A COMPARISON STUDY OF MALAYSIAN MICA AS LOSS CIRCULATION MATERIAL IN HIGH PRESSURE HIGH TEMPERATURE DRILLING FLUID

By

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CERTIFICATION OF APPROVAL

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Approved by,

_____

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

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 by unspecified sources or persons.

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Zaiyinul Ain Bin Zainal Rafit

ABSTRACT

It has been observed for years that lost circulation is one of the troublesome and costly problems encountered during drilling operation even with the best drilling practices. Loss circulation can be classified according to the severity of mud lost rate into the stealing formation. Loss circulation material (LCM) is one of the methods to solve this problem considering the fact that lost circulation is one of the most serious and expensive problems.

There are several other materials that have been used as LCM in industries. Unfortunately, neither of the LCM materials are the product of Malaysia. This proposal discusses on the basic understanding of the chosen topic, which is The Comparison Study of Malaysian Mica as LCM in HTHP Water-Based Drilling Fluid.

Preliminary research has led to a further study on the subject until the satisfactory result is obtained. This research will be a stepping stone for future research on the potential drilling fluid additives which is obtainable from abundant local resources. Malaysian Mica will be experimented for possible use as LCM and to be compared with the characteristics of the existing commercialized Mica in the market. The source of Mica is taken from Tapah, Malaysia supplied by KAOLIN(M) SDN BHD, a quarry operating company.

However due to some constraints face by the author, Calcium Carbonate is used as comparative LCM instead of imported Mica.

This project has involved many laboratory works to test the efficiency of Malaysian Mica as LCM in water-based drilling fluid. Finally, this project has identified the suitability of Malaysian Mica to be use in drilling operation as LCM. However, additional works are required to further study on the Malaysian Mica.

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INTRODUCTION

1.1 Background of Study

Drilling fluid performance is a major component that contributes to the drilling operations success. This fluid is mainly used to promote borehole stability, removing drilled cuttings from borehole, cool and lubricate the bit and drillstring and to control the subsurface pressure.

Drilling fluid unable to achieve its optimum performances because there are undesirable formation conditions causing drilling fluids lost to the formation. A proper designed drilling fluid will enable an operator to achieve and overcome the desired geological objectives at the lowest overall cost.

According to Ross. M. C., Williford J., and Sanders M. W, fluid loss has long been recognized as a major concern when determining completion costs and assessing well management. Even with best drilling practices, fluid circulation loss is still occurring. For this reason, many researches have been dedicated to investigate various methods, materials and equipments to address the scenarios from which results in fluid loss.

Lost circulation defines the loss of drilling fluid into the formation voids instead of returning up to the surface. Loss circulation occurs as mud pressure applied is more than formation pressure, thus causing mud flows into fractured formation. This process is known as overbalanced drilling. Lost circulation can take place while drilling is in progress or during “trips”, when pressure surges occur because of the lowering of drillpipe or casing in the hole. After the lost circulation, the level of the drilling fluid in the annulus may drop and stable at a particular level, depending on the formation pressure (Nayberg T., 1987). Loss zone can be classified as seepage loss (minor loss),

partial loss and complete loss(major loss). Loss circulation problem is both troublesome and costly such as lost rig time, stuck pipes, blow outs and reduction in production.

To reduce the loss circulation problem, the selection of proper Loss Circulation Material (LCM) is vital so that the material is able to block the passage for fluid to diffuse and loss to the formation. There are several famous LCM used in the oil well drilling industry by SCOMI Oil Tools Sdn Bhd (a Malaysian company) such as calcium carbonates, Mica and wallnut shells. Focusing on Mica material, Malaysian is one of the biggest Mica producers in Asia and it was found that the only area that produces Mica is in Tapah, Perak. Currently, Mica is used in industrial applications such as paints and cosmetic. In the rubber industry, Mica is used to mould the lubricant, fluxing agent in welding electrodes and reinforcement in plastics [1]. From the lists, there is no application of Malaysian Mica in oil and gas industry especially to be set as LCM in drilling fluid.

1.2 Problem Statement

1. The usage of imported Mica from India or Unites States causes the drilling fluids to be expensive.
2. There is no track record of locally produced Mica being used as LCM in drilling fluids.
3. No significant research available on the usage of Malaysian Mica in HPHT water-based drilling fluid (WBM).

1.3 Objectives

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

1. Formulate water-based mud that is compatible with LCM chosen and laboratory testing with current technology.
2. Study the suitability of LCM chosen in HPHT water-based drilling fluid.
3. Compare the efficiency and compatibility of imported LCM used in industry.

1.4 Scope of Study

The research involve in understanding LCM in drilling fluid. This project involves the identification of the appropriate LCM and the method of studying and evaluating effectiveness of LCM in WBM.

The scope of study mainly investigates the fluid loss properties of the commercialized LCM and Malaysian Mica. The study is divided into two stages; the first stage involves research on the basic properties of the Malaysian Mica and determining an ideal formulation to be developed. The second stage will focus on experimental work in the lab, using different amount of Malaysian Mica with particular attention given to the characteristics of Malaysian Mica and its fluid loss behavior.

1.5 Relevancy and Feasibility of the Project

This project is relevant to the author's field of study since loss circulation is one of the concerns in drilling process. This study will be one of the earliest researches to be carried out using Malaysian Mica as LCM in water-based mud. Moreover, most of drilling operation in Malaysia is using water-based mud. The source of Malaysian Mica is from Tapah, Perak [1]. In this project, the author has applied fluid mechanics and drilling process theory to find cost-effective LCM for loss circulation problem and create methods of environmental sustainability, conservation and protecting efforts to evaluate the effectiveness of the LCM. As a petroleum engineer, the author has evaluated the current LCM to find the most cost-effective solution where the author has proposed Malaysian Mica as a new type of LCM to the industry.

The project is feasible since it is within the scope and time frame. The author has completed the research and literature review in related topics by reading books, journals and research papers at the end of the first semester while preparing the material after the mid-semester break. Research has been done for better understanding on loss circulation material and the experimental work on the fluid loss factor. The research approximately took two (2) months time. All the involved variables was identified and understood to make the desired drilling fluid. Once the desired drilling fluid is formulated, the lab work begins to find the favorable properties of Malaysian Mica.

CHAPTER 2

LITERATURE REVIEW

2.1 Literature Review

Loss circulation is a major problem in determining the completion cost and during the assessment of well management. Numerous papers have been written over the years on loss circulation. Many of these papers describe a specific method that has been used to address the problem, and a number of innovative devices and fluids have been developed.

According to *Rheological Properties of Drilling Muds in Deep Offshore Conditions* published in 2001, in order to design drilling fluid, a few important characteristics of drilling fluid must be known and tested which are fluid density, rheology properties, fluid loss properties by filtration and pH level. These significant properties are said to be important as to ensure drilling fluid's appropriate strength, viscosity, gel strength, yield point, mud pressure and its compatibility with downhole equipments.

When it comes to high pressure high temperature (HPHT) condition, there are certain required performances of the fluid properties need to be achieved so that the drilling fluid would be compatible in HPHT well. An article entitled *High Pressure, High Temperature Well Construction* written by A.Keelan et al. in 1998, mention the required performances as follows:

Table 1: Required Performances for HPHT Drilling Fluid (A. Keelan et al. 1998)

Properties	Required Performance
Plastic viscosity	As low as reasonable possible to minimize ECD.
Yield stress and gel	Sufficient to prevent sag, but not so high as to cause gelation, or high surge and swab pressures.
HPHT fluid loss	As low as reasonable possible to prevent formation damage and risk of differential sticking.
HPHT rheology	Predictable in order to control sag, gelation and ECD.
Compressibility	Must be known to estimate downholes pressures and ECD.

Besides these performances, the article did mention about some disadvantages of oil-based mud (OBM) which the highly soluble gas in base fluid lead to increasing in gas volume rapidly when fluid heading to surface. This could lead to less fluid circulation back to surface or can even be hazardous. In addition, OBM also has higher thermal expansion than water-based mud (WBM). Therefore, the mixing of any new LCM should meet the required properties and compatible with LCM used in the industry.

According to the journal *Effect of Material Type and Size Distribution on Performance of Loss/Seepage Control Material* there are four types of formations are responsible for lost circulation. There are natural fractured formations, cavernous formations, highly permeable formations or unconsolidated formations and induced fracture formations. For all type of formations, circulation losses could occur in varying degrees even with the best drilling practices and the severity of these losses is an indicator of the mud loss to the formation. Loss zones can be classified as:

Table 2: Loss Zone Classification (Ali A. Pilehvari 2002)

Type of Loss Zones	Lost Severity (bbl/hr)
Seepage Loss	1-10
Partial Loss	10-500
Complete Loss	>500

Besides that, Pilehvari A. and Nyshadham R. mentioned a wide variety of materials have been used to combat lost circulation over the years. The choice of lost circulation material usage in a given case is influenced to some degree by cost and availability in a given drilling area. They classified LCM into fibers, flakes, granules and mixtures. The fibrous LCM are used mainly in drilling mud to reduce the mud loss into large fractures or vugular formations (pore spaces consisting of cavities or vugs) [4], where as flaky type LCM's can plug and bridge many types of porous formations to establish an effective seal over many permeable formations. The granular LCM's form bridges at the formation face and within the formation matrix, thus providing an effective seal, which depends primarily on proper particle size distribution. Finally blended LCM's are combination of granular, flake and fibrous materials that will penetrate fractures, vugs or extremely permeable zones and seal them off more effectively.

For the study of LCM, the paper entitled *Laboratory Study of Lost Circulation Materials for Use in Both Oil-Based and Water-Based Drilling Mud* published by Nayberg T. on 1987 was reviewed. The objective of this paper is to give a rough idea on estimating the appropriate loss circulation material (LCM) to be used in drilling fluid to prevent loss circulation. In this paper, LCM can be divided into three groups according to their morphology: fiber (ex.: raw cotton and cedar wood fibers), flakes (ex.: cellophane, MICA and cork) and granules (ex.: grounded walnut shell and gilsonite). There are four basic factors affecting the performance of a LCM which are the concentration of LCM in mud, LCM particle size distribution, the size of largest particles in the material and the quantity of the largest particles.

Table 3: Groups of LCM

Fibrous Materials	Flake Materials	Granular Materials
• Raw Cotton • Wood Fibre • Bark Fibre • Textile Fibre • Mineral Wool • Straw • Asbestos • Peat Moss • Feathers • Flax	• Mica • Cellophane • Cork • Cotton seed • Plastics	• Nut Shells • Nut hulls • Rice hulls • Wood • Corn cobs • Asphalt • Ground Rubber • Salt • Bentonite • Limestone • Perlite

Mark W. Sanders, Jason T. Scorsone and James E. Friedheim in their journal published in 2010 describes and discusses the development of high fluid loss, high strength pill system and its optimization using innovative testing methods to ensure that it meets field criteria to solve loss circulation problems. In this paper, it is also stated that the level of complexity for evaluating LCM procedures vary. The test methods range from using simple, low pressure, API fluid loss test that use filter paper, to more sophisticated tests involving slots, ceramic discs or natural cores.

Referring to the application and usage of LCM in Malaysia, the author need to look into materials used by Malaysian Drilling Fluid company such as Scomi Oil Tools Sdn Bhd. Scomi Oil Tools have been utilizing some common LCM such as Calcium carbonates, Mica, Wallnut shells and other formulated LCM with their proprietary names [2]. Focusing on Mica material, Malaysian is one of the biggest Mica producer in

Asia and the only area that producing it is in Tapah, Perak. Up to now, Mica is used in industrial applications such as paints and in cosmetic applications, moulds lubricant in the rubber industry, fluxing agent in welding electrodes and reinforcement in plastics [1] and there is no application of Malaysian MICA in oil and gas industry especially to be set as LCM in drilling fluid.

It is found that the muscovite type Mica [2] used by Scomi Oil Tools is mainly from Gujerat, India and United States of America (USA). One of the biggest Mica suppliers in Malaysia; KAOLIN (M) SDN BHD has produced chemically hydrated potassium alumina silicate which is a muscovite type of Mica whereby the platy (thin, plate-like) structure is perfect for anti-blocking and anti-sticking agent [3]. Therefore, in order to overcome the high expenditure on imported Mica for LCM in drilling fluid, there must be a way to utilize Malaysian Mica in the industry.

However, recently, a well-known Malaysian Service Company; Scomi Oil Tools Sdn. Bhd had increased the application of Calcium Carbonate (OPTA-CARB) as LCM in WBM drilling fluid. Calcium Carbonate used for LCM is mainly in the form of limestone; a granular material which is able to form bridges at the formation face and within the formation matrix, thus providing an effective seal, which depends primarily on proper particle size distribution. Calcium Carbonate is also used as an acid soluble weighting material for drill-in or workover fluids with a density of 14.0 ppg or less [5]. In a water based system the pH of the drilling fluid needs to be above 7.0 since the Calcium Carbonate is acid soluble. It is proven by the test done by GEO Drilling Fluid Incorporation where Calcium Carbonate is 98 to 99.5% soluble in 7.5-15% hydrochloric. At a lower pH, it will begin to dissolve and contaminate the drilling fluid with calcium and will no longer be effective as a lost circulation material [5]. However, the solubility with acid solution does give benefit as there will be minimized permanent plugging of pore especially on the productive formation.

A case study of drilling activity at Pakistan proved a successful usage of LCM of OPTA *CARB*-Sized Calcium Carbonate. The result was, the depleted “Patala” formation was killed and sealed successfully with the OPTA *CARB*-Sized Calcium

Carbonate pills [7]. The well that had been producing from "Patala" formation for several years; with a depleting production rate, the operator wanted to deepen the existing well in order to explore a new horizon. The well faced several challenges, primarily; high temperature and losses in depleted formation.

STEELSEAL, a Calcium Carbonate LCM formulated by Halliburton have proved there is no adverse effect on the rheological properties, even in concentrations as high as 100 ppb. Laboratory studies show that STEELSEAL treatments help lower both spurt loss and particle-plugging test (PPT) values on 20- and 35-micron discs [8] which in other term, the drilling fluid properties remain unimpaired.

Furthermore, STEELSEAL performance can be seen at several stages. At pre-treatment, active fluid system in a concentration of 20-30 ppb can help prevent seepage or lost circulation while drilling through depleted zones, or in preparation for running casing and cementing. 50 to 100 ppb also successfully curing losses by controlling severe losses without plugging downhole drilling tools thus save the trip time [9].

In addition, Calcium carbonate also acts as a weight material to increase mud weight, which may contribute to other problems such as a higher ECD and overbalanced drilling but this may be compensated for by the use of salt such as Potassium Chloride to increase the water phase density. However, there have been case studies in certain formations in Qatar where damage has been done after using Calcium Carbonate.

CALCIUM CARBONATE TREATMENT

Table 1

Weight, ppg	Viscosity, sec/qt	Soda ash, lb/bbl	Caustic soda, lb/bbl	Type	Size, µ	Concentration, lb/bbl
9.2-10.5	120	0.75	0.5	Fine Medium	5-15 15-45	100 50-70

Figure 1: Calcium Carbonate Treatment

In order to quantify the LCM leak-off characteristics and evaluate compressibility of water (Cw) and spurt loss values, fluid-loss tests were performed with samples from each of the three zones. The cores were first saturated with API brine followed by the LCM under an overbalanced pressure of 1,000 psi to obtain fluid loss data.

Tests indicated fluid-loss control was achieved with no spurt loss. Thus, filtrate volumes would be lost if control were needed for a long period of time. A result of packed carbonate particles and partially dehydrated gel residue, the resulting filter cakes also showed a tenacious behavior [10]. To compare "after fluid loss" values with "before fluid loss" values, they then compared end pieces from the same core samples which finally shows lower reaction rate constants.

As a comparison to both Calcium Carbonate and Mica, Drilling Specialties Company in its Lost Circulation Guide says in treating entire drilling fluid system, Solids control equipment such as hydro cyclones; flow line cleaners; mud cleaners; and centrifuges cannot be used when treating the total circulating system unless very fine LCM is used. Again caution should be exercised when using products like fine calcium carbonate, as this will cause an increase in mud weight. Similarly, fine flake materials such as mica and some fiber materials should be avoided as this may increase the resistance to flow and thus increase surge and swab pressures" [9]. The author also find out that Mica and other flake type LCM's result in a lower fracture propagation pressure and it is not recommended. In productive zones, Industry studies indicate that fibrous LCM's can cause formation damage to productive zones. Core tests showed fluid invasion to the extent that fibers passed completely through the cores on return permeability tests. In tight sands, which will be fractured, this may not be of concern. [9]

2.2 Theory

Drilling fluis can be classied depending on the base fluid that is used. Generally, there are 3 types of drilling fluids which are water-based muds (WBMs), oil-based muds (OBMs), and synthetic-based muds (SBMs).

WBMs are commonly regarded as not harmful to the marine environment. WBMs are also generally used in offshore drilling. However, OBMs provide a number of advantages over WBMs that include superior borehole stability, thinner filter cake, excellent lubricate, and less risk of stuck pipe. The major disadvantage of OBMs is that the base fluid which consists of high level of toxicity poses an environmental hazard if it is released into the ocean either through a spill or on cuttings.

2.2.1 Process of mixing and testing drilling fluids

First and foremost, the mud formulation for water and oil/synthetic based muds is created using the mud formulator shown in figure 1. The mud formulator is an excel spreadsheet utilized to calculate the appropriate amount of products to be used to mix one lab barrel of mud which is almost 350ml in the laboratory. The final weight, type of mud, products such as weighting material, emulsifiers, viscosifiers, fluid loss agent and others are keyed into this spreadsheet and calculated.

Next, the base fluids and products are weighed according to the formulation calculated. The chemicals are then mixed according to the mixing time and order. In oil/synthetic based mud, the emulsifiers are commonly added first into the base fluid such as base oil, followed by the viscosifiers, fluid loss agent and finally the weighting material. In the laboratory, generally, the mixing time for water based mud is 45 minutes and for oil based mud is one hour. Once the mud is mixed, the initial properties of the mud are tested.

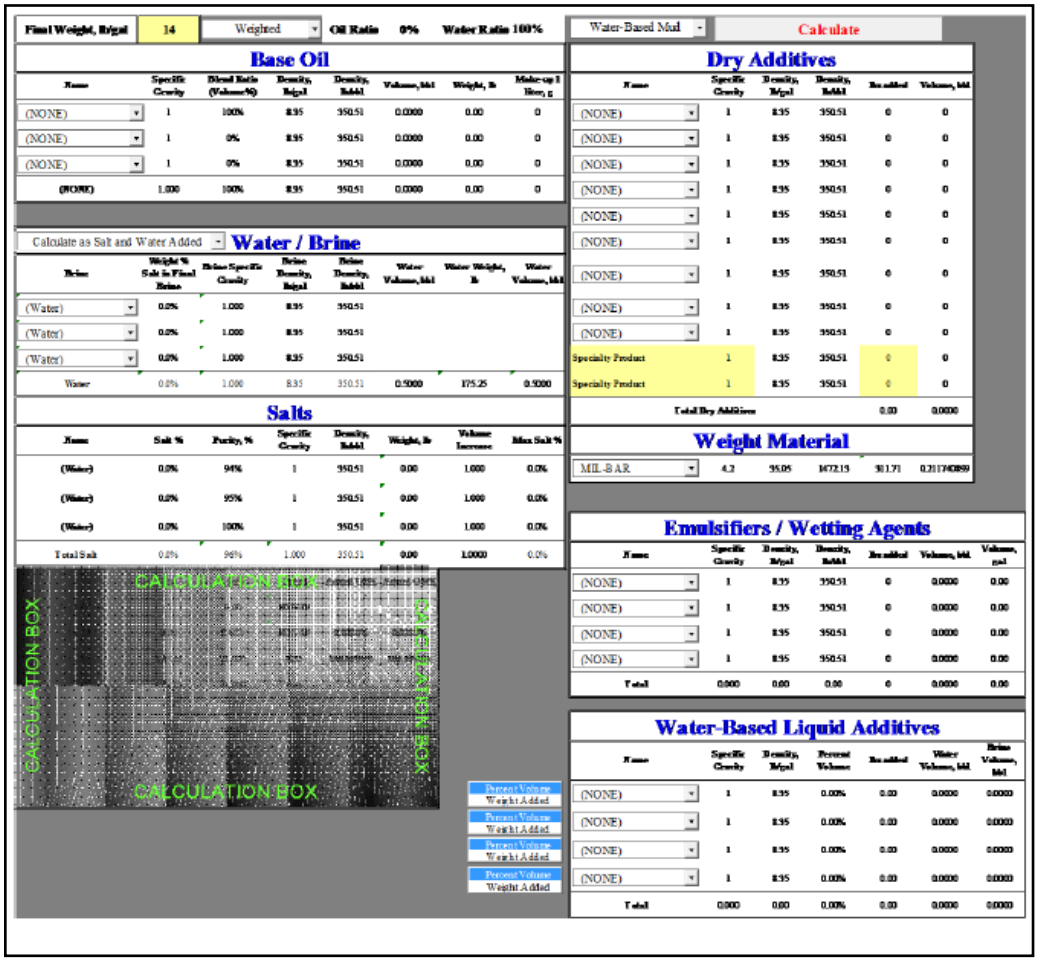

Figure 2: Example of Mud Formulator Spreadsheet

2.2.2 Properties of drilling fluids

Properties of the mud that we test for in the laboratory depend on the type of drilling fluid used. Figure 3 shows the main properties of mud that the author test upon in the laboratory and some properties testing carried out for only for a specific type of drilling fluid.

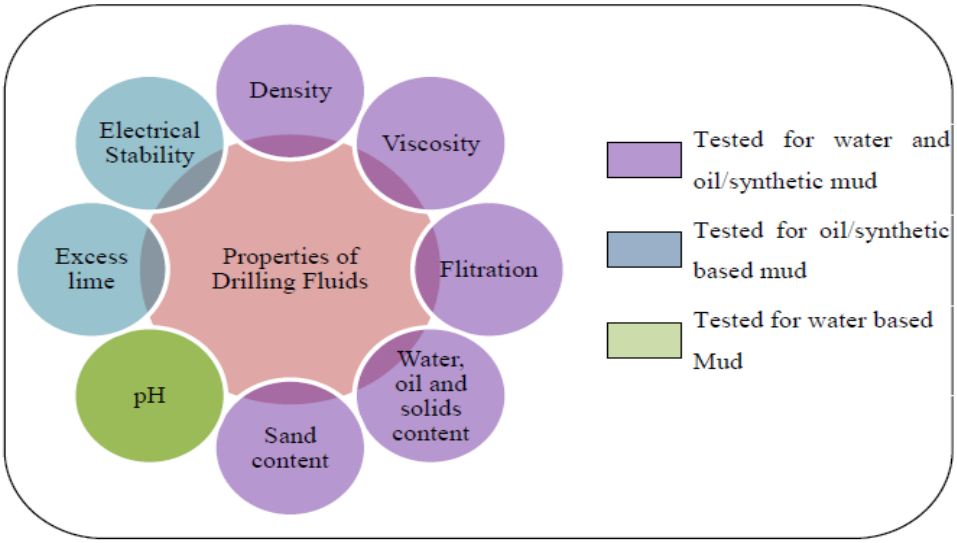

Figure 3: Properties of Drilling Fluids

Density

The density of any fluid, which is the mass per unit volume of the fluid, is directly related to the amount and average specific gravity of the solids in the system. Hydrostatic pressure which is exerted by the fluid column in the wellbore should be maintained ideally slightly higher than the formation pressure. This is to insure maximum penetration rate with minimal danger from formation fluids entering the 19 wellbore and also to aid in keeping the borehole open. Equations below are used to calculate the hydrostatic pressure exerted by the fluid column:

Hydrostatic Pressure (psi) = 0.052 × Depth (ft) × Fluid Density (lbm / gal)

Fluid density is generally expressed in lbm/gal (lbm/ft3 in some locations) and in specific gravity or g/cm3. Common method for checking the density of any drilling fluid which is the regular mud balance shown in Figure 4.

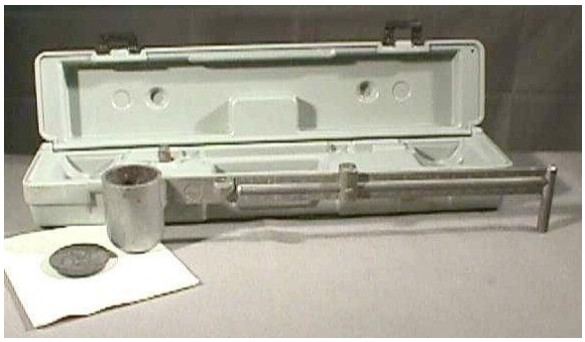

Figure 4: Regular Mud Balance

Viscosity

Viscosity of fluids defined as the resistance of fluids to flow. Viscosity measured in the unit of poise which is equivalent to dyne-sec/cm2. One poise represents a high viscosity, therefore the generally unit that represents the fluids is centipoises. A centipoises is equivalent to 1/100 poise or 1 millipascal-second. This property of fluids is significant in hole cleaning to control the settling rate of drill cuttings generated by the drill bit through moving fluid and bring them up to the surface.

There are two main apparatus that the author has utilized in the laboratory which are marsh funnel and direct indicating viscometer. Marsh funnel shown in Figure 5 is a simple device for routine measurement of drilling fluids viscosity. The viscosity measured through this apparatus is known as funnel viscosity. The Marsh funnel is dimensioned so that the outflow time of one quart freshwater (946 cm3) at a temperature of 70° ± 5°F (21° ± 3°C) is 26 ± 0.5 seconds. Thus, fluid which records a time more than 26 ± 0.5 seconds using the marsh funnel is more viscous compared to freshwater and vice versa(Baker Hughes Drilling Fluid Reference Manual, 2006).

Figure 5: Marsh Funnel Viscosity

Besides that, there is an easier way to measure the flow properties of the fluid. This equipment is called general equipment V-G (viscosity-gel) meter, or direct indicator viscometer as illustrated in Figure 6.

Figure 6: Fann 35 Direct Indicating Viscometer

Gel strength

Gel strengths of drilling fluid indicate the thixotropic properties and they are measurements of the attractive forces under static conditions in relationship to time. Generally, gel strengths will increase with time, temperature, and increase in solids. The gel strength determines the pressure required to break circulation when the drilling is 24 shutdown for a certain time. If the gel strength is high, a very high pressure is required to initiate the flow of the fluid in the wellbore.

At times it may be necessary to break circulation at intervals while running into the hole rather than to initiate flow in the entire wellbore at the same time in order to minimize the pressure spike to initiate circulation. Besides that, the fluid should have sufficient gel strength to provide the suspension property under static condition. This property should be able to help the fluid to suspend weight material and drill cuttings when the circulation ceases(Baker Hughes Drilling Fluid Reference Manual, 2006).

Gel strength is measured by using the V-G meter. Gel strength must be measured at 10 seconds (initial gel), 10 minutes and 30 minutes intervals. Sometimes, in the laboratory the gel strength is also measured at one hour interval. The gear for the V-G meter is switched from 600rpm to 300rpm and then is switched off. After the testing time interval for example 10 seconds, the gear is switched to 300rpm and the gel strength is measured. The gel strength is measured in the unit of lb/ 100ft2. The types of gel strength are described in the Figure 7.

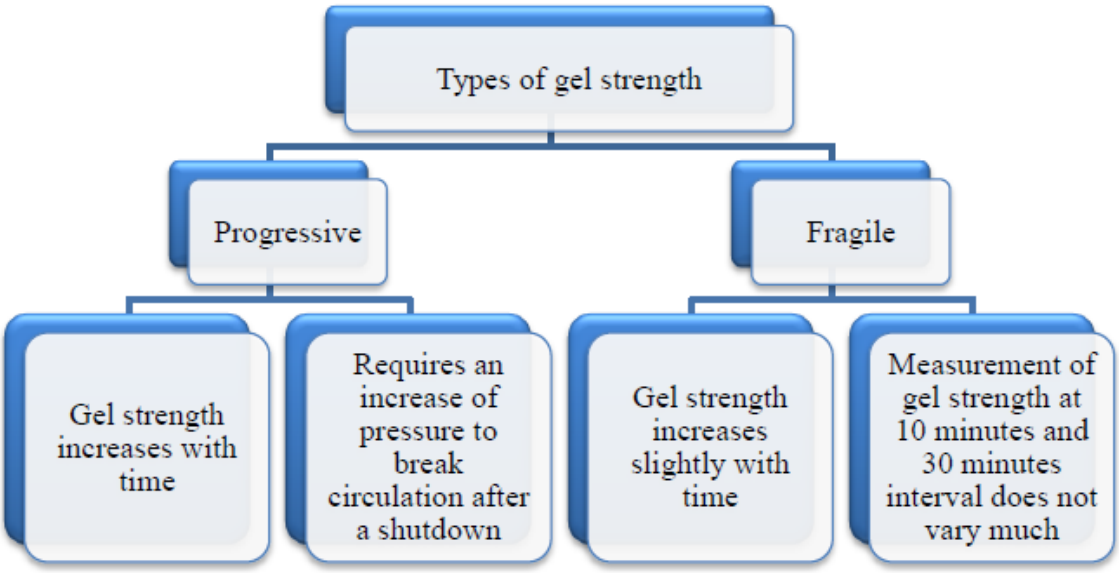

Figure 7: Types of Gel Strength

Filtration

Filtration control is one of the main factors considered essential in drilling. Filtration measures the relative amount of fluid lost through permeable formations or membranes when subjected to pressure. Thus, it is important to minimize the filtrate invasion to the formations. When drilling permeable formations, filtration rate is often the most important property where the hydrostatic pressure exceeds the formation pressure. Proper control of filtration improves the borehole stability chemically. This is because controlling the fluid loss minimizes the potentially detrimental interaction between the filtrate and the formation. Filtrate invasion may be controlled by the type and quantity of colloidal material and by filtration control materials.

Besides that, controlling fluid loss helps to put off or reduce wall sticking and drag. Filtration control is also significant in formation evaluation as invasion of mud filtrate may influence the readings taken. The readings may represent the mud filtrate rather than the formation fluid properties. Besides that, quality of filter cake which is the suspended solids of a drilling fluid that deposited on a porous medium during the process of filtration is also important. The fluid loss amount is inversed to the thickness of filter cake deposited. The physical property of a cake is stated in notations like “hard”, “soft,” “tough,” rubbery” and “firm”.

There are two types of filtration which are static and dynamic. In static filtration, the drilling fluid is tested when it is not in motion whereas dynamic filtration occurs when the drilling fluid tested is being circulated. In static filtration the wall cake will continue to be deposited as the drilling fluid is not in motion, the velocity is zero. When a drilling fluid is tested using dynamic filtration, the velocity of the fluid will erode the wall of mud cake as it is deposited. The state of equilibrium exists if the rate of erosion equals the rate of build-up of the wall cake.

The high temperature/high pressure (HTHP) test is conducted using the HTHP filter press shown in the picture below at a temperature greater than ambient and it requires differential pressure of 500 psi. The HTHP filtrate is collected for a period of 30 minutes in cubic centimeters and the filtrate volume is doubled to correct it to the filter area of the API filtration test. The permeable medium used is the same as that used for the low temperature test. The filter cake should also be assessed for thickness and consistency after the filtrate loss has been tested (Baker Hughes Drilling Fluid Reference Manual, 2006).

Figure 8: High Temperature High Pressure (HTHP) Filter Press

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

The assessment on the efficiency of Malaysian MICA as LCM in comparison with Calcium Carbonate is done in water-based drilling fluid. The main criteria for evaluating the LCM are through running the fluid loss experiment.

Figure 9 shows the general flow of the research methodology while the detail description is described in the

Table 4.

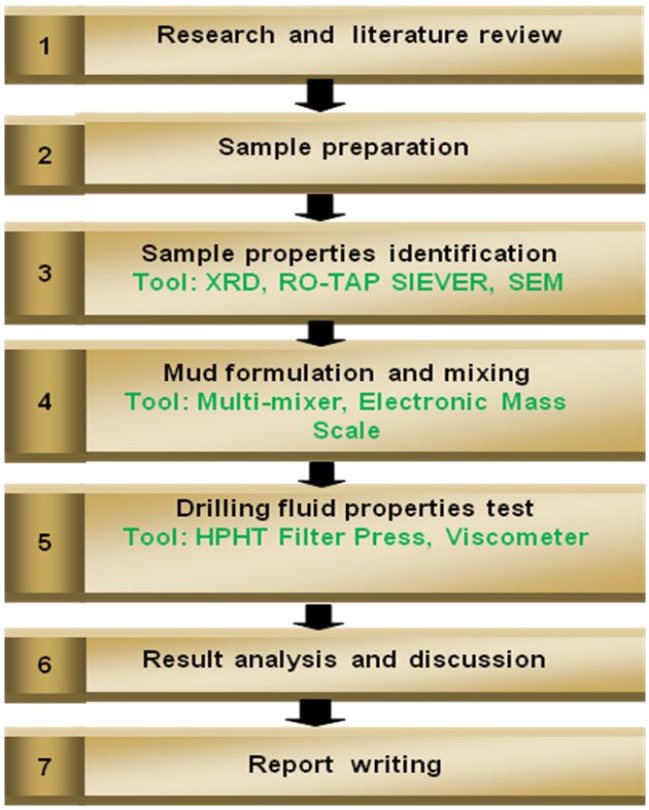

Figure 9: Research Methodology

Table 4: Activities and Description

Activities	Description	
Research and Review Literatures	- Building the research base - Extract relevant parameters and procedures	
Preparation of LCM and mud formulation	- Order MICA in powder form prior to mix with mud - Design mud formulation for water-base mud and oil based mud system to analyze the LCM applicability and effectiveness. - Tools required (multimixer)	
Testing mud with industrial used LCM	- Prepare water-based mud and with current uses Calcium Carbonate. - Measure all the properties of mud prior to comparison with Malaysian MICA later	
Testing mud with new LCM	**Properties**	**Tools Required**
	Density	Mud Balance
	Viscosity	March Funnel
	pH value	pH Meter
	- Plastic Viscosity - Gel Strength - Yield Point	FANN Viscometer FANN High Pressure High Temperature Filter Press Viscometer
	- Filtrate Volume - Mud cake thickness	High Pressure High Temperature Filter Press
Analyze the Results	Discuss the findings from the results obtained and make a conclusion out of the study	
Report Writing	Compilation of all works into a final report	

3.2 Gantt Chart and Key Milestone

Table 5: Gantt chart for the first semester project implementation

	WEEK														
ACTIVITIES	**1**	**2**	**3**	**4**	**5**	**6**	**7**		**8**	**9**	**10**	**11**	**12**	**13**	**14**
Selection of Project Topic															
Study on LCM and current technology of testing LCM															
Study and do research about Malaysian MICA															
Submission of Extended Proposal															
Proposal Defense															
Construct experiment to study on industrial used LCM design															
Analyze the features and parameters that can be measured															
Submission of Interim Draft Report															
Submission of Interim Report															

Mid Semester Break

Table 6: Gantt chart for the second semester project implementation

	WEEK														
ACTIVITIES	**1**	**2**	**3**	**4**	**5**	**6**	**7**		**8**	**9**	**10**	**11**	**12**	**13**	**14**
LCM preparation															
Conduct experiment to study new LCM (Malaysian MICA)															
Conduct experiment to further test LCM (Oversea Mica/$CaCO^3$)															
Submission of progress report															
Compare and analyze the results															
Submission of technical paper															
Submission of dissertation (soft bound)															
Oral presentation															
Submission of project dissertation (hard bound)															

Legend:

Completed timeline

Dateline for submission/present

CHAPTER 4

RESULTS AND DISCUSSIONS

The results for this experiment can be divided into two sections:

i. Physical and physical properties of the materials
    a. XRD test
    b. SEM(scanning electron Microscope)
    c. Particle Size Distribution
ii. Properties of materials in drilling fluid
    a. Rheology properties
    b. Plastic Viscosity
    c. Yield Point
    d. Gel Strength
    e. pH
    f. Fluid Loss
iii. Limitations and constraints

4.1 Physical and chemical properties of materials

The materials mentioned in this section are Malaysian Mica and Calcium Carbonate. Both materials are tested as comparison of LCM, which is used to prevent fluid loss in drilling fluid. Calcium Carbonate is used instead of oversea Mica as LCM because there are limitations and constraints faced by the author as it will be explained later.

4.1.1 XRD Results

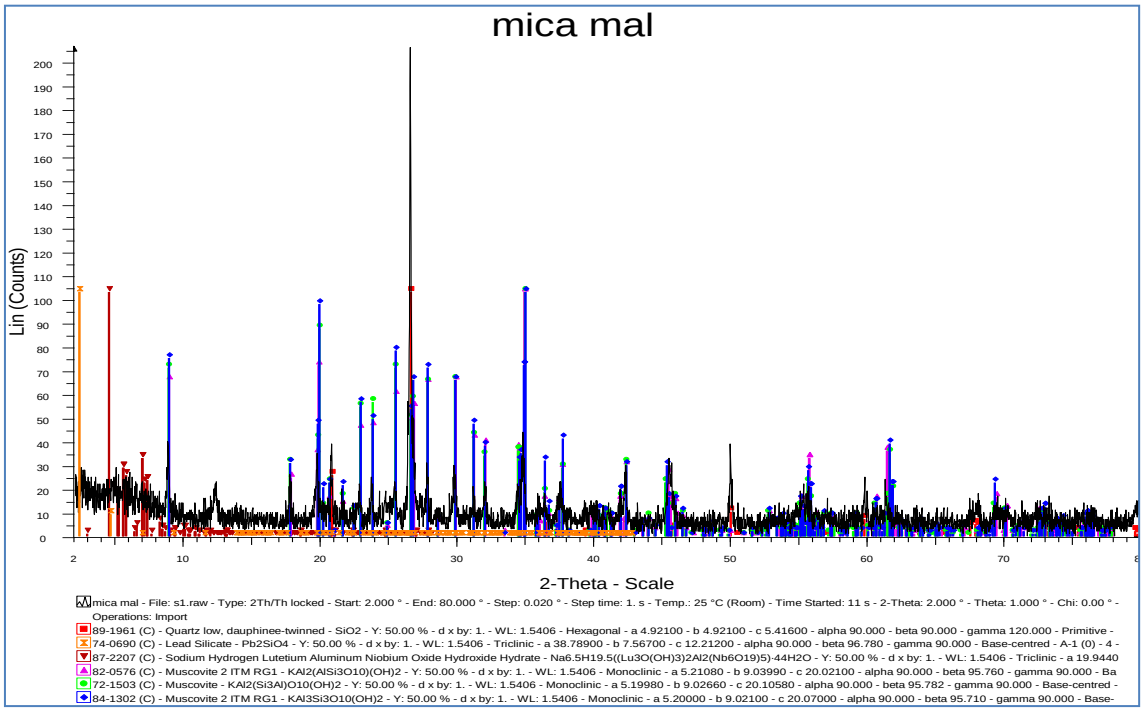

Figure 10: XRD Results for Malaysian Mica

4.1.2 Scanning Electron Microscope (SEM)

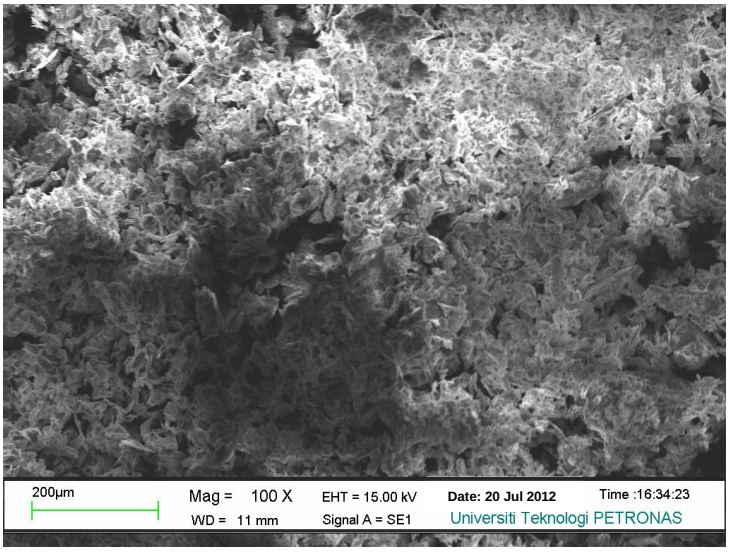

Figure 11: Malaysian Mica at 100X magnification

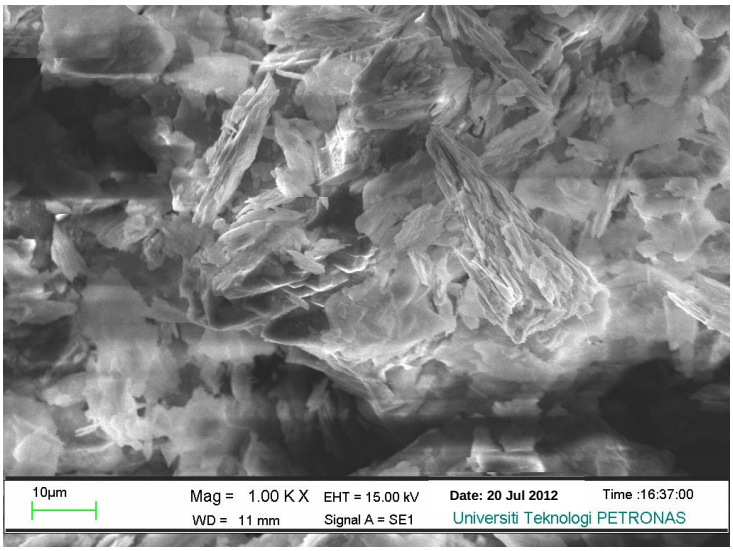

Figure 12: Malaysian Mica at 1000X magnification

4.1.3 Particle Size Distribution (RO-TAP Siever)

Table 7: Particle Size Distribution for Malaysian Mica

Screen Size, µm	Screen Size, mm	Blank Weight, g	Final Weight, g	Weight of Residue, g	Weight %, Retained
1180	1.18	351.17	351.35	0.18	0.04
600	0.6	329.89	503.72	173.83	34.61
425	0.425	296.35	432	135.65	27.01
300	0.3	355.15	463.21	108.06	21.52
212	0.212	275.94	307.96	32.02	6.38
150	0.15	276.43	279.32	2.89	0.58
0	0	387.73	437.34	49.61	9.88
Total Sample Weight ,g =				502.24	100.00

Table 8: Particle Size Distribution for Calcium Carbonate

Screen Size, µm	Screen Size, mm	Blank Weight, g	Final Weight, g	Weight of Residue, g	Weight %, Retained
1180	1.18	434.7	434.7	0.00	0.00
600	0.6	403.2	403.2	0.00	0.00
425	0.425	366.7	373.7	7.00	1.50
300	0.3	358.1	434.2	76.10	16.36
212	0.212	346	428.4	82.40	17.71
150	0.15	292.8	451.8	159.00	34.17
0	0	393.2	534	140.80	30.26
Total Sample Weight ,g =				465.3	100.00

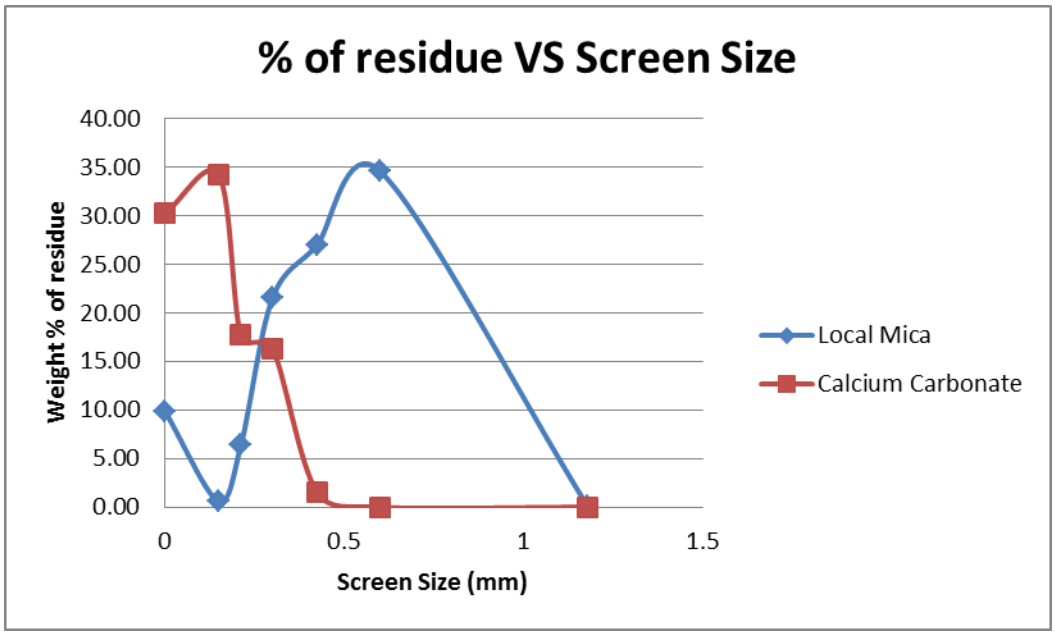

Figure 13: PSD of LCM samples

4.1.4 Discussion on Physical and Chemical Properties

Based on both the XRD results interpretation, Malaysian Mica is from Mica Muscovite(ground Mica) with general chemical formula of KAl2(AlSi3O10)(F,OH)2. This validates the early findings of mineralogy of Malaysian Mica through literature reviews. Meanwhile sized Calcium Carbonate used in the project is actually ground limestone with its general chemical formula of $CaCO^3$. Based on these results, direct comparison on physical properties of Malaysian Mica and Calcium Carbonate cannot be made since the materials are different. However, comparison as LCM in drilling fluid still applicable as Malaysian Mica is expected to be alternative material for LCM.

The most important element to make direct comparison between both LCM is the particle size. As mentioned by Ali A Pilehvari and Venkata R. Nyshadham, there are four factors affecting LCM performances which are the concentration of LCM in mud, LCM particle size distribution, the size of largest particles in the material and the quantity of the largest particles. Thus, there is no doubt that amount of similar particle size should be the main manipulated variables. Hence, sieving test are used to get the desirable particles size of both LCM materials.

The siever used for this experiment is called the RO-TAP Siever. Multiple screen sizes, varying from 1800μm up to 0μm are used. They are all stacked up according to size, 1800μm being the most top while 0μm the most bottom. The material is put into the top sieve and the machine is started. Sieving takes place and the particles are automatically distributed. Table 7 and Table 8 show the particle size distributions (PSD) of Malaysian Mica and Calcium Carbonates materials respectively.

Based on Table 7, the highest amount of Malaysian Mica residue is gathered with screen size of 600μm unlike in Table 8 as the highest amount of Calcium Carbonate residue is filtered at screen size of 150 μm. Malaysian Mica has a maximum weight residue of 173.83g which is equivalent to 34.61 % when converted to percentage. Meanwhile Calcium Carbonate has a maximum weight residue of 159g which is equivalent to 34.17 %. The data in Table 7 and Table 8 are used to plot Figure 13; the particle size distribution (PSD) for Malaysian Mica and Calcium Carbonate.

Figure 13 shows that as the screen size increases, the weight percentage of Malaysian Mica also increases until reaching its optimum point at screen size 600µm before declining to almost zero gram at 1.18mm screen size. After that, the weight percentage starts to decrease ending at screen size 2mm. In contra with Malaysian Mica, weight percentage of Calcium Carbonate shows declining trend after reaching optimum point at screen size 150 µm. There is no residue at screen size of 600µm and 1.18mm. Due to inconsistence trending of PSD between Malaysian Mica and Calcium Carbonate, the author decides to take the appropriate range of particle size at the intersection point of two lines as shown in Figure 13. The accumulation of particles at screen size 0 µm to 300 µm are selected as LCM samples for this project hence will be taken into consideration throughout the experiments.

4.2 Discussion on properties of materials in drilling fluid

The experiments were conducted according to the standard which has stipulated in American Petroleum Institute - API 13B-2; ‘’Recommended Practice Standard Procedure for Testing Oil-Based Drilling Fluid’’ (Appendix 1). Drilling mud samples were prepared by increasing the amount of LCM 1 (Malaysian Mica) and LCM 2 (Calcium Carbonate). Table 9 below shows the formulations of the mud that have been tested at three stages; initial, after hot roll and using HPHT Viscometer:

Table 9: Tested Formulation

Product	**Ori**	**Mica-T1**	**Mica-T2**	**Mica-T3**	**Mica-T4**	**$CaCO^3$-T1**	**$CaCO^3$-T2**	**$CaCO^3$-T3**	**$CaCO^3$-T4**
Fresh Water	330	330	330	330	330	330	330	330	330
Soda Ash	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30
Potassium Chloride	44	44	44	44	44	44	44	44	44
HYDRO-PAC LV	2.50	2.50	2.50	2.50	2.50	2.50	2.50	2.50	2.50
HYDRO-ZAN	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
DRILL-BAR	83.66	75.83	68.01	60.18	57.00	75.63	67.60	59.56	53.00
Caustic Soda	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
Malaysian Mica		10.00	20.00	30.00	40.00				
Calcium Carbonate						10.00	20.00	30.00	40.00
Initial	**Ori**	**Mica-T1**	**Mica-T2**	**Mica-T3**	**Mica-T4**	**$CaCO^3$-T1**	**$CaCO^3$-T2**	**$CaCO^3$-T3**	**$CaCO^3$-T4**
Mud weight, lb/gal	10.5	10.5	10.5	10.5	10.5	10.5	10.5	10.5	10.5

Rheological properties at									
600 RPM	63	68	59	62	63	48	43	55	58
300 RPM	47	48	41.5	44	43	33	28	37	39
200 RPM	39	39	34	36	37	28	23	32	35
100 RPM	28	28	24	26	27	20	16	23	27
6 RPM	15	10	16	12	13	9	5	8	9
3 RPM	8	8	7	7	8	5	4	6	7
PV, cP	16	20	17.5	18	20	15	15	18	19
YP, lb/100 ft2	31	28	24	26	23	18	13	19	20
Gel 10 sec, lb/100 ft^2	14	18	19	13	12	10	8	11	13
Gel 10 min, lb/100 ft^2	23	20	21	18	17	16	9	15	17
pH	12.23	12.33	12.35	11.64	11.5	11.97	11.95	12.22	12.31
HPHT after hotroll	**Ori**	**Mica-T1**	**Mica-T2**	**Mica-T3**	**Mica-T4**	**$CaCO^3$-T1**	**$CaCO^3$-T2**	**$CaCO^3$-T3**	**$CaCO^3$-T4**
Rheological properties at									
600 RPM	18	20	16	19	22	16	14	15	17
300 RPM	12	13	10	12	14	10	8	10	12
200 RPM	10	10	9	9.5	11	7	4	6	7
100 RPM	7	7	6	8	9	8	9	1.2	
6 RPM	1.5	2	2	3	3	0	0	0	
3 RPM	1	1	1	2	2	0	0	0	
PV, cP	6	7	6	7	8	6	6	5	5
YP, lb/100 ft2	6	6	4	5	6	4	2	5	7
Gel 10 sec, lb/100 ft^2	7	9	10	6	5	7	4	6	8
Gel 10 min, lb/100 ft^2	12	10	11	8	7	13	6	11	14
HPHT, cc/30min	5.42	6.92	6.67	5	5.42	5.08	4.67	4.42	5
cake thickness, mm									
HPHT Viscometer	**Ori**	**Mica-T1**	**Mica-T2**	**Mica-T3**	**Mica-T4**	**$CaCO^3$-T1**	**$CaCO^3$-T2**	**$CaCO^3$-T3**	**$CaCO^3$-T4**
Mud weight, lb/gal	10.5	10.5	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Rheological properties at									
600 RPM	5.5	5.8	6.5	6.7	6.9	4	4.6	6.9	7.4
300 RPM	2.8	2.9	3.5	5.3	5.7	2.9	3.9	7.3	8.5
200 RPM	0	0	0	2.9	3.2	1.3	2.4	7.3	8.4
100 RPM	0	0	0	0	1	0	0	1.2	1.8
6 RPM	0	0	0	0	0	0	0	0	0.9
3 RPM	0	0	0	0	0	0	0	0	0
PV, cP	2.7	2.9	3	1.4	1.2	1.1	0.7	-0.4	-1.1
YP, lb/100 ft2	0.1	0	0.5	3.9	4.5	1.8	3.2	7.7	9.6
Gel 10 sec, lb/100 ft^2	0	0	0	0	0	0	0	0	0
Gel 10 min, lb/100 ft^2	0	0	0	0	0	0	0	0	0
HPHT, cc/30min	5.42	6.92	6.67	5	5.42	5.08	4.67	4.42	5

4.2.1 Plastic viscosity (PV)

Plastic viscosity is a function of solids concentration and shape. It can be increased by addition of more lost circulation material in the mud. It will be expected to increase with decreasing particle size with the same volume of solids. Moreover, it also can be increased by addition of more lost circulation material in the mud. This can be proven in the experiment on $CaCO^3$ while Malaysian Mica at its highest amount, decreases the PV. PV should be as low as possible in order to have low pumping rate for mud circulation or minimizing Equivalent Circulation Density (ECD). The interesting part here is that mud PV properties become stable after being hot rolled. The trend as shown is the same where mud with Malaysian Mica shows an increasing PV value.

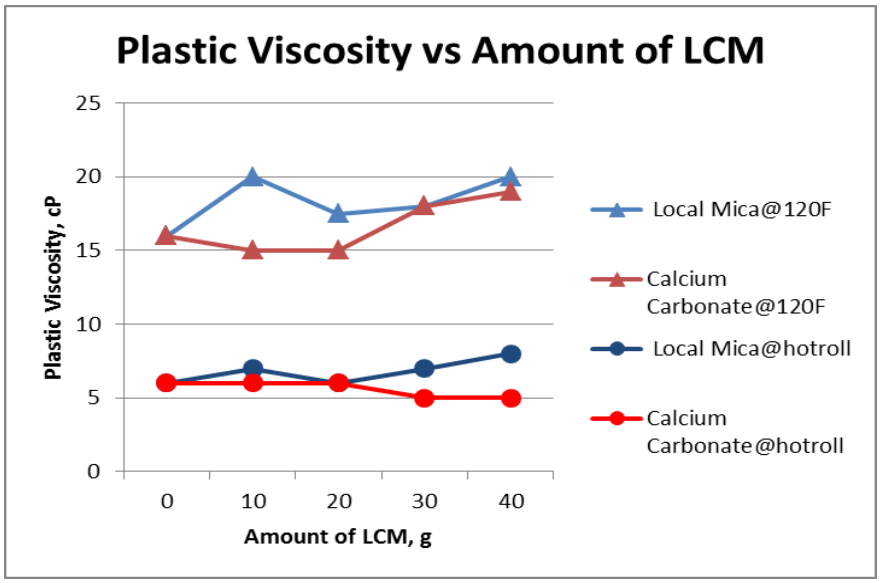

Figure 14: Plastic Viscosity before and after hot roll

4.2.2 Yield point (YP)

Yield point is the attractive force in the mud under flow conditions. The value of yield point will increase as the amount of solid increased. The magnitude of these forces will depend on the type of their solid present, the ion concentration in the liquid phase (Growcock F, 2005). However, Figure 15shows the value of YP for mud decreases as the concentration of LCM increased and at LCM amount of 30g, the YP increases back. Similar to plastic velocity, YP should be as low as possible in order to have sufficient to prevent sag, but not so high as to cause gelation, or high surge and swab pressures. The YP properties of the mud become stable after hot rolled similar to PV properties with the lower value of YP compared to initial YP value.

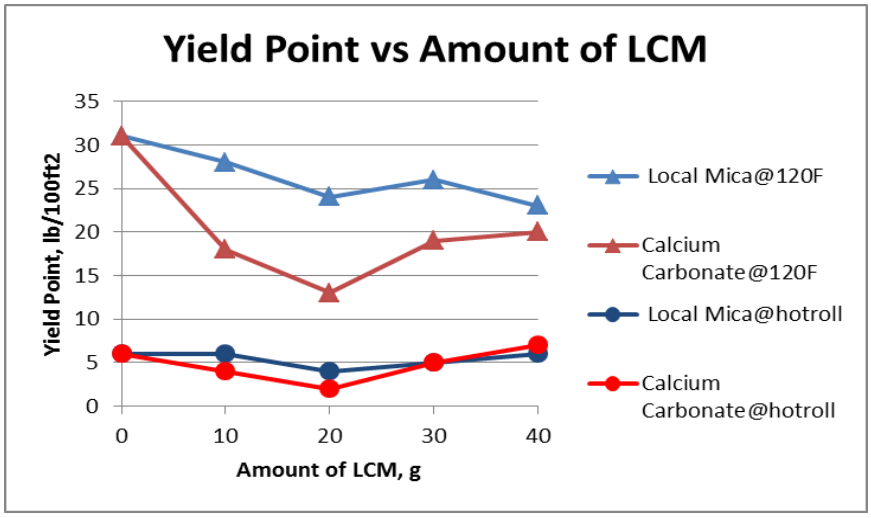

Figure 15: Yield Point before and after hot roll

4.2.3 Gel Strength

Gel strength indicates the pressure required to initiate flow after the mud has been static for some time and the suspension properties of the mud. It is the ability of a drilling fluid to suspend the cutting when the drilling fluid is in stationary condition. Gel strength, 10 seconds and 10 minutes indicate the strength of attractive forces in drilling fluid under static condition. Excessive forces are caused by high solids concentration leading to flocculation. The 10 minutes gel strength will lead to a higher

flocculation since it has more time. The best drilling fluid has fragile gel strength where the force needed to break the circulation is low over time. In general, gel strength should be optimizing which can prevent sag. However, too high gel strengths are not desirable and can even be dangerous. However, there is no significant change of gel strength by Malaysian Mica compared to $CaCO^3$ because the concentration of Malaysian Mica does not give significant change to the gel strength reading. The trend of gel strength before and after hot roll shows a similar trend. The difference is on the value as the gel strength after hot roll is lower which might indicates the real situation in well bore.

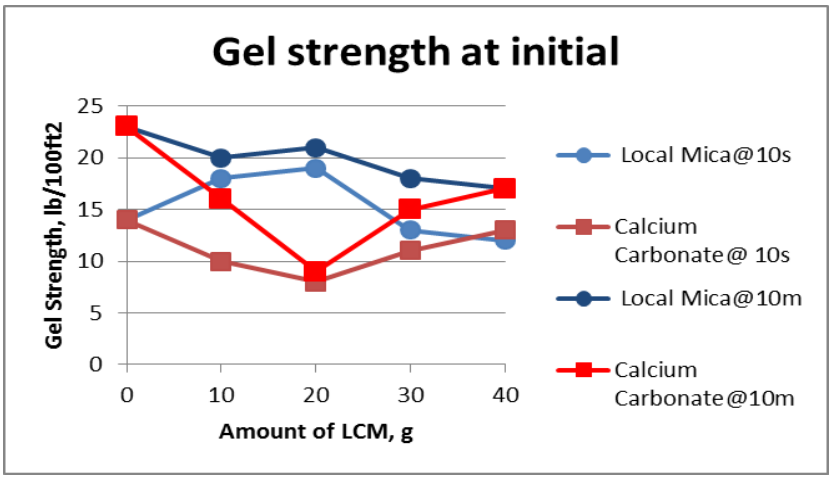

Figure 16: Gel Strength at 120F

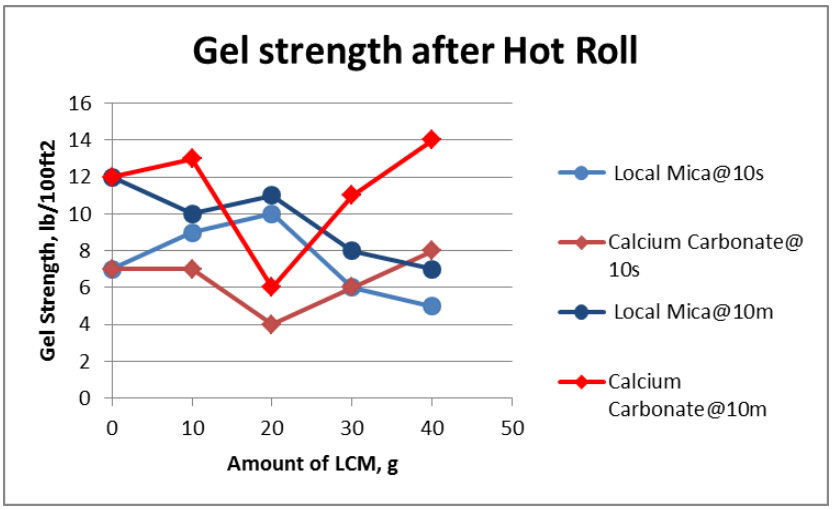

Figure 17: Gel Strength after hot roll

4.2.3 Fluid Loss and Mud Cake

Based on the experiment, it is observed that the solid from the mud will form a layer of solid called “mud cake” on the filter paper where the mud is pressurized. Filtrate volume from the experiment indicates the amount of fluid loss from the mud to the formation where it simulates the quantity of fluid loss inside the wellbore. The preferable filter cake should be thin, impermeable, and have correct solids distribution to prevent fluid loss effectively. In normal conditions, Thick filter cake will increase the chance of stuck pipe. The lower the filtrate volume the thinner the mud cakes, means that good fluid loss control in mud.

An impressive result of fluid loss is shown in Figure 18 when both LCM increases, the fluid loss decreases. Only when LCM amount of 40g, fluid loss starts to increase back maybe due to excessive Mica in the drilling fluid.. It shows that there must have optimum amount of LCM to be used in the formulation. It validates that Malaysian Mica can be used as an alternative LCM with better formulation. Based on the results and discussions above, the optimum concentration of Malaysian Mica and Calcium Carbonates is 30g thus it shows there is a potential comparable study from both LCM.

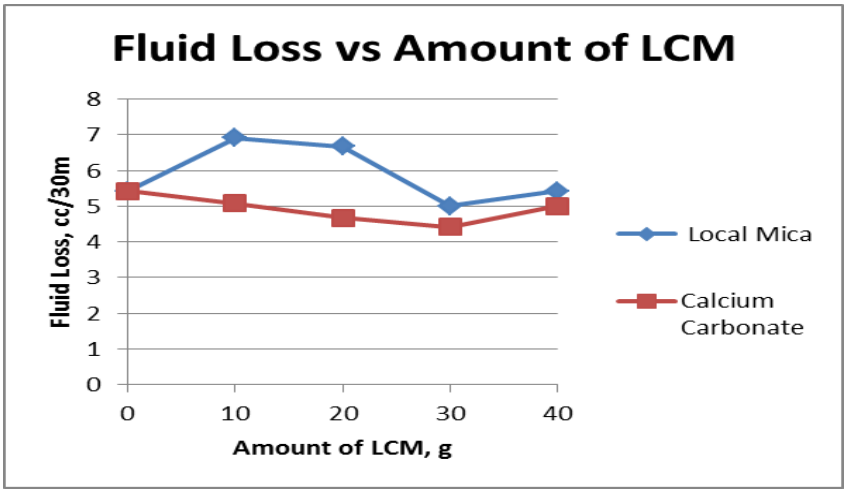

Figure 18: Fluid Loss

4.3 Limitations and constraints

There are some constraints the author face upon completing the project especially in the LCM preparation activity. According to the timeline, the author should have started the experiment provided that both samples from local and oversea Mica are ready. However, the first experiment is out of schedule as it delayed about a month due to:

i. Late confirmation from sample supplier on the unavailability oversea Mica in stock. In addition, oversea Mica is recently no longer being used as LCM in WBM drilling fluid due to safety reasons.

ii. Unavailability of oversea or other LCM materials in UTP lab with same range of sizes which is around 20 to 60 mesh. Therefore, the author needs to do sieving tests to each available LCM material in the lab which surely takes time.

iii. Late confirmation on which backup LCM materials that is suitable to replace oversea Mica. The last option is by using Calcium Carbonate as it is recently being used by Scomi Oil Tools Sdn. Bhd to replace the oversea Mica.

iv. Unable to get preferable Calcium Carbonate particle size corresponding to Malaysian Mica particle size. Therefore, the range of particle size taken for the experiment is the intersection point between both LCM in PSD graph.

v. XRD and SEM machine can only uses to test on Malaysian Mica as the material is available early. This is due to first four constraints above. The available machine at block 16 is malfunction and the only available machine is fully booked until next year. Also, the author has done a survey at other universities, also fully booked by their students and also UTP students.

vi. The difficulties of using HPHT Viscometer at UTP Mud Lab where there are limited personnel who know how to use the machine.

vii. The result of rheological properties using HPHT Viscometer shows there is no value for gel strength, very low PV and YP value. Most viscosity reading at 200rpm, 100rpm, 6rpm and 3rpm is zero cP. Therefore, it is somehow indicates that the water-based drilling fluid formulated by the author maybe have some faultiness. The author believes that the HPHT Viscometer have simulated the drilling fluid at real condition of wellbore as it has been pressured up to 100psi and 250F. (See Appendix 2).

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The aim of the project is to identify the effectiveness of Malaysian MICA as a LCM and this is achieved by comparison with Calcium Carbonate instead of conventionally MICA imported from overseas. The comparative study mainly covers on rheological characterization, fluid loss control and pH test.

The project has been involved a lot of experimental work in the UTP concrete lab and UTP mud lab. Malaysian Mica is proven:

i. Possess the characteristics of LCM which is able to form bridges between pores in the formation. It is approved by the declining of fluid loss volume.

ii. Potential to have preferred drilling fluid properties. It is approved by the PV and YP value trending which will be able to be as low as possible for value optimization.

iii. Able to stabilized drilling fluid properties after being hot rolled.

iv. To lead to a more cost efficient alternative to the normally used imported Mica.

5.2 Recommendations

There are still a lot of things need to be done first before the product can be commercialized to the market as the experiments only covered the testing of the mud with certain size of Malaysian Mica only. Various experimental testing should be done. The author recommends the following:

1. Further testing with all different particle size (fine, medium and coarse) are still needed to confirm the effectiveness of using Malaysian Mica as lost circulation material in the industry.

2. More tests should be conducted to get an accurate result such as formation damage system test, X-Ray fluorescence test, and etc. These tests should be able to justify, identify and investigate further the properties of the fluid and the Malaysian Mica itself.

3. Better experimental practice should be exercised all the time so that the expected result is approaching the theoretical result.

4. Various ways of experiments should be test such as; varying the temperature and pressure to check the effect on drilling fluid properties stability.

5. Mixing of certain sizes of particles can be implemented as it is believed different particles size can be like a “supportive team” to each other when it comes to forming bridges or plugs.

6. Combining different properties of LCM such as flacky shape with granular can be a good combination.

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APPENDIX 1: Recommended Practice Standard Procedure For Field Testing Oil-Based Drilling Fluid (1998), American Petroleum Institute.

Recommended Practice for Field Testing of Oil-based Drilling Fluids

1 Scope

This Recommended Practice provides standard procedures for determining the following characteristics of oil-based drilling fluids:

a) drilling fluid density (mud weight);

b) viscosity and gel strength;

c) filtration;

d) oil, water and solids contents;

e) alkalinity, chloride content and calcium content;

f) electrical stability;

g) lime and calcium contents, calcium chloride and sodium chloride contents;

h) low-gravity solids and weighting material contents.

Annexes A, B, C, D, H, I, K and L provide additional test methods that may optionally be used for the determination of

i) shear strength,

j) oil and water contents from cuttings,

k) drilling fluid activity,

l) aniline point,

m) cuttings activity,

n) active sulfides.

o) PPA test method for cells with set screws.

p) PPA test method for cells with screw-on caps.

Annexes F, G and J provide procedures that may optionally be used for

q) sampling, inspection and rejection,

r) rig-site sampling,

s) calibration and verification of glassware, thermometers, viscometers, retort kit cups and drilling fluid balances.

Annex E provides examples of calculations for

t) lime, salinity and solids content.

Annex M contains an example of a drilling fluid report form.

2 Terms and definitions

For the purposes of this Standard, the following term and definition applies:

2.1
ACS reagent grade
grade of chemical meeting the purity standards specified by the American Chemical Society (ACS)

2.2
API
American Petroleum Institute, 1220 L Street NW, Washington, DC 20005

2.3
CAS
Chemical Abstracting Service

2.4
USC
United States Customary unit, shown in parentheses following SI unit

3 Abbreviations

ACS	American Chemical Society
BAD	Base alkalinity demand
EDTA	eithylenediaminetetraacetic acid
ES	electrical stability
HT/HP	high temperature, high pressure
OCMA	Oilfield Chemical Manufacturer's Association
PNP	propylene glycol normal-propyl ether
PTFE	polytetrafluoreoethylene, brand name Teflon®
TC	to contain
TD	to deliver
R_{300}	viscometer reading at 300 r/min
R_{600}	viscometer reading at 600 r/min
?	static filtration rate

m_1 mass of retort cup, lid and body with steel wool, g

m_2 mass of retort cup, lid, body and cuttings, g

m_3 mass of empty liquid receiver, g

m_4 mass of liquid receiver and fluid collected during solids analysis, g

m_5 mass of solids remaining in retort cup following solids analysis, g

R static filtration rate

V volume of liquid collected in receiver, ml

V_o volume of oil, cm^3

V_s volume of solids, cm^3

V_1 volume of filtrate after 7,5 min, cm^3

V_2 volume of filtrate after 30 min, cm^3

V_w volume of water, cm^3

η_P viscosity of plastic viscosity

η_Y viscosity of yield point

η_A appararent viscosity

φ_o volume fraction of oil

φ_s volume fraction of solids

φ_w volume fraction of water

ρ density

$\nabla\rho$ density gradient

4 Determination of drilling fluid density (mud weight)

4.1 Principle

A procedure is given for determining the mass of a given volume of liquid (= density). The density of drilling fluid is expressed as grams per cubic centimetre, kilograms per cubic metre, pounds per gallon or pounds per cubic foot.

4.2 Apparatus

a) Any **density-measuring instrument** having an accuracy of ± 0,01 g/cm^3, ± 10 kg/m^3, ± 0,1 lb/gal, or ± 0,5 lb/ft^3.

The mud balance is the instrument generally used for drilling fluid density determinations. The mud balance is designed such that the drilling fluid holding cup, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the

beam to allow for accurate balancing. Attachments for extending the range of the balance may be used when necessary.

The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 1,00 g/cm^3 or 1 000 kg/m^3 at 21 °C (70 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

b) **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F).

4.3 Procedure

4.3.1 The instrument base should be set on a flat, level surface.

4.3.2 Measure the temperature of the drilling fluid and record.

4.3.3 Fill the clean, dry cup with drilling fluid to be tested; put the cap on the filled drilling-fluid holding cup and rotate the cap until it is firmly seated. Ensure that some of the drilling fluid is expelled through the hole in the cap, in order to free any trapped air or gas.

4.3.4 Holding the cap firmly on the drilling-fluid holding cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

4.3.5 Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the centreline.

4.3.6 Read the drilling fluid density at the edge of the rider toward the drilling-fluid cup. Make appropriate corrections when a range extender is used.

4.4 Calculation

4.4.1 Report the drilling fluid density, ρ_s, to the nearest 0,01 g/cm^3 , 10 kg/m^3, 0,1 lb/gal or 0,5 lb/ft^3.

4.4.2 To convert the reading to other units, use the following:

$$\rho_s = 1\,000 \times \text{g/cm}^3 \quad (1)$$

$$\rho_s = 16 \times \text{lb/ft}^3 \quad (2)$$

$$\rho_s = 119{,}8 \times \text{lb/US gal} \quad (3)$$

where ρ_s is the density, expressed in kilograms per cubic metre.

$$\nabla\rho_s = 9{,}81 \times \text{g/cm}^3 \quad (4)$$

$$\nabla\rho_s = 0{,}0226 \times \text{psi/1 000 ft} \quad (5)$$

where $\nabla\rho_s$ is the drilling fluid density gradient, expressed in kilopascals per metre.

A list of density conversions from SI to USC units is given in Table 1.

Table 1 — Density conversions between SI and USC units

Grams per cubic centimetre[a] g/cm^3	Kilograms per cubic metre kg/m^3	Pounds per US gallon (lb/US gal)	Pounds per cubic foot (lb/ft^3)
0,70	700	5,8	43,6
0,80	800	6,7	49,8
0,90	900	7,5	56,1
1,00	1 000	8,345[b]	62,3
1,10	1 100	9,2	68,5
1,20	1 200	10,0	74,8
1,30	1 300	10,9	81,0
1,40	1 400	11,7	87,2
1,50	1 500	12,5	93,5
1,60	1 600	13,4	99,7
1,70	1 700	14,2	105,9
1,80	1 800	15,0	112,1
1,90	1 900	15,9	118,4
2,00	2 000	16,7	124,6
2,10	2 100	17,5	130,8
2,20	2 200	18,4	137,1
2,30	2 300	19,2	143,3
2,40	2 400	20,0	149,5
2,50	2 500	20,9	155,8
2,60	2 600	21,7	162,0
2,70	2 700	22,5	168,2
2,80	2 800	23,4	174,4
2,90	2 900	24,2	180,7

[a] Same value as relative density.

[b] Accurate conversion factor.

5 Alternative method for determination of drilling fluid density

5.1 Principle

5.1.1 The pressurized mud balance provides a more accurate method for determining the density of a drilling fluid containing entrained air or gas than does the conventional mud balance. The pressurized mud balance is similar in operation to the conventional mud balance, the difference being that the slurry sample is placed in a fixed-volume sample cup under pressure.

5.1.2 The purpose of placing the sample under pressure is to minimize the effect of entrained air or gas upon slurry density measurements. By pressurizing the sample cup, any entrained air or gas is decreased to a negligible volume, thus providing a slurry density measurement more closely in agreement with that obtained under downhole conditions.

5.2 Apparatus

a) Any **density-measuring instrument** having an accuracy of ± 0,01 g/cm^3, ± 10 kg/m^3, ± 0,1 lb/gal, or ± 0,5 lb/ft^3.

The pressurized mud balance is the instrument generally used for density determinations of pressurized drilling fluids. The pressurized mud balance is designed such that the drilling-fluid holding cup and screw-on lid, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing.

Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 1,0 g/cm^3 or 1 000 kg/m^3 at 21 °C (69,8 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

b) **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F).

5.3 Procedure

5.3.1 Measure the temperature of the drilling fluid and record.

5.3.2 Fill the sample cup to a level slightly (approximately 6 mm) below the upper edge of the cup.

5.3.3 Place the lid on the cup with the attached check-valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Any excess slurry will be expelled through the check-valve. When the lid has been placed on the cup, pull the check-valve up into the closed position, rinse off the cup and threads with water, and screw the threaded cap on the cup.

5.3.4 The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submersing its end in the slurry with the piston rod completely inside. Then draw the piston rod upward, thereby filling the cylinder with slurry. This volume should be expelled with the plunger action and refilled with fresh slurry sample to ensure that this plunger volume is not diluted with liquid remaining from the last clean-up of the plunger mechanism.

5.3.5 Push the nose of the plunger onto the mating O-ring surface of the cap valve. Pressurize the sample cup by maintaining a downward force on the cylinder housing in order to hold the check-valve down (open) and at the same time to force the piston rod inside. A force of approximately 225 N (50 lbf) or greater should be maintained on the piston rod.

5.3.6 The check-valve in the lid is pressure-actuated; when the inside of the cup is pressurized, the check-valve is pushed upward into the closed position. To close the valve gradually ease up on the cylinder housing while maintaining pressure on the piston rod. When the check-valve closes, release pressure on the piston rod before disconnecting the plunger.

5.3.7 The pressurized slurry sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place instrument on the knife edge. Move the sliding weight right or left until the beam is balanced. The beam is balanced when the attached bubble is centred between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of grams per cubic centimetre, pounds per gallon, and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1 000 feet.

5.3.8 To release the pressure inside the cup, reconnect the empty plunger assembly and push downward on the cylinder housing.

5.3.9 Clean the cup and rinse thoroughly with base oil.

5.4 Calculation

Report the drilling fluid density to the nearest 0,01 g/cm^3 , 10 kg/m^3, 0,1 lb/gal, or 0,5 lb/ft^3.

For conversions, use the formulas given in 4.4.2.

6 Viscosity and gel strength

6.1 Principle

Viscosity and gel strength are measurements that relate to the flow properties (rheology) of drilling fluids. The following instruments are used to measure viscosity and/or gel strength of drilling fluids:

a) Marsh funnel — a simple device for indicating viscosity on a routine basis;

b) direct-indicating viscometer — a mechanical device for measurement of viscosity at varying shear rates.

NOTE Information on the rheology of drilling fluids can be found in API RP 13D.

6.2 Determination of viscosity using the Marsh funnel

6.2.1 Apparatus

a) **Marsh funnel**, calibrated to deliver 946 cm^3 (1 quart) of fresh water at a temperature of 21 ± 3 °C (70 ± 5 °F) in 26 ± 0,5 s, with a graduated cup as a receiver.

The Marsh funnel shall have the following characteristics:

1) **funnel cone**, length 305 mm (12,0 in), diameter 152 mm (6,0 in) and a capacity to bottom of screen of 1 500 cm^3 (1,6 quarts);

2) **orifice**, length 50,8 mm (2,0 in) and inside diameter 4,7 mm (0,185 in);

3) **screen**, with 1,6 mm (0,063 in) openings (12 mesh); fixed at 19,0 mm (0,748 in) below top of funnel.

b) **Graduated cup,** with capacity at least 946 cm^3 (1 quart).

c) **Stopwatch**.

d) **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F).

6.2.2 Procedure

6.2.2.1 Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill until fluid reaches the bottom of the screen.

6.2.2.2 Remove finger and start the stopwatch. Measure the time for drilling fluid to fill to the 946 cm^3 (1 quart) mark of the cup.

6.2.2.3 Measure the temperature of the fluid, in degrees Celsius (degrees Fahrenheit).

6.2.2.4 Report the time (6.2.2.2), to the nearest second, with the volume, as the Marsh funnel viscosity. Report the temperature (6.2.2.3) of the fluid to the nearest degree Celsius (degree Fahrenheit).

6.3 Determination of viscosity and/or gel strength using a direct-indicating viscometer

6.3.1 Apparatus

a) **Direct-indicating viscometer,** powered by an electric motor or a hand crank.

Drilling fluid is placed in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity. The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob. Instrument constants should be adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 r/min and 600 r/min.

The components shall meet the following specifications.

1) **Rotor sleeve**

Inside diameter	36,83 mm (1,450 in)
Total length:	87,0 mm (3,425 in)
Scribed line:	58,4 mm (2,30 in) above the bottom of sleeve, with two rows of 3,18 mm (0,125 in) holes spaced 120° (2,09 rad) apart, around rotor sleeve just below scribed line.

2) **Bob**, closed, with flat base and tapered top

Diameter:	34,49 mm (1,358 in)
Cylinder length:	38,0 mm (1,496 in)

3) **Torsion spring constant:**

386 dyne-cm/degree deflection

4) **Rotor sleeve speeds**

High speed:	600 r/min
Low speed:	300 r/min

NOTE Other rotor speeds are available in viscometers from various manufacturers.

b) **Stopwatch.**

c) **Thermostatically controlled viscometer cup.**

d) **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F).

6.3.2 Procedure

6.3.2.1 Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 100 cm^3) in the cup for displacement of fluid due to the viscometer bob and sleeve. Immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay from the time of drilling fluid sampling. Testing should be carried out at either (50 ± 1) °C [(120 ± 2) °F] or (65 ± 1) °C [(150 ± 2) °F]. The place of sampling should be stated on the report.

The maximum recommended operating temperature is 90 °C (200 °F). If fluids have to be tested above this temperature, either a solid metal bob, or a hollow metal bob with a completely dry interior should be used.

CAUTION Liquid trapped inside a hollow bob may vaporize when immersed in high-temperature fluid and cause the bob to explode.

6.3.2.2 Heat (or cool) the sample to the selected temperature. Use intermittent or constant shear at 600 r/min to stir the sample while heating (or cooling) to obtain a uniform sample temperature. After the cup temperature reaches the selected temperature, immerse the thermometer into the sample and continue stirring until the sample reaches the selected temperature. Record the temperature of the sample.

6.3.2.3 With the sleeve rotating at 600 r/min, wait for the viscometer dial reading to reach a steady value (the time required is dependent on the drilling fluid characteristics). Record the dial reading R_{600} in pascals for 600 r/min.

6.3.2.4 Reduce the rotor speed to 300 r/min and wait for the dial reading to reach steady value. Record the dial reading R_{300} in pascals for 300 r/min.

6.3.2.5 Stir the drilling fluid sample for 10 s at 600 r/min.

6.3.2.6 Allow drilling fluid sample to stand undisturbed for 10 s. Slowly and steadily turn the hand-wheel in the appropriate direction to produce a positive dial reading. Record the maximum reading as the initial gel strength. For instruments having a 3 r/min speed, the maximum reading attained after starting rotation at 3 r/min is the initial gel strength. Record the initial gel strength (10-second gel) in pounds per 100 square feet.

NOTE To convert the dial reading to pounds per 100 square feet: 1 Pa = 0,48 lb/100 ft^2.

6.3.2.7 Restir the drilling fluid sample at 600 r/min for 10 s and then allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in 6.3.2.6 and report the maximum reading as the 10-minute gel in pascals (pounds per 100 square feet).

NOTE To convert the dial reading to pounds per 100 square feet: 1 Pa = 0,48 lb/100 ft^2.

6.3.3 Calculation

$$\eta_P = R_{600} - R_{300} \quad (6)$$

$$\eta_Y = 0{,}48 \times (R_{300} - \eta_P) \quad (7)$$

$$\eta_A = R_{600}/2 \quad (8)$$

where

η_P is the plastic viscosity, in millipascal seconds;

NOTE Plastic viscosity is commonly known in the industry by the abbreviation PV.

η_Y is the yield point, in pascals;

η_A is the apparent viscosity, in millipascal seconds;

R_{600} is the dial reading at 600 r/min, in pascals (pounds per 100 square feet);

R_{300} is the dial reading at 300 r/min, in pascals (pounds per 100 square feet).

NOTE 1 To convert to CGS units of centipoise, 1 mPa·s = 1 cP.

NOTE 2 When calculating values in USC units, the yield point (in pounds per 100 square feet) is calculated as follows:

$$\eta_Y = R_{300} - \eta_P$$

7 Filtration

7.1 Principle

7.1.1 Measurement of the filtration behaviour and the filter cake characteristics of an oil-based drilling fluid are fundamental to the treatment and control of a drilling fluid, as are the characteristics of the filtrate, such as the oil, water or emulsion content.

7.1.2 Filtration characteristics of an oil-based drilling fluid are affected by the quantity, type and size of solid particles and emulsified water in the drilling fluid, and by properties of the liquid phase. Interactions of these various components can be influenced by temperature and pressure.

7.1.3 Filtration tests are performed at ambient (low) temperature and at high-temperature conditions to provide data for comparison purposes. Two filtration procedures are given: one for testing up to 175 °C (350 °F) and one for testing from 175 °C (350 °F) to 230 °C (450 °F). Use only the filtration equipment and procedure specified for the temperature required.

NOTE No low-temperature filtration test procedure for oil-based drilling fluids is specified herein, but it can be performed much like the water-based drilling fluid test provided in ISO 10414-1.

7.1.4 Either the 175 cm^3, 250 cm^3, or 500 cm^3 unit can be used for testing filtration up to and including 175 °C (350 °F). For testing above 175 °C (350 °F), only the 500 cm^3 unit shall be used. It shall be equipped with a thermocouple to measure the temperature of drilling fluid in the cell, and it shall use a porous filter media.

7.2 High temperature/high pressure test up to 175 °C (350 °F)

7.2.1 Apparatus

a) **High-temperature/high-pressure filter press**, consisting of:

1) **filter cell**, to contain working pressures up to 9 000 kPa (1 300 psi) at temperature;

2) **pressurized gas source**, such as carbon dioxide or nitrogen, with regulators;

NOTE Nitrogen is preferred.

3) **heating system**, to heat to 175 °C (350 °F);

4) **high-pressure filtrate collection vessel**, maintained at proper back-pressure (see Table 2) to avoid flashing or evaporation of the filtrate;

5) **filter cell**, containing a thermometer well, fitted with a removable end, a filter-media support and with oil-resistant seals.

NOTE Valve stems on each end of the cell can be opened or closed during the test.

CAUTION Not all manufacturers' equipment is capable of achieving the same temperatures and pressures. Rigid adherence to manufacturer's recommendations as to sample volumes, temperatures and pressures is essential. Failure to do so could result in serious injury.

Do not use nitrous oxide cartridges as pressure sources for HT/HP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas train carbonate analysis (see annex I).

Table 2 — Recommended minimum back-pressure

Test temperature		Vapour pressure		Minimum back-pressure	
°C	(°F)	kPa	(psi)	kPa	(psi)
100	212	101	14,7	690	100
120	250	207	30	690	100
150	300	462	67	690	100
Limit of "normal" field testing					
175	350	932	135	1 104	160
200	400	1 704	247	1 898	275
230	450	2 912	422	3 105	450

b) **Filter medium**[1], including Whatman No. 50 or S&S 576, or equivalent filter paper, for temperatures to 200 °C (400 °F).

c) **Mechanical or electronic timer**, with at least a 30 min interval.

d) **Thermometer**, with a range up to 260 °C (500 °F), and with a 12,5 cm (5 in) or longer stem.

e) **Long, slender graduated cylinder** (TC), with a capacity of 10 cm^3 or 20 cm^3.

f) **Graduated cylinder**, optional, (TC), with a capacity of 25 cm^3.

g) **Field mixer**, cup type, to operate at 10 r/min, 1 000 r/min and 15 000 r/min

h) **Ruler**, measured in millimetres, to measure filter cake thickness.

7.2.2 Procedure for temperatures up to 175 °C (350 °F)

7.2.2.1 Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 6 °C (10 °F) above the desired test temperature. Adjust the thermostat to the desired test temperature.

If the filtration unit is equipped with a thermocouple to measure drilling fluid temperature inside the cell (test temperature), then that temperature should be monitored and maintained during the filtration test. Results may differ from this standard procedure, which uses the cell wall temperature as the test temperature. Note if the thermocouple method was used.

7.2.2.2 Stir the drilling fluid sample for 5 min using the field mixer. Pour the fluid sample into the filter cell, leaving at least 2,5 cm (1 in) space in the cell to allow for fluid expansion. Install the filter paper in the cell.

7.2.2.3 Complete the assembly of the filter cell, with both top and bottom valves closed, and place it in the heating jacket. Transfer the thermometer from the heating jacket into the well of the filter cell.

7.2.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place. Be sure the collection vessel is completely free of water or oil.

7.2.2.5 Connect the regulated pressure source to the upper valve. Connect a similar regulated pressure source to the filtrate collection vessel, and lock these connections in place.

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1) Whatman No. 50 and S&S 576 are examples of suitable products available commercially. This information is given for the convenience of users of this Standard and does not constitute an endorsement by API of these products.

7.2.2.6 Keeping the two valve stems closed, adjust the pressure on the upper pressure regulator to 690 kPa (100 psi) higher than the minimum back-pressure value, as shown in Table 2. Set the lower regulator to the minimum back-pressure value, as shown in Table 2, for the test temperature. Maintain this pressure until the test temperature is reached.

NOTE If the time required to reach test temperature exceeds 1 h, the heater may be defective and the validity of the test is questionable.

7.2.2.7 When the sample reaches the selected test temperature, open the lower valve stem and immediately increase the pressure on the upper regulator to 3 450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ± 3 °C (± 5 °F) during the test. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

7.2.2.8 Collect the filtrate in the long, slender graduated cylinder (or optional graduated cylinder). Read the volume of the 30-min total (water plus oil) filtrate. Also read volumes of solid and aqueous phases, if present.

NOTE The long, slender glass cylinder allows more accurate detection and measurements of volumes of oil, water and solids in the filtrate. Heating of the cylinder near an emulsion interface can improve separation of water, solids and oil in the filtrate.

7.2.2.9 Immediately after collecting the 30-min filtrate, close the upper and lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 50 °C (125 °F). Keep the cell upright during cooling, depressurization and disassembly.

CAUTION Pressure in the filter cell can be dangerously high, even after the cell is cooled. Opening cell before pressure is released can result in injury.

7.2.2.10 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. Ensure that pressure is fully released before dislodging the cap. Carefully disassemble the cell.

7.2.2.11 Pour the liquid from the cell.

7.2.2.12 Remove the filter cake on the filter paper. Measure the filter cake thickness, at its centre, to the nearest millimetre.

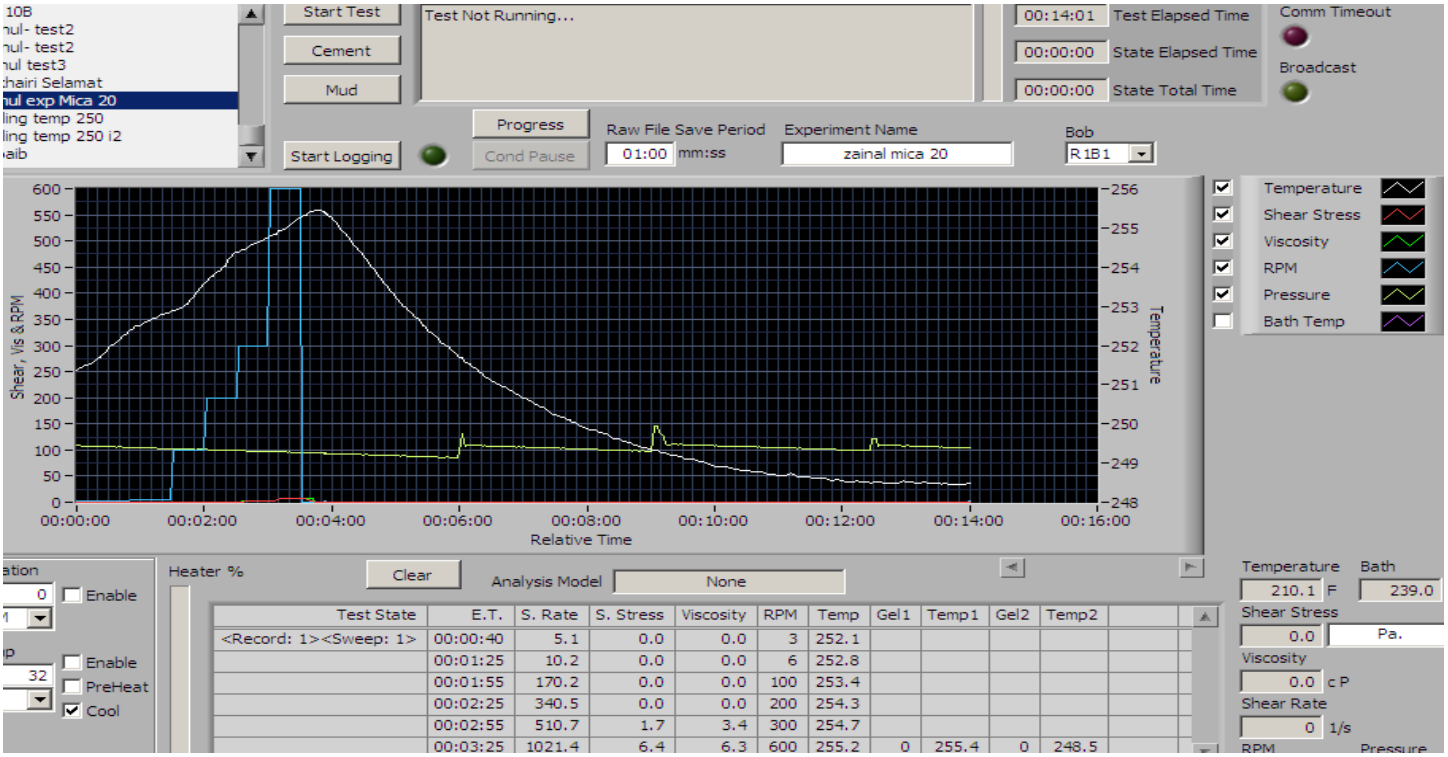
7.2.2.13 Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics. To minimize settling, the times for heat-up and cool-down should be minimized and the cake should be recovered and examined promptly.

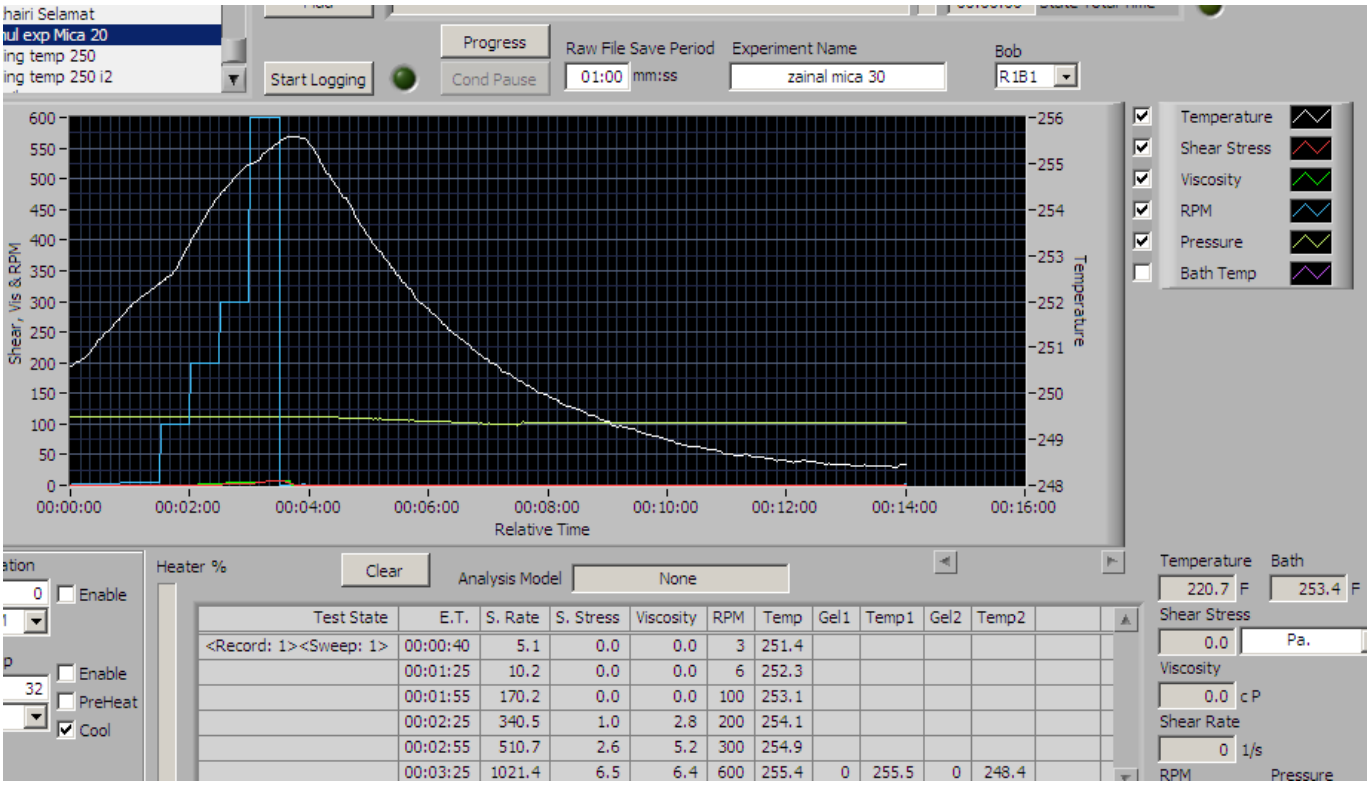
7.2.3 Calculation

7.2.3.1 The filtrate volume should be corrected to a filter area of 4 580 mm^2 (7,1 in^2). HT/HP filter cells usually have half the standard filter area or 2 258 mm^2 (3,5 in^2), thus double the observed volume before reporting.

7.2.3.2 Report the cake thickness to the nearest millimetre (32^{nd} inch).

APPENDIX 2: Example of Rheology Test Results Using HPHT Viscometer

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Bob
R1B1
Temperature
Shear Stress
Viscosity
RPM
Pressure
Bath Temp
Shear, Vis & RPM
Temperature
Relative Time
Heater %
Clear
Analysis Model
None
Enable
PreHeat
Cool
Test State
E.T.
S. Rate
S. Stress
Viscosity
RPM
Temp
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Temp1
Gel2
Temp2
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Temperature
Bath
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Shear Stress
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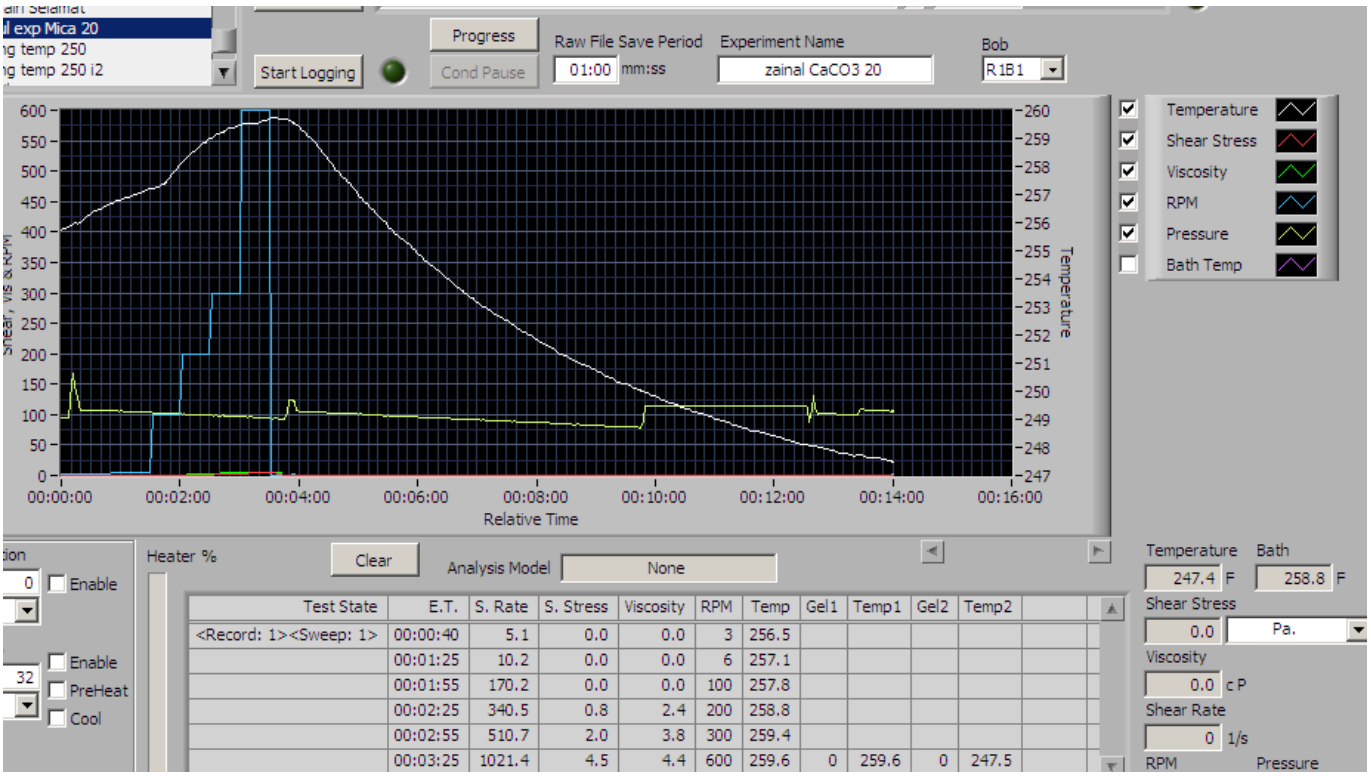
Pa.
Viscosity
0.0 c P
Shear Rate
0 1/s
RPM
Pressure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S. Stress
Viscosity
RPM
Temp
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Temp1
Gel2
Temp2
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00:02:55 510.7 2.0 3.8 300 259.4
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Temperature
Bath
247.4 F
258.8 F
Shear Stress
0.0
Pa.
Viscosity
0.0 c P
Shear Rate
0 1/s
RPM
Pressure