds causes the mud being too thick and to solve this problem, the muds need to be treated with the thinning agent to normalise the rheology.

For barite that has a density of 4.2 SG it need less amount of powder to increase the weight of the mud, whereas calcium carbonate with 2.7 SG the amount of powder needed is massive and causing the muds concentrated with calcium carbonate thus having high rheological property.

Despite of the high rheology property, the electrical show stability as it exceed the acceptable range for both mud and proven it is possible for the cockle shell to be used in the drilling fluid system.

Confi-Drill

12 ppg

OWR = 80/20

Cockle Shell Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Spec. Range
600 RPM	148	156	
300 RPM	88	95	
200 RPM	65	72	
100 RPM	41	47	
6 RPM	11	14	
3 RPM	9	13	
Gels (10s/10min)	13/18	15/17	
PV	60	61	25-50
YP	28	34	7-16
	BHR	AHR	
E.S volt	1156	874	300-400
API LP/LT, (ml)		0	
API LP/LT Mud cake (inch)		1/32	

Table 17: Commercial Calcium Carbonate 12 ppg OBM

Confi-Drill

12 ppg

OWR = 80/20

Commercial Calcium Carbonate

Rheology Properties at 27°C	BHR, cP	AHR, cP	Spec. Range
600 RPM	180	189	
300 RPM	112	119	
200 RPM	83	89	
100 RPM	53	59	
6 RPM	17	20	
3 RPM	15	19	
Gels (10s/10min)	15/17	17/19	
PV	68	70	25-50
YP	44	49	7-16
	BHR	AHR	
E.S volt	975	748	300-400
API LP/LT, (ml)		0	

API LP/LT Mud cake (inch)		1/32	
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Table 18: Cockle Shell's Calcium Carbonate 12 ppg OBM

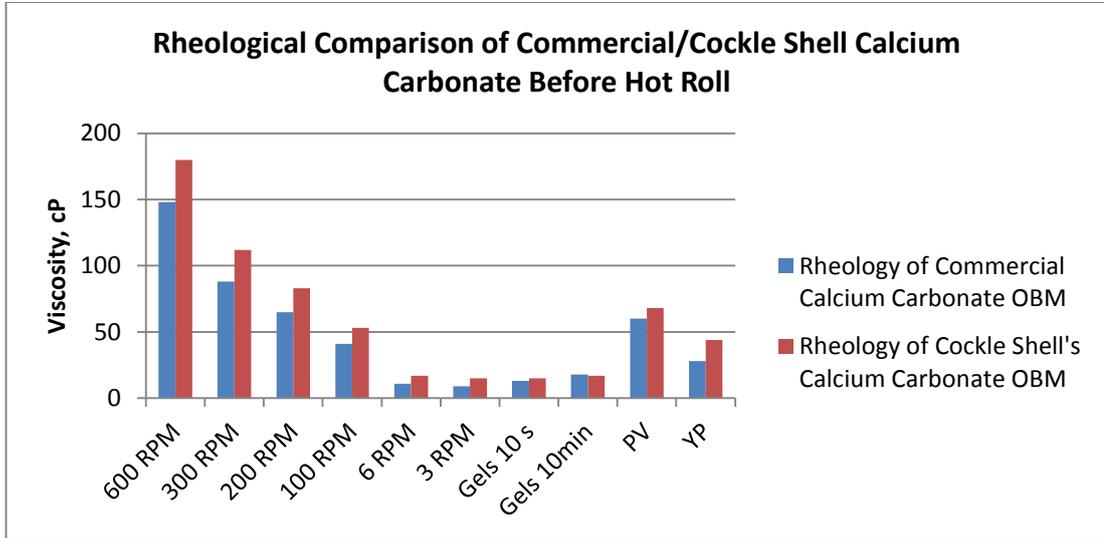


Figure 31: Rheological Comparison Graph of Commercial & Cockle Shell OBM BHR 12 ppg

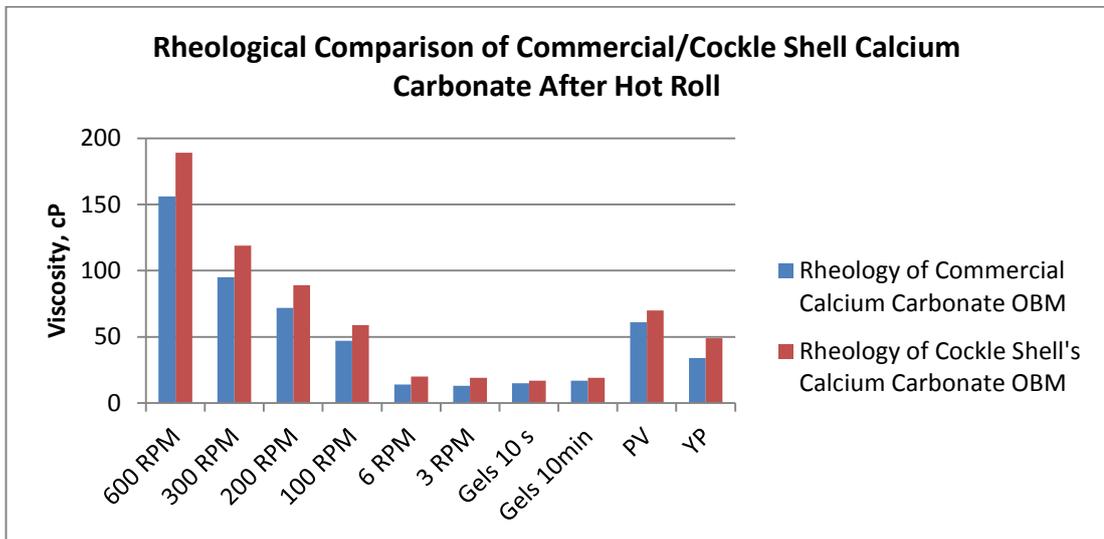


Figure 32: Rheological Comparison Graph of Commercial & Cockle Shell OBM AHR 12 ppg

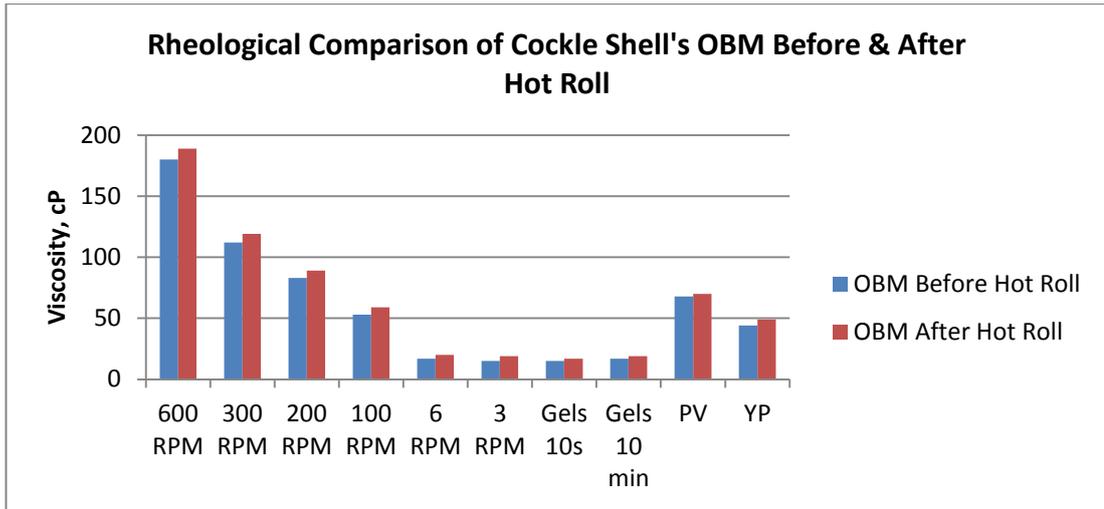


Figure 33: Rheological Comparison Graph of Cockle Shell's OBM before & after Hot Roll 12 ppg

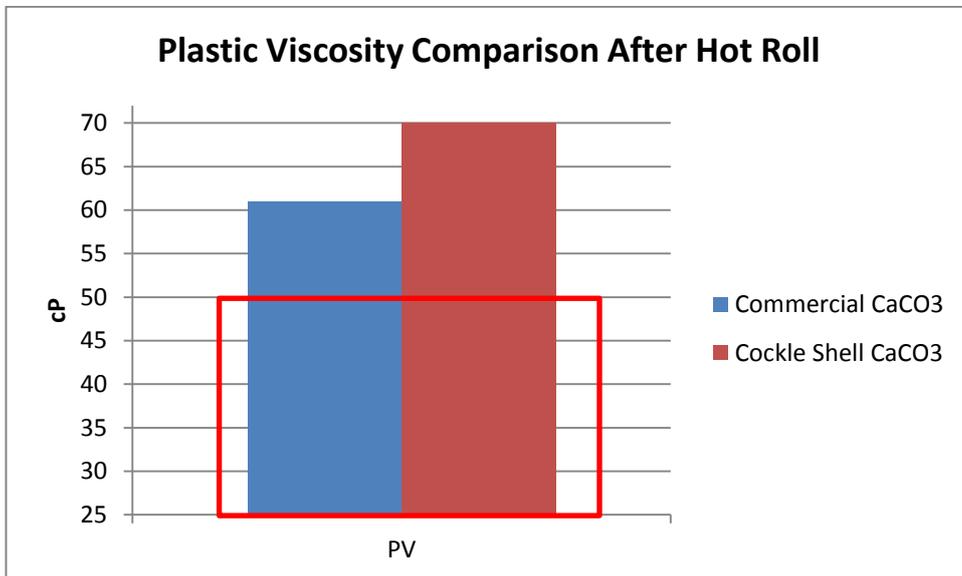


Figure 34: 12 ppg plastic viscosity comparisons after hot roll

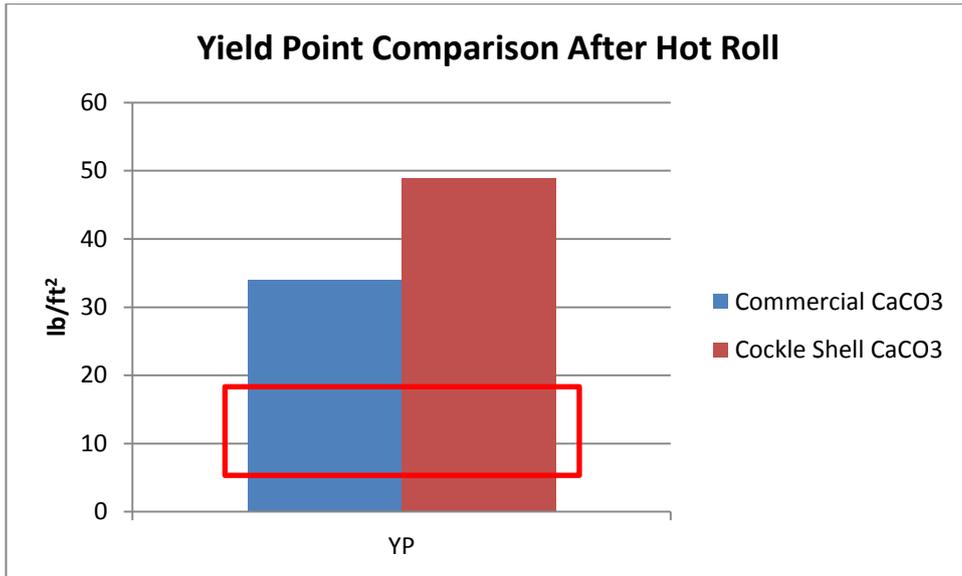


Figure 35: 12 ppg yield point comparison after hot roll

4.3.5 Rheology of 13 ppg OBM Results

Going up to 13 ppg mud, the data for 600 rpm is no longer available since the mud is too thick and the viscometer failed to read the rheology of the muds. Hence, with this limitation, the properties for PV and YP could not be obtained.

This problem can be addressed by adding thinning agent in the mud in order the rheology to be acceptable. It cannot be treated by using more solvent, since the mud weight will be lessened due to the excess of solvent in the mud system.

Confi-Drill

13 ppg

OWR = 80/20

Cockle Shell Calcium Carbonate

Rheology Properties at 81°F	BHR, cP	AHR, cP	Spec. Range
600 RPM	298	N/A	
300 RPM	178	188	
200 RPM	130	138	
100 RPM	80	87	
6 RPM	22	29	
3 RPM	19	27	
Gels (10s/10min)	19/32	21/34	
PV	120	N/A	25-50
YP	58	N/A	7-16
	BHR	AHR	
E.S volt	1351	1071	300-400
API LP/LT, (ml)		0	
API LP/LT Mud cake (inch)		1/32	

Table 19: Commercial Calcium Carbonate 13 ppg OBM

Confi-Drill

13 ppg

OWR = 80/20

Commercial Calcium Carbonate

Rheology Properties at 81°F	BHR, cP	AHR, cP	Spec. Range
600 RPM	N/A	N/A	
300 RPM	201	213	
200 RPM	149	160	
100 RPM	92	101	
6 RPM	28	34	
3 RPM	25	31	
Gels (10s/10min)	20/33	24/39	
PV	N/A	N/A	25-50
YP	N/A	N/A	7-16
	BHR	AHR	
E.S volt	1214	994	300-400
API LP/LT, (ml)		0	

API LP/LT Mud cake (inch)		1/32	
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Table 20: Cockle Shell's Calcium Carbonate 12 ppg OBM

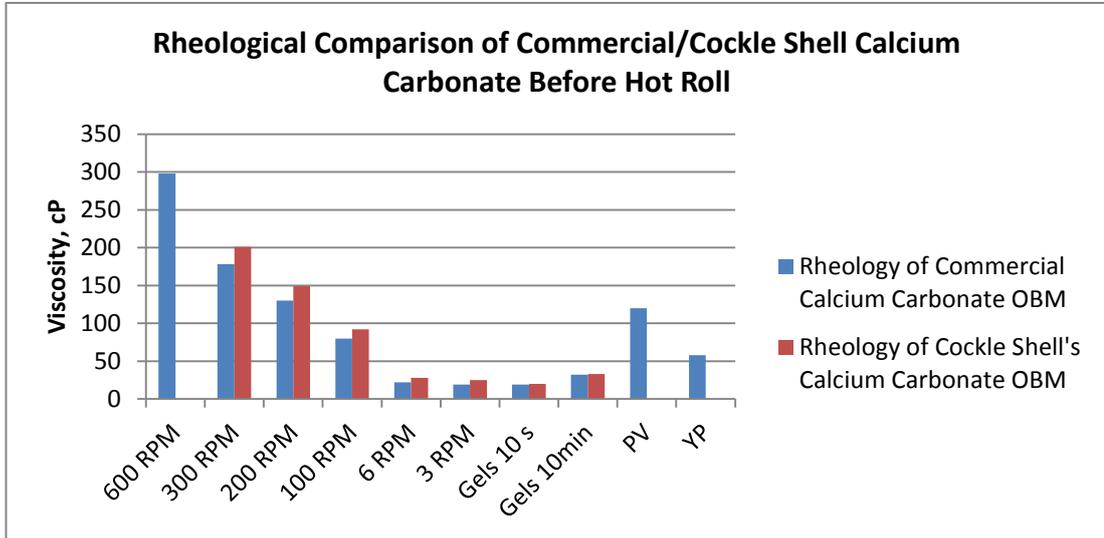


Figure 36: Rheological Comparison Graph of Commercial & Cockle Shell OBM BHR 13 ppg

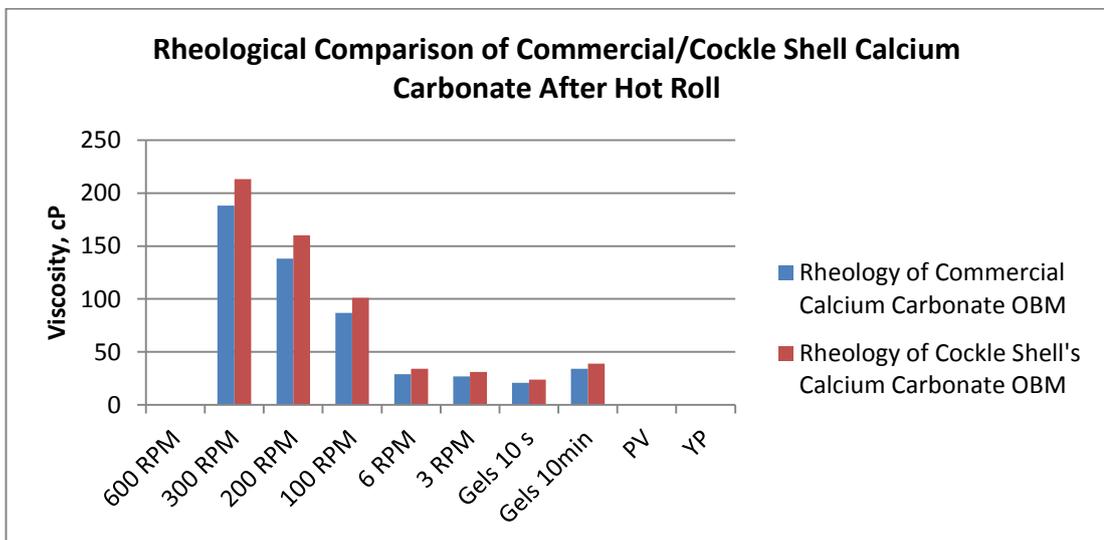


Figure 37: Rheological Comparison Graph of Commercial & Cockle Shell OBM AHR 13 ppg

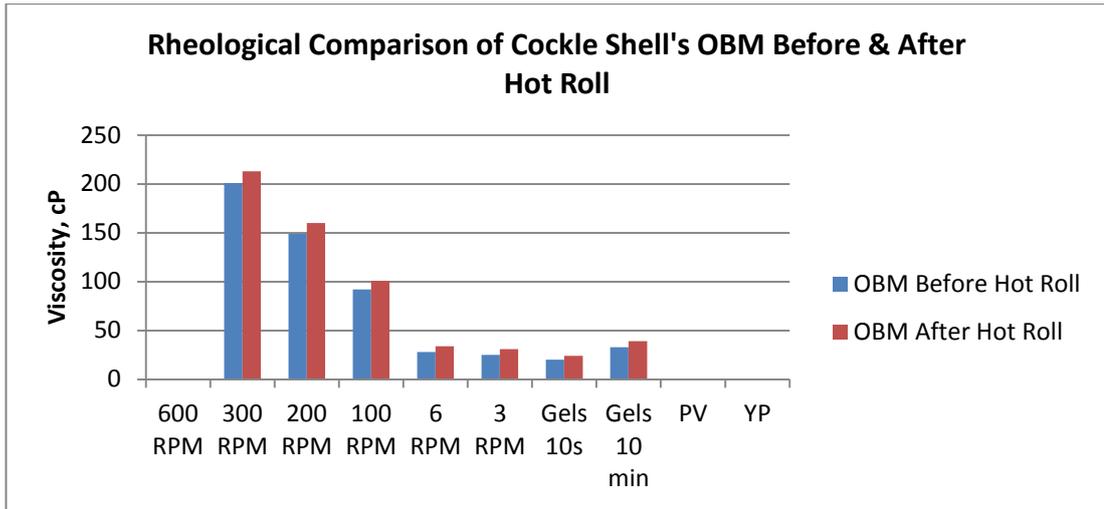


Figure 38: Rheological Comparison Graph of Cockle Shell's OBM before & after Hot Roll 13 ppg

4.4 Discussion

4.4.1 Rheology

There are some key values for treating and maintaining drilling fluids, below are the properties that describe drilling fluid viscosity and rheological properties[16]:

- Funnel viscosity (sec/qt)
- Effective viscosity (cP or mPa•sec)
- Apparent viscosity (cP or mPa•sec)
- Plastic viscosity (cP or mPa•sec)
- Yield Point (lb/100 ft² or Pa)
- Gel Strengths (lb/100 ft² or Pa)

In industry specification, among the properties that are commonly focussed to be used are plastic viscosity, yield Point and gels strength.

Plastic viscosity (PV) is in centipoise or milipascal seconds is calculated from mud viscometer data as $PV (cP) = \theta_{600} - \theta_{300}$. In the results above we see that in overall mud from cockle shell calcium carbonate exhibit higher rheology properties compared to commercial calcium carbonate. Plastic viscosity is usually known as the resistance to flow by mechanical friction. Often affected by,

- Solids concentration

- Size and shape of solids
- Viscosity of the fluid phase
- The oil to water ratio
- Type of emulsifiers in invert emulsion fluids

With the affected reason above since in the literature review explained the difference morphology of cockle shell calcium carbonate and commercial calcium carbonate are aragonite and calcite respectively, it may have affect the rheological properties of the mud system.

Moreover as the result shown for 13 ppg mud the solids concentration was too much and cause the rheology reading is not possible to be read in the Fann viscometer.

A yield point unit is in pounds per 100 square feet (lb/100 ft²) it can be calculated from VG meter as follows:

$$YP = \theta_{300} - PV$$

From [16] Yield point measurement is depend to:

- The properties and surface of solids that exist in the fluid.
- The solids concentration
- Electrical stability of these solids

Therefore, with the point number 1 stated, we can assume that the properties and surface of calcium carbonate from cockle shell will alter the YP of the mud.

4.4.2 Electrical Stability

Electrical stability indicated the water phase in the mud whether it is well emulsified or not according to [16]. Mud with higher value will be resulting in strong emulsion and more stable fluids. Since oil and synthetic oil do not allow the electricity flow through them hence with the oil wetting condition help to block the current flow inside the mud.

Followings are the justification for the stability difference between commercial calcium carbonate and cockle shell calcium carbonate after hot rolling.

Water-wet Solids

The results shown that the electrical stability for the calcium carbonate from cockle shell mud decreases significantly after hot rolling. This is because by the water wet solids which the emulsion did not provide the oil wetting condition on the solids especially cockle shells calcium carbonate. Therefore with the lack of oil wetting condition, the solids are behaving like water-wet solids thus affecting the electrical stability.

Emulsion Strength

According to [16], new mud system water droplets are bigger and weakly emulsified. Hence, the emulsion stability is low for the new mud system using cockle shell calcium carbonate. Even though, the electric stability before hot roll showed good reading but the emulsion strength might be compromised with the high temperature in hot rolling oven. The heat that was affecting the emulsion strength during hot rolling can be countered by increasing the emulsion concentration. Lastly, it is important to conduct further research to find the suitable emulsion that will react correctly with calcium carbonate from cockle shell to produce oil wet condition.

Chapter 5

Conclusion & Recommendation

5.1 Conclusion

As the conclusion for this extended proposal, author strongly support for this study to be continued as it has potential on making use the cockle shell waste as the raw material for drilling fluids as weighting agent. In additional to that, it will preserve the natural resources for Calcium Carbonate which is limestone, dolomite and chalk. Even though they are naturally renewable but it takes time to reform and ready to be used commercially.

Earlier in the literature reviews mention, technically the potential for the Calcium Carbonate harvested from cockle shells is there as the molecular structure between the commercial and cockle shell calcium carbonate are only the difference while both of them have almost identical physical properties. Moreover, the studies shown that cockle shells actually have high concentrations of Calcium Carbonate thus make it viable to be one of the raw material sources.

Base on the results produced so far, the possibility of using cockle shell in the drilling fluid is highly possible with the prove of the rheology properties comparison between commercial calcium carbonate and cockle shell's calcium carbonate. The rheology properties shown that both of them have only slight difference which cockle shell's mud shows slightly high values in term of rheology. Despite of the slight increase, it still manage to stay between the acceptable ranges.

5.2 Recommendation

For recommendation, these studies on sagging time for the powders need to be done with other chemicals that will affect the sagging time for the powders. Therefore, it needs further evaluation with full mud check and also to be evaluated in simulation with real condition compared to room temperature.

Further study needed to be conducted, with the presence of the High Pressure and High Temperature Test as the test is important on determining oil base mud filter cake and also amount of brine that escapes from the mud invert emulsion system.

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APPENDICES

APPENDIX A-1

Scomi
Scomi Oiltools

CONFI-MUL P

Product Data Sheet

Product Description

CONFI-MUL P is a formulated blend of emulsifiers for use as a primary emulsifier in the SCOMI Oiltools Invert drilling mud system, CONFI-DRILL.

Typical Properties

APPEARANCE	Liquid	SOLUBILITY IN WATER @ 20 °C	Insoluble
SPECIFIC GRAVITY	0.88 - 0.95	FLASH POINT	>90 °C (194 °F)
COLOUR	Dark	POUR POINT	14 F (-10 C)

Applications/ Functions

CONFI-MUL P produces stable emulsions which are resistant to high temperatures and to contamination. CONFI-MUL P imparts good fluid loss properties to the mud. Lime is required to fully activate the emulsion and provide tight fluid loss control. It is recommended that CONFI-MUL P be used in conjunction with CONFI-MUL S in order to produce invert muds that have excellent emulsion stability with variety of base oils.

Advantages

- > CONFI-MUL P Improves emulsion stability.
- > CONFI-MUL P has secondary wetting agent capabilities.
- > CONFI-MUL P helps maintain HTHP fluid loss in a water-free state.
- > CONFI-MUL P will enhance thermal stability and increase contamination tolerance of oil mud.
- > CONFI-MUL P is stable to temperatures above 204 °C (400 °F)

Recommended Treatment

Treatments levels range from 2 - 6 (bbl) (5.7 - 22.8 kg/m³), depending on properties desired as well as other components in the present system.

Recommended Handling

Consult MSDS before use and use personal protective equipment as advised.

Packaging

CONFI-MUL P is packaged in 55 gallon (208 litre) drums. Store away from heat and fire sources.

November 2010

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APPENDIX A-2

Scomi
Scomi Oiltools

CONFI-MUL S

Product Data Sheet

Product Description

CONFI-MUL S is a proprietary surfactant blend for use as a secondary emulsifier in invert drilling mud systems.

Typical Properties

APPEARANCE	Liquid	SOLUBILITY IN WATER @ 20 °C	Insoluble
SPECIFIC GRAVITY	0.88 – 0.96	FLASH POINT	>60 °C (140 °F)
COLOUR	Dark	POUR POINT	25 °F (-5 °C)

Applications/ Functions

CONFI-MUL S offers emulsion stability as well as high temperature tolerance and resistance to contamination. CONFI-MUL S does not require lime, however the use of lime will enhance the emulsion stability and the HTHP Fluid Loss.

Advantages

- > CONFI-MUL S Improves emulsion stability.
- > CONFI-MUL S has secondary wetting agent capabilities.
- > CONFI-MUL S helps maintain HTHP fluid loss in a water-free state.
- > CONFI-MUL S will enhance thermal stability and increase contamination tolerance of oil mud.

Recommended Treatment

Treatments can range from 2 - 6 lb/bbl (5.7 - 22.8 kg/m³), depending on properties desired as well as other components in the present system.

Recommended Handling

Consult MSDS before use and use personal protective equipment as advised.

Packaging

CONFI-MUL S is packaged in 55 gallon (208 litre) drums. Store at moderate temperature in dry, well ventilated area. Keep in original container and tightly closed.

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APPENDIX A-3

Scomi
Scomi Oiltools

CONFI-GEL

Product Data Sheet

Product Description

CONFI-GEL is a high yielding organophilic viscosifier and gelling agent for synthetic and oil-based muds. It increases rheology, improves the carrying capacity and gel strength enhancing the suspension of weighting materials and other solids.

Typical Properties

COMMON NAME	Organophilic Clay	CHEMICAL FORMULA	Proprietary
APPEARANCE	Powder	SOLUBILITY IN WATER @ 20 °C	Dispensable
SPECIFIC GRAVITY	1.5 -1.7	ODOUR	Fatty Odour
COLOUR	Off White to Tan		

Applications/ Functions

CONFI-GEL is an effective viscosifier in oil and synthetic base drilling, coring, workover and completion fluids increasing the carrying capacity and hole cleaning characteristic of the fluid. It may also be used in specialty fluids such as casing packs, packer fluids, lost circulation pills and spotting fluids, where viscosity is required.

Advantages

- High performance viscosifier for oil- and synthetic based fluids.
- Improves filter cake quality for reduced fluid loss.
- Provides efficient gelling capabilities for blending casing packs and packer fluids.
- Increases hole-cleaning capacity and weight-material suspension.

Recommended Treatment

Normal treatment may vary from 2 - 10 lb/bbl (5.7 – 28.5 kg/m³) or higher depending on the base fluid type and the application. Special treatment such as casing packs and packer fluids will generally range 10 - 15 lb/bbl (28.5 – 42.75 kg/m³). Pilot test all treatments.

Limitations

- Requires supplementary viscosifiers when high low end rheology required.
- Needs temperature and shear to fully yield.
- Effectiveness depends on type of base fluids.
- Effectiveness reduced at temperatures above 300 °F (149 °C).
- Higher concentrations required in high oil water ratio systems.

Recommended Handling

Consult MSDS before use and use personal protective equipment as advised.

Packaging

CONFI-GEL is packaged in 50 lb and 25 kg sacks. Store in a dry location away from sources of heat or ignition.

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APPENDIX A-4

HALLIBURTON | Baroid

ADAPTA®

Filtration Control Agent

Product Data Sheet

Product Description

ADAPTA® filtration control agent is a cross-linked polymer that can provide filtration control in all non-aqueous systems up to 425°F (218°C). It can also provide secondary viscosity and is suitable for use in fluids designed for Deep-water application. ADAPTA filtration control agent is the primary filtration control agent for the ACCOLADE® technology high performance clay-free drilling.

Applications / Functions

- Can reduce HPHT in all oil and synthetic mud systems in temperature ranges up to 425°F (218°C)
- Can provide secondary viscosity

Advantages

- Is an extremely effective HPHT filtrate reducer in concentrations as low as 1.0 to 4.0 lb/bbl (2.9-11.4 kg/m³)
- Easily mixed in dry form through the hopper with rapid results
- Stable to 425°F (218°C)
- Allows formulation of deep-water fluids with excellent low temperature tolerance

Typical Properties

- | | |
|--------------------|------------------|
| • Appearance | Off-white powder |
| • Specific gravity | 1.03 |

Recommended Treatment

- Add 1.0 to 4.0 lb/bbl (2.9-11.4 kg/m³) for HPHT filtration up to 350°F (177°C).
- Add 4.0 to 6.0 lb/bbl (11.4-17 kg/m³) for HTHP filtration greater than 350°F (177°C).

Packaging

ADAPTA filtration control agent is packaged in 55.1-lb (25-kg) bags.

www.halliburton.com/baroid

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