CHAPTER 1
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 drill string, and to control the subsurface pressure.

For drilling fluids perform these functions and allow drilling to continue, the drilling fluids must be present in the borehole. Unfortunately, undesirable formation conditions are encountered 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 still occurring. For this reason, much research has been dedicated to investigating various methods and equipment to address the scenarios from which fluid loss results.

Lost circulation is a term used to define the loss of drilling fluid into the formation voids instead of returning up to the surface. Loss circulation occurs when applying more mud pressure on the formation than it is strong enough to withstand, thereby mud flows into fracture that have been created. 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 occurs, the level of the drilling fluid in the annulus may drop and stabile 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.

1.2 Problem Statement

Mica used in the drilling fluid to ensure the control loss circulation in a wellbore formation. Micas' are usually imported from India. Having to import Mica is one of the reasons why drilling fluids are expensive. This project sees whether or not the Mica found in Malaysia is suitable as an additive for the drilling fluid.

It is best to be able to use the Mica found in Malaysia due to several economical reasons, the first is that the balance of payment of a country decreases. Money leaves the country when you import items, not only do you pay for the item; you have to also pay for the imports and tariffs that come with it. However if the Mica was produce locally, manufactures not only save money on the transaction and transportation cost, they also save money on paying import tariffs. Thus, making the much more attractive to be produce in a larger amount.

Now that we have seen the benefit it gives to the private company, we will see how much it helps the economy of our country as a whole. The campaign "Belilah Barangan Buatan Malaysia" will now be applicable in a larger framework.

Due to the benefit of both the private companies and our country, I am determined to study if the Mica available in Malaysia are suitable for forming drilling fluids.

1.3 Objectives

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

- Develop LCM from local Mica (Malaysia)
    - Formulate oil based mud with LCM chosen and testing with current technology
    - Evaluate the efficiency and compatibility of Malaysian.

1.4 Scope of Study

The research will involve in the understanding of LCM in drilling fluid. The study of this project can be broken down to the identification of the appropriate LCM and the method of studying and evaluating effectiveness of LCM in oil-based drilling fluid.

The scope of study mainly investigates the fluid loss properties of the Malaysian Mica. The study will be divided into two stages; the first stage involves researching the basic properties of the Mica and determining an ideal formulation to be developed. The second stage will focus on experimental work in the lab, using the 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 majoring since loss circulation is one of the focus areas in drilling process. LCM study as the technology of using Malaysian Mica instead of importing from overseas as LCM is not yet been used in the industry. The source of Malaysian Mica is from Tapah, Perak. 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 new LCM.

The project is feasible since it is within the scope and time frame. The first step in this project will be getting an introduction to the related topics by reading books, journals and research papers. Research has been done in order to understand better on loss circulation material and how to go about the experiment work on the fluid loss factor. The research approximately took 1 month time. All the involved variables was identified and understood to make the desired drilling fluid. That process took about 2 months to complete. Once the desired drilling fluid is formed, the lab work begins to find the suitable formulation to test the Malaysian Mica. 1 month was needed to perform that process and finally it took about 1 month to analysis the results obtained from experiment.

CHAPTER 2

LITERATURE REVIEW AND THEORY

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 the journal Effect of Material Type and Size Distribution on Performance of Loss/Seepage Control Material. In general, four types of formations are responsible for lost circulation which is natural fractured formations, cavernous formations, highly permeable formations or unconsolidated formations and induced fracture formations. Even with the best drilling practices, circulation losses can occurs in varying degrees and the severity of these losses is an indicator of the mud loss to the formation. Loss zones can be classified as:

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

Table 1: Loss Zone Classification (Ali A. Pilehvari 2002)

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). Based on this paper, 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.

Besides that, the journal entitled Effect of Material Type and Size Distribution on Performance of Loss/Seepage Control Material by Pilehvari A. and Nyshadham R. on 2002 has been reviewed. A wide variety of materials have been used to combat lost circulation over the years. The choice of lost circulation material to use in a given case is influenced to some degree by cost and availability in a given drilling area. According to the journal, for the purposes of classification, LCM's can be divided into fibers, flakes, granules and mixtures. The fibrous LCM's are used mainly in drilling muds to lessen the mud loss into large fractures or vugular formations, whereas flaky type LCM's can plug and bridge many types of porous formations to stop the mud loss or 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 to build a bridge having decreasing permeability, as it is being laid down. 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.

The journal entitled High Fluid Loss, High Strength Loss Circulations Material by Mark W. Sanders, Jason T. Scorsone and James E. Friedheim published in 2010 was also reviewed. This paper is describes and discussing 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 levels 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.

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.

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

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.

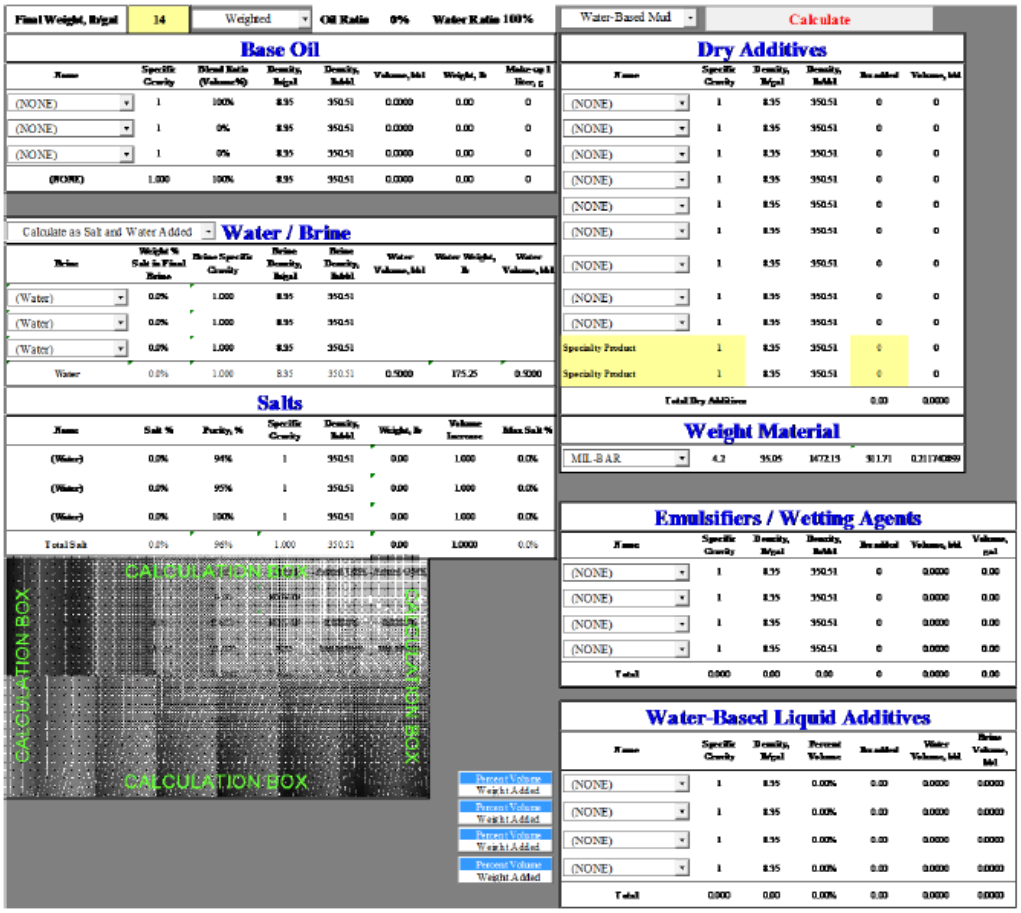

Figure 1 : Mud Formulator Spreadsheet

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.

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 2 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.

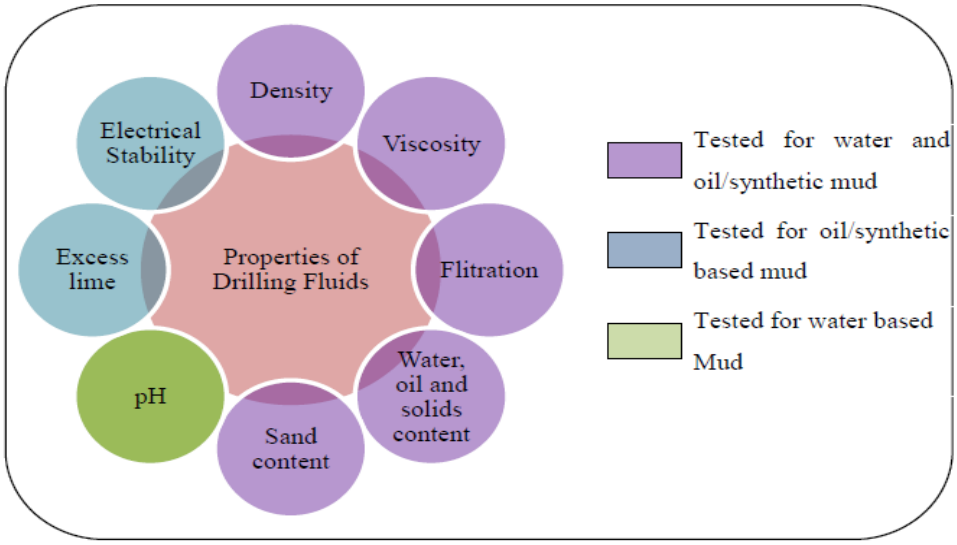

Figure 2 : Properties of Drilling Fluids

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 3.

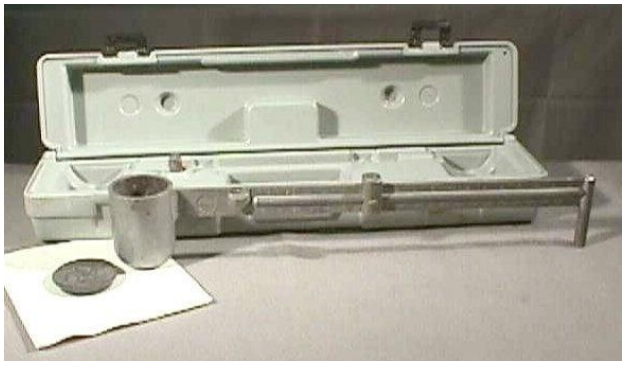

Figure 3 : Regular Mud Balance

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 9 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 4 : 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 5.

Figure 5 : 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 6.

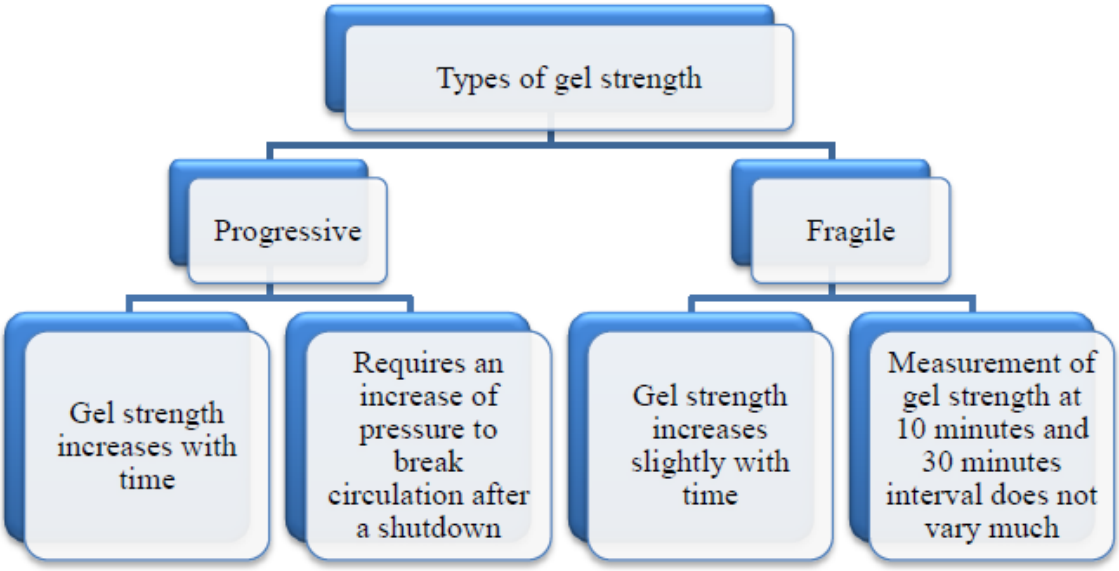

Figure 6 : Types of Gel Strength

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.

There are two types of test that the author has utilized all the way through internship which are standard API low-temperature/low pressure test and high temperature/high pressure (HTHP) test. The standard API low-temperature/low-pressure shown in figure below uses the standard API filter press pressured to a differential of 100 psi.

Figure 7 : Standard API Filter Press

The standard API low-pressure filter press consists of a cylindrical cell three inches in I.D. and five inches high to place the fluid. The bottom of the cell is fitted with a sheet of Whitman No. 50 filter paper. Pressure is applied to the top of the cell at 100 psi. The filtrate which known as API filtrate is collected over a period of 30 minutes and recorded in cubic centimetres. Filter cake thickness usually measured in 1/32 of an inch.

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 overseas mica will be done in oil-based mud. The main criteria for evaluate the LCM is through running the loss circulation experiment. Besides that several studies and experiment conducted on the properties of the LCM such as mud density, rheology of mud, filtration and thickness of mud cake.

There are 2 types of experiments are being carried out in this project. First, is to test the physical properties of the Malaysian and India Mica. Physical properties of the materials that are tested:-

1. Mineralogy of the material (XRD machine)
2. Particle Size Distribution (Sieving method)
3. Particle Shape (Using SEM)

Second experiment that is carried out is upon mixing the mud using the Malaysian Mica(Appendix 1).

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 oil based mud system to analyze the LCM applicability and effectiveness - Tools required (multimixer)
Testing mud plus	- Prepare oil based mud with current uses Mica - Measure all the properties of with Malaysian Mica

industrial used LCM ★		
Testing mud plus new LCM ★	Properties	Tools Required
	Density	Mud Balance
	Viscosity	March Funnel
	Electric Stability	ES Meter
	- Plastic Viscosity - Gel Strength - Yield Point	FANN (Model 35A) 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	

Table 2: Activities and Description

3.2 Project Activities

3.2.1 Sample Collection

A field trip is conducted on 24th November 2011 to Bidor, Malaysia. This field trip is purposely to identify the source of Mica and the type of Mica which is extracted by KAOLIN(M), Bidor, Malaysia. Sample preparation are done in the quary itself. The sample is in the form of powder.

3.2.2 Sample Processing

Figure 9: Processes of Mica

The quarry which is operated by KAOLIN(M) is situated at Bidor, Malaysia. The Mica is being extracted from the ground. By using bulldozer, the Mica is extracted from the ground and transported by a lorry to another place. After it is gathered, Mica will undergo sieving and washing process to remove impurities. After that, Mica is gathered in a pond. The wet Mica in the pond will go through Filter press operation where, the wet mica will be pressed to remove the water. Finally, Mica will be dried and packed according to specifications.

3.3 Gantt Chart and Key Milestone

Figure 3 and 4 below shows the schedule and timeline of this project carried out for the period of 8 months. It consists of two parts which was divided into two semesters called Final Year Project I and Final Year Project II and was 14 weeks each.

Legend:

Processes

Milestones

No	Detail / Week	1	2	3	4	5	6	7	Mid Semester Break	8	9	10	11	12	13	14
1	Selection of Project Topic															
2	Study on Lost Circulation Material															
3	Submission of Extended Proposal															
4	Proposal Defense															
5	Study on Malaysian Mica and drilling fluid															
6	Introduction to drilling fluid equipments and lab															
7	Submission of Interim Draft Report															
8	Submission of Interim Report															

Table 3: Gantt chart for the first semester project implementation

No	Detail / Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Studying Malaysian Mica & learning of drilling fluid																
2	Submission of Progress Report									★							
3	Physical, chemical and drilling fluid experiments																
4	Poster Submission												★				
6	Submission of Dissertation (softbound)													★			
7	Submission of Technical Paper														★		
8	Oral Presentation															★	
9	Submission of Dissertation (hard bound)																★

Mid Semester Break

Table 4: Gantt chart for the second semester project implementation

CHAPTER 4
RESULTS AND DISCUSSION

Results can be divided into 2 parts:-

a. Physical and Chemical properties of the materials
    a. XRD test
    b. SEM(scanning electron Microscope)
    c. Particle Size Distribution

b. Properties of materials in drilling fluid
    a. Rheology
    b. Plastic Viscosity
    c. Yield Point
    d. Gel Strength
    e. Electric Stability
    f. Fluid Loss

4.1 Physical and chemical properties

Firstly, physical properties of Malaysia and India Mica are tested to ensure that the correct samples are being used in the project for comparison purpose. Results of the tests are shown below:-

4.1.1 XRD Results

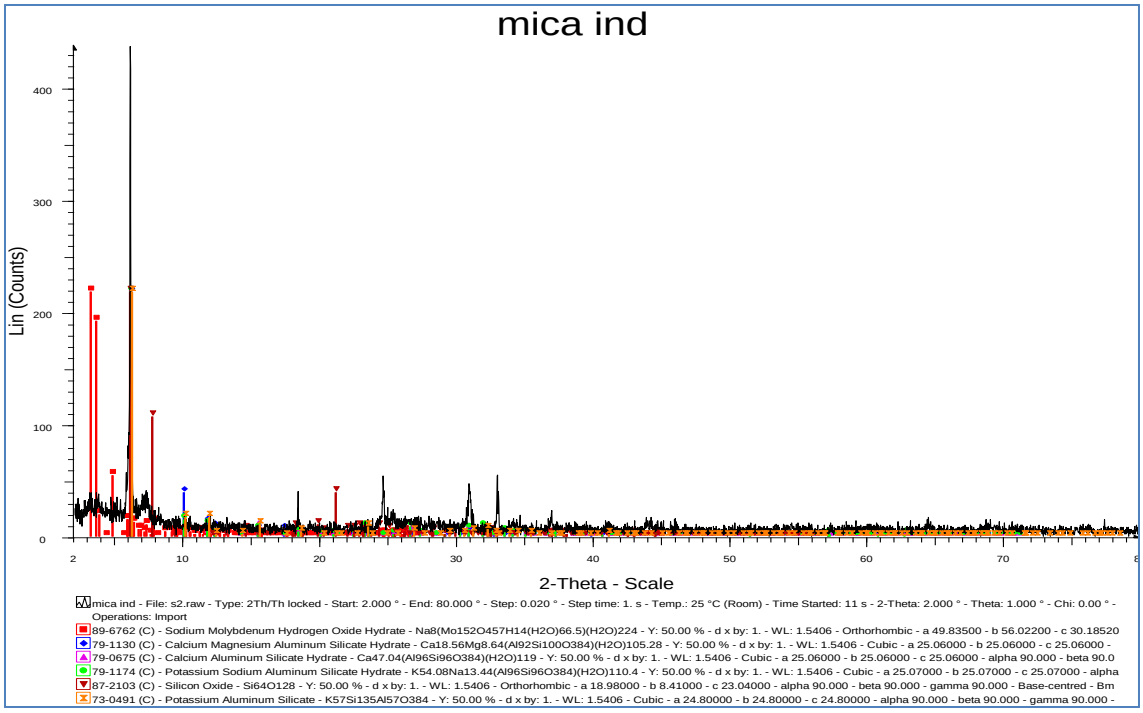

Figure 10: XRD result on Indian Mica

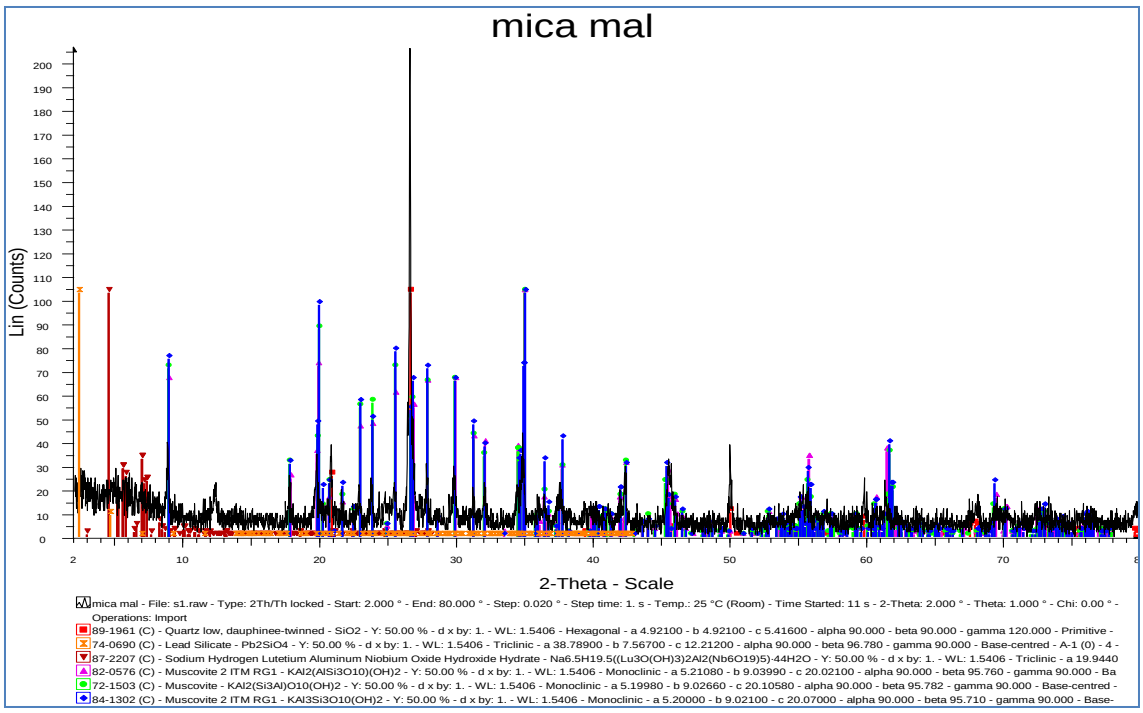

Figure 11: XRD result on Malaysian Mica

4.1.2 Particle Size Distribution(RO-TAP Siever and Malvern Mastersizer 2000)

a. Indian Mica

Sieve no.	screen size, µm	Screen opening, mm	blank wt, g	final wt, g	residue, g	Cummulative wt, g	Cummulative wt%, g	wt%, retained
20	853	0.853	604.9	606.02	1.12	0	0	5.6
40	422	0.422	563.5	565.86	2.36	0.3	1.5	11.8
60	250	0.25	552.5	556.92	4.42	4.72	23.6	22.1
100	150	0.15	541.2	548.52	7.32	12.04	60.2	36.6
200	75	0.075	506.6	510.95	4.35	16.39	81.95	21.75
400	37	0.037	338.6	339.07	0.47	16.86	84.3	2.35
	0	Pan	486.1	486.1	0	16.86	84.3	0
					20			100
				Sample Weight, g =	20			

Table 5: Partile Size Distribution for Indian Mica

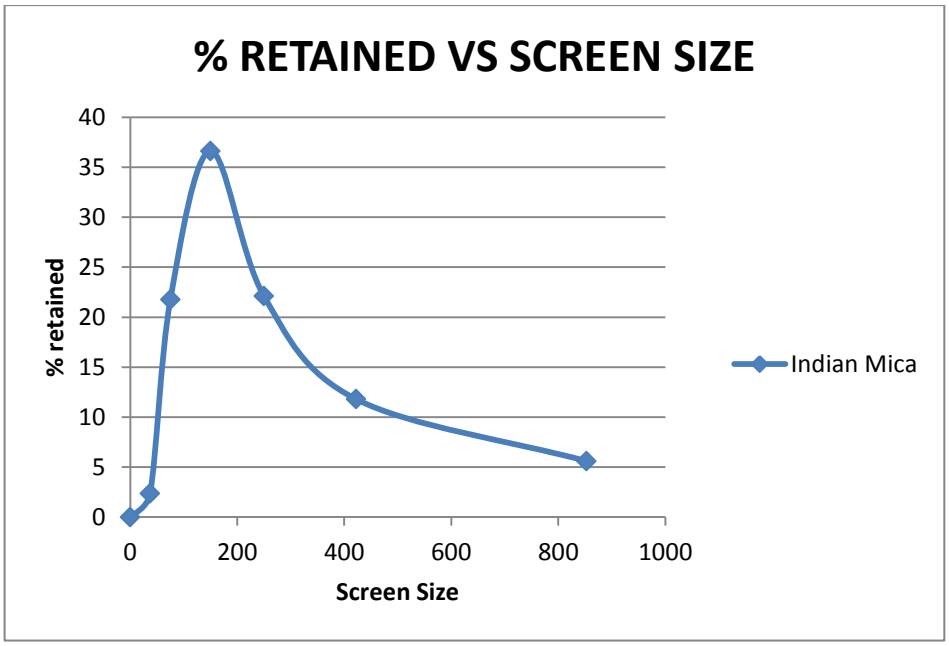

Figure 12: PSD Graph for Indian Mica

b. Malaysian Mica

Sieve no.	screen size, µm	Screen opening, mm	blank wt, g	final wt, g	residue, g	Cummulative wt, g	Cummulative wt%, g	wt%, retained
20	853	0.853	604.9	604.9	0	0	0	0
40	422	0.422	563.5	563.8	0.3	0.3	1.5	1.5
60	250	0.25	552.5	552.8	0.3	0.6	3	1.5
100	150	0.15	541.2	541.8	0.6	1.2	6	3
200	75	0.075	506.6	510.1	3.5	4.7	23.5	17.5
400	37	0.037	338.6	344.5	5.9	10.6	53	29.5
	0	Pan	486.1	495.5	9.4	20	100	47
					20			100
				Sample Weight, g =	20			

Table 6: Particle Size Distribution for Malaysian Mica

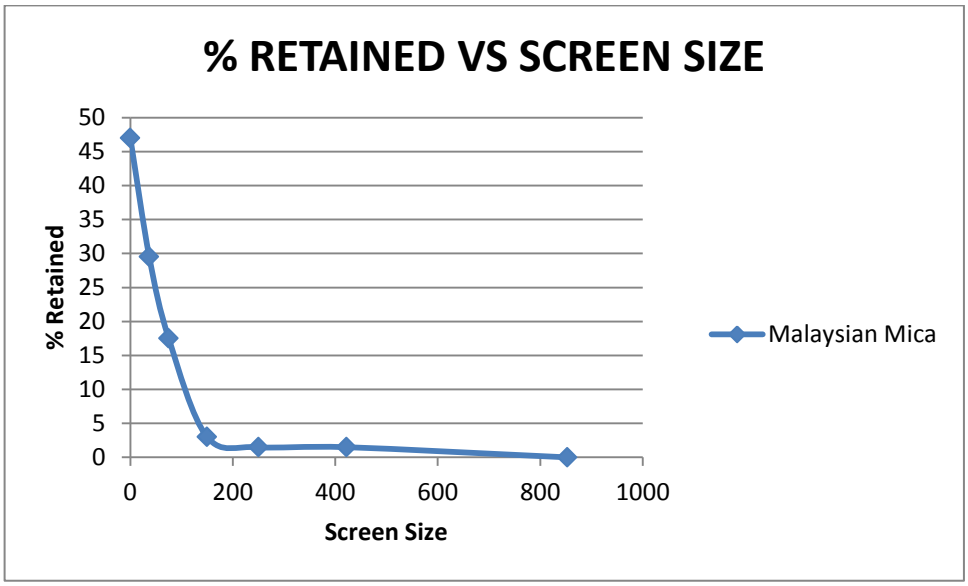

Figure 13: PSD Graph for Malaysian Mica

Based on the table, 47% of the sample falls on the pan. This shows that the sample is smaller than 37 micron. To determine the particle size distribution of this sample, MALVERN MASTERSIZER 2000 has been used. The results are illustrated in the next page:-

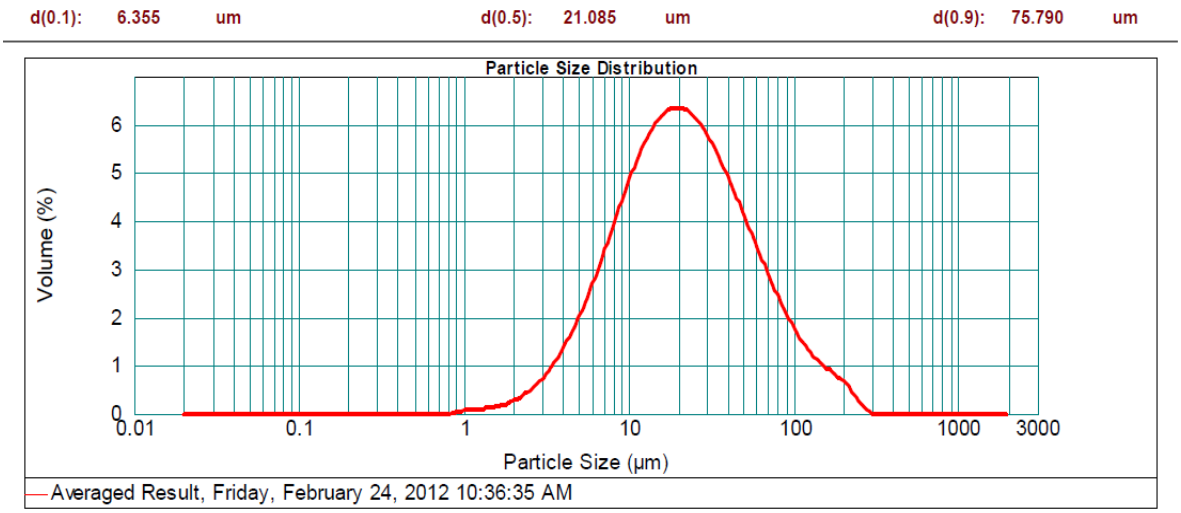

Figure 14: PSD Graph for Indian Mica using Malvern

4.1.3 Scanning Electron Microscope(SEM)

a. Indian Mica

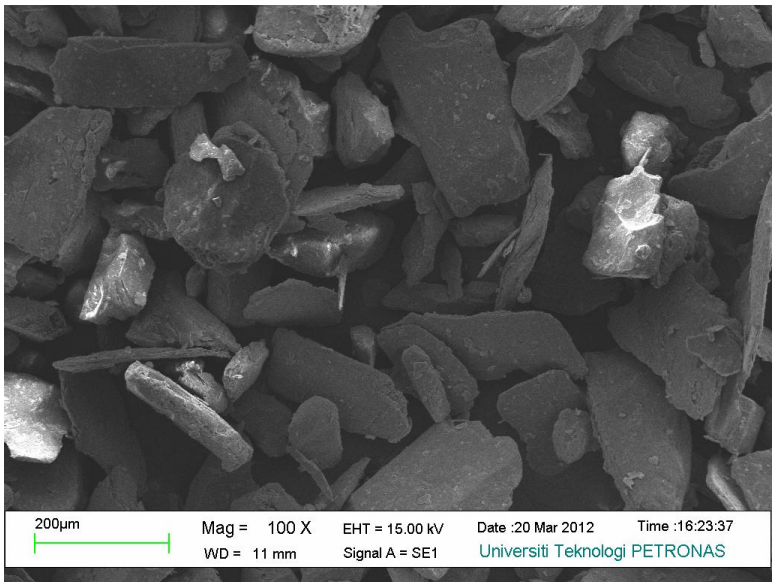

Figure 15 : Indian Mica at 100X magnification

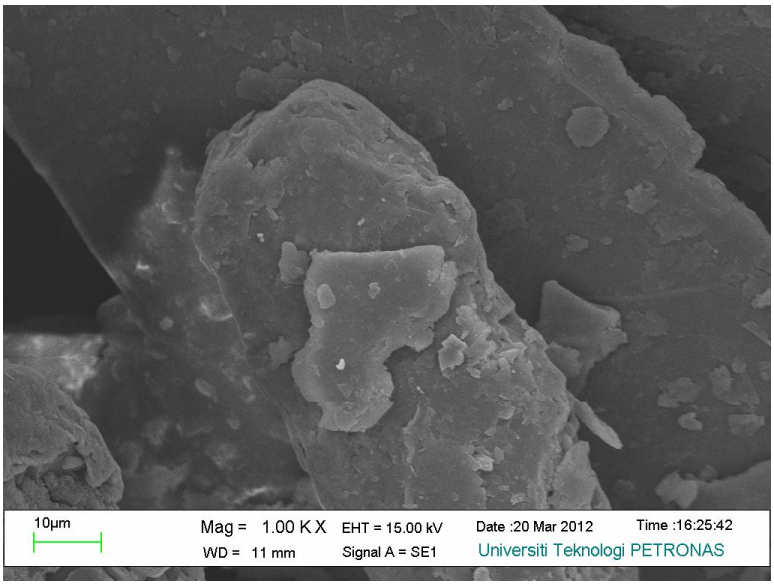

Figure 16 : Indian Mica at 1000X magnification

b. Malaysian Mica

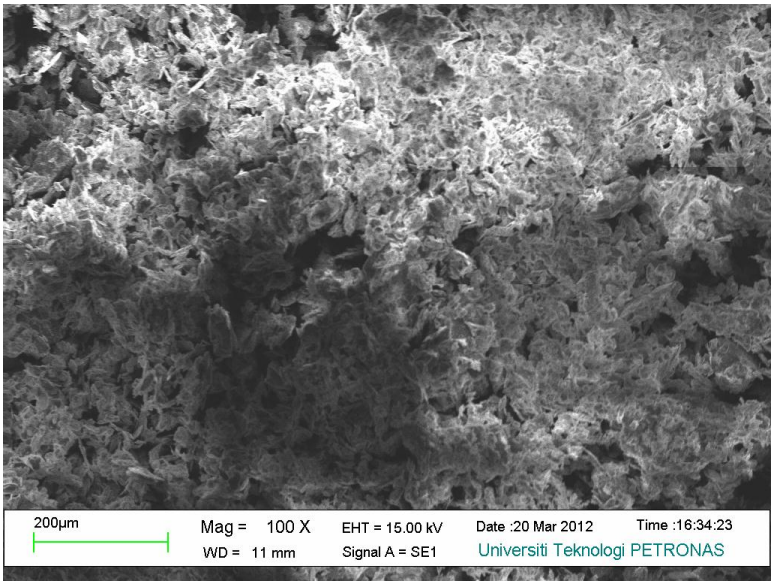

Figure 17 : Malaysian Mica at 100X magnification

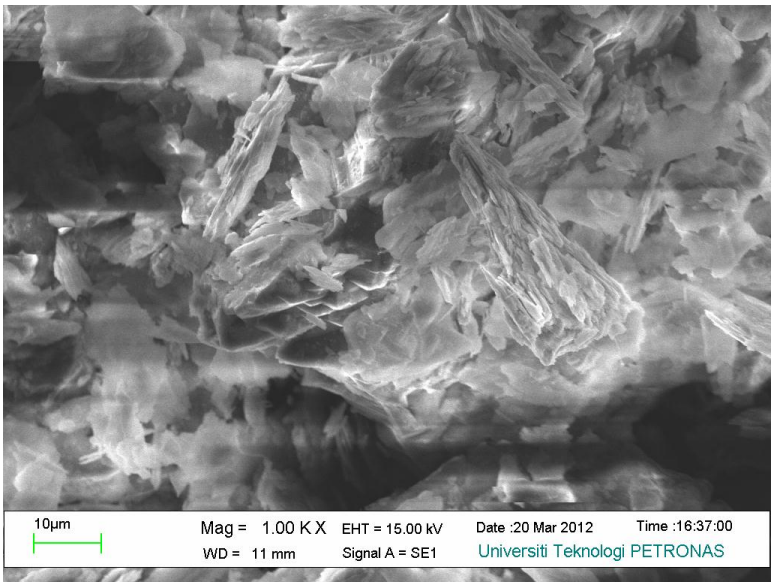

Figure 18 : Malaysian Mica at 1000X magnification

4.2 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 and Indian Mica is from Mica Biotite with general chemical formula of K(Mg,Fe)3AlSi3O10(F,OH)2. Based on these results, direct comparison of Indian Mica and Malaysian Mica cannot be made since the materials are different.

The average particle size distribution for Indian Mica is 150 micrometer whereas the average particle size distribution is 21.085 micrometer.

Based on the Scanning Electron Microscope, the both the Micas are fairly flaky and layered. This is assumed due to the sedimentation process.

In conclusion, to compare both LCMs', the basic criteria is to have the same particle size distribution (PSD). In our case, the PSD is far different. Since, direct comparison cannot be done in the project. The author has decided to test the compatibility of Malaysian Mica to be used in certain formations.

Based on the Scanning Electron Microscope, the both the Micas are fairly flaky and layered. This is assumed due to the sedimentation process.

4.3 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). Sample A actually is the base (OBM without LCM) case for this experiment. Other drilling mud samples were prepared in order to measure the change in properties of the mud. Below are the formulations of the mud that have been tested.

Formulations	**A**	**B1**	**B2**	**B3**	**B4**	**B5**
SARAPAR 147, lb/bbl	161.9	161.9	161.9	161.9	161.9	161.9
Water,lb/bbl	54.5	54.5	54.5	54.5	54.5	54.5
CaCl2, lb/bbl	16.3	16.3	16.3	16.3	16.3	16.3
ECCO-MUL E, lb/bbl	10	10	10	10	10	10
CARBO-GEL II, lb/bbl	8	8	8	8	8	8
CARBO-TROL A9, lb/bbl	7	7	7	7	7	7
Lime, lb/bbl	7	7	7	7	7	7
API Barite, lb/bbl	239.3	239.3	239.3	239.3	239.3	239.3
Malaysian Mica, lb/bbl	0	10	20	30	40	50
RESULTS						
Rheology Temperature	***120°F***	***120°F***	***120°F***	***120°F***	***120°F***	***120°F***
600 rpm	67	72	76	81	85	88
300 rpm	40	43	45	48	50	52
200 rpm	30	32	34	37	39	40
100 rpm	20	21	22	23	23	24
6 rpm (6 - 10)	7	7	8	8	9	9
3 rpm	6	6	7	8	8	8
Plastic Viscosity, cP (ALAP)	27	29	31	33	35	36
Yield Point, lb/100 ft^2 (12 - 16)	13	14	14	15	15	16
Gels, 10 sec	8	9	9	10	10	11
Gels, 10 min	13	13	13	13	13	14
Electrical Stability, volts **(** > 400)	617	694	790	824	856	889
HPHT at 250°F, mL (filter paper) (< 8.0)	6.8	6.4	6.2	6.0	6.4	6.6
PPA at 250'F and 500psi, mL						
Ceramic Disk (P/N: 170-53-3) (20 micron)	6.8	6.6	6.2	5.8	6.2	6.6
Ceramic Disk (P/N: 170-51) (40 micron)	7.6	7.2	7.0	6.8	7	7.4

Table 7: Mud formulations and Results

4.4 Discussion on Properties of materials in drilling fluid

- **Mud Weight**

The major determinant of mud weight in a drilling fluid is API Barite. As the amount of API barite is increased, the mud weight increases as well. Density is the most important mud property affecting penetration rate. For any given formation pressure, the higher the density, the greater will be the differential pressure. Selection of mud weight is very dependent on the differential pressure of the well bore and other parameters. Less than sufficient mud weight in a formation may cause lost circulation. So, the mud weight must be sufficient to confine the formation fluid but not great enough to cause other problems such as stuck pipe. In the experiment, the mud weight chosen to be set is 12 ppg since the recommended the amount of mud weight in the field is around 8 to 12 ppg based on Scomi Oiltools manual handbook.

- **Plastic Viscosity**

Viscosity is the term that describes resistance to flow. So high force need to be applied for move the high viscosity liquids, whereas low viscosity fluids flow relatively required less force and easy to move. Plastic viscosity is a function of solids concentration and shape. 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 as the amounts of LCM are increased, the value of PV also increased. In short, PV should be as low as possible in order to have low pumping rate for mud circulation.

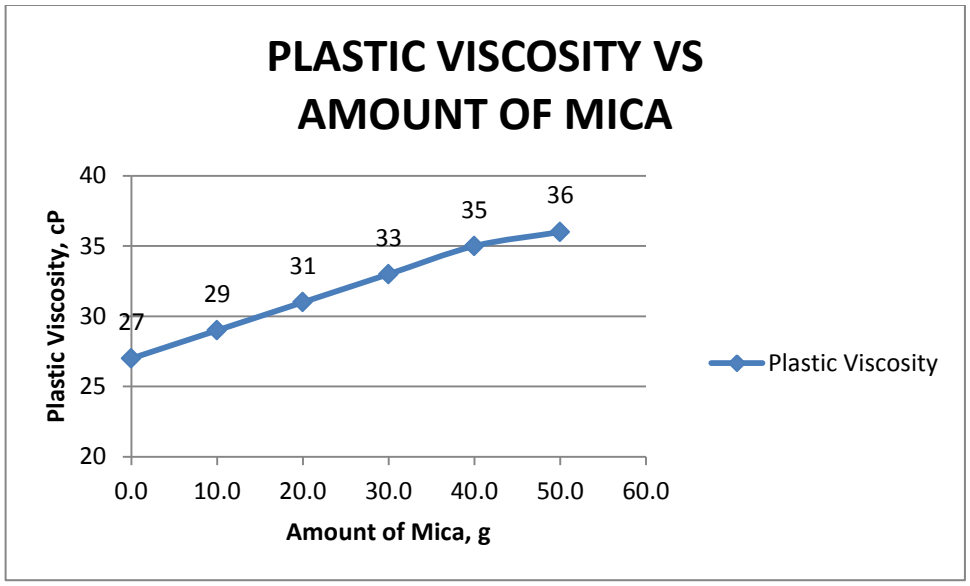

Figure 19: Plastic Viscosity VS Amout of Mica

- **Yield Point**

Yield point is the attractive force in the mud under flow conditions. The magnitude of these forces will depend on the type of their solid present, the ion concentration in the liquid phase (Growcock F, 2005). From the figure below which represents by the mud plus LCM, the value of yield point for mud increasedas the concentration of LCM increased.

The value of yield point will increase as the amount of solid increased. It is similar compared to the actual results.

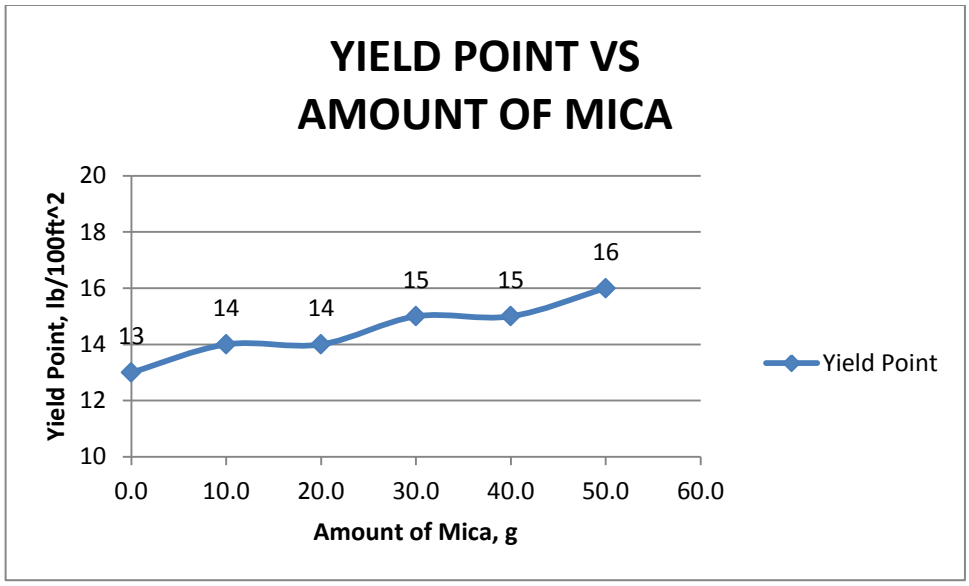

Figure 20: Yield Point VS Amout of Mica

- **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. In short, gel strength 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 forces needed to break the circulation are low over time.

As both the graph shown, they illustrate that the values obtained tend to decrease as the amount of LCM is increased. In general, high gel strengths are not desirable and can even be dangerous. However, the concentration of Malaysian Mica does not give significant change to the gel strength reading.

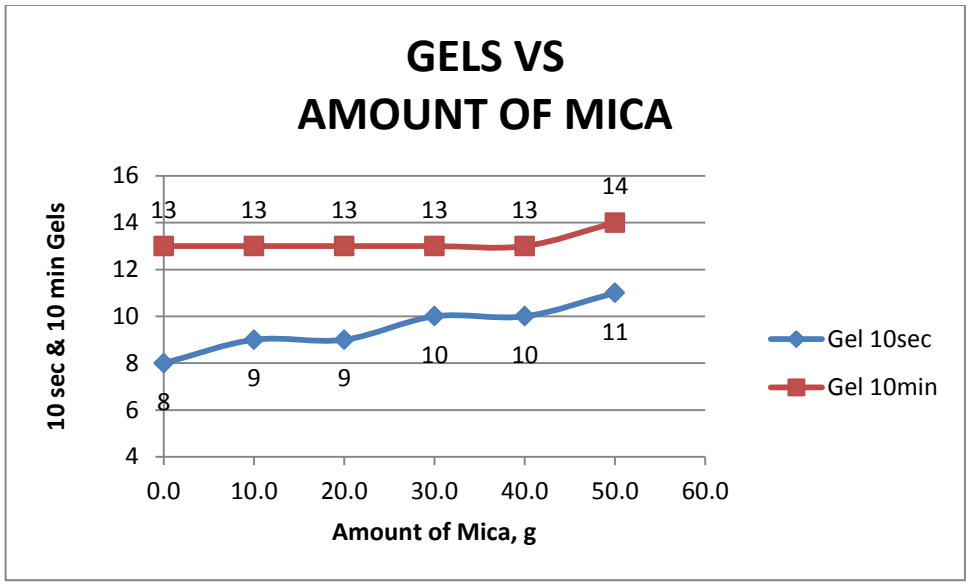

Figure 21: Gel Strength VS Amout of Mica

- **MUD CAKE AND FILTRATE**

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. When the LCM concentration is increased, the filtrate volume will reduce until one point, and then it will start increasing after reaching the optimum point due to excessive Mica in the drilling fluid. Since our Mica is about 21micronmeter in average. It works better in 20 micron ceramic disk compared to 40 micron.

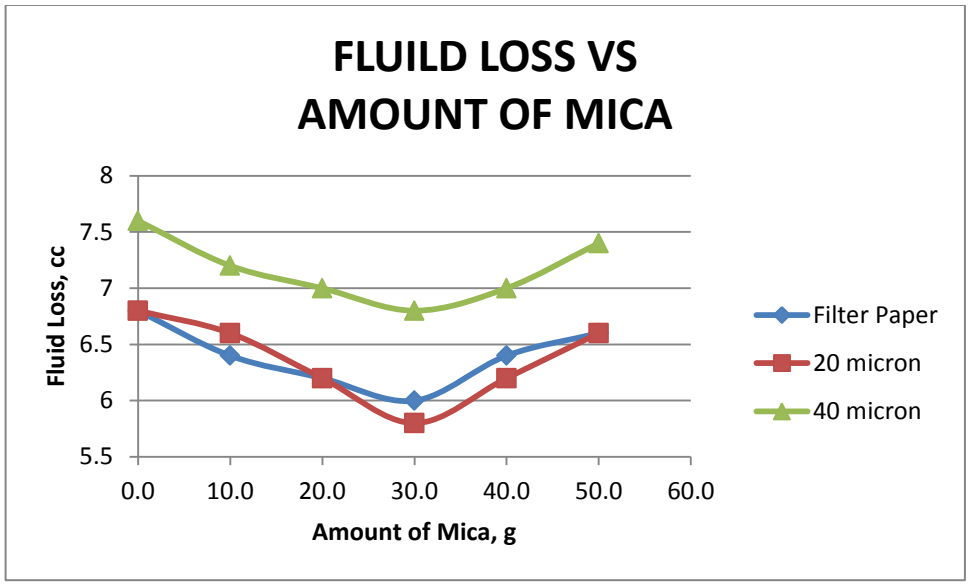

Figure 22: Fluid Loss VS Amout of Mica

Based on the results and discussions above, the optimum concentration of Malaysian Mica is 30g. So, this concentration was chosen to be compared to the formulation without to evaluate the properties Malaysian Mica as LCM. The results are shown below:

Formulations	A	B3
SARAPAR 147, lb/bbl	161.9	161.9
Water,lb/bbl	54.5	54.5
CaCl2, lb/bbl	16.3	16.3
ECCO-MUL E, lb/bbl	10	10
CARBO-GEL II, lb/bbl	8	8
CARBO-TROL A9, lb/bbl	7	7
Lime, lb/bbl	7	7
MIL-BAR, lb/bbl	239.3	239.3
Malaysian Mica, lb/bbl	0	30
Results		
Plastic Viscosity, cP (ALAP)	27	33
Yield Point, lb/100 ft2 (12 - 16)	13	15
Gels, 10 sec	8	10
Gels, 10 min	13	13
Electrical Stability, volts (> 400)	617	824
HPHT at 250°F, mL (filter paper) (< 8.0)	6.8	6.0
PPA at 250'F and 500psi, mL		
Ceramic Disk (P/N: 170-53-3) (20 micron)	6.8	5.8
Ceramic Disk (P/N: 170-51) (40 micron)	7.6	6.8

Table 8: With and Without Mica mud formulations and Results

Based on the results, viscosity is increased about 22.2%, the yield point is increased about 15.4%, the gel strength is around the same, and the amount of filtrate is decreased by 11.8% for filter paper, 14.7% for 20 micron ceramic disk and 10.5% for 40 micron ceramic disk. In short, the properties of Malaysian Mica as LCM can be used in the drilling fluid industry depending to the type of formation problem.

CHAPTER 5
CONCLUSION

5.1 Conclusion

The aim of the project to identify the effectiveness of Malaysian Mica as a Loss Circulation Material (LCM) is achieved for certain formations. Lost circulation material is very important in preventing mud losses to the formation. Even with the best drilling practices lost circulation still occur. Thus it is essential to put lost circulation material to minimize mud losses to the formation and Malaysian Mica was chosen to be the lost circulation material in this project.

Overall, it is justified that Malaysian Mica is appropriate and can be used as a new LCM because of its availability, cost effective, and effective in combating loss circulation problem for 20micron and 40 micron formations.

5.2 Recommendation

However, 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 ultra-fine Malaysian Mica only. 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. 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.

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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 (32nd inch).