ABSTRACT

Wellbore stability in shale formation has long been a significant issue in petroleum drilling industry for over century. The major cause of wellbores stability problem in shale formation facing by many drilling engineers actually originated from interaction between water-based muds with shale. The adsorption of water particle by shale will force the clay mineral to swell. This particular phenomenon of shale swelling will lead to various stability problems such and eventually lead to wellbore failure. In this project, a study on an improved water-based mud (WBM) containing nanoparticle (nanosilica) and its performance in maintaining wellbore stability in shale formation will be showed.

This project will show the process of producing nanosilica from siliceous sand. Due to its commercial availability, this nanosilica can be easily engineered to meet the specification of the formation especially the particle size. Characterization of the nanosilica was completed by using Transmission Electron Microscope (TEM). The analysis on nanosilica particle size shows that it is suitable for providing plugging mechanism in WBM. The use of nanoparticle such as nanosilica to decrease the water adsorption of shale by physically plugging nanoscale pores holds the potential to remove a major hurdle in confidently applying water-base mud in shale formation.

In this project, rheological properties of designated WBM are determined to analyze the relationship between nanosilica concentration and various rheological properties.

Ultimately, this project includes the study of fluid loss and plugging effect with respected to concentration of nanosilica in WBM by using laboratory experimental approach. Therefore, a deeper understanding on wellbore stability in shale formation can be achieved through this project.

ACKNOWLEDGEMENT

In completion of this Final Year Project, I am honored to take this opportunity to express my deepest appreciation to all parties that involved in making this project as an important and great achievement for me. Throughout this project, I have learned a lot of new things and gained so much experience that will be a enormous value for me in the future.

First and foremost, I would like to thank UTP‟s Petroleum Engineering Department for providing me guidance to complete the project and in the same time organizing the project activities for me. With their strong provision and advice, I was able to strategize and conduct my project very well.

Special thanks should go to my Final Year Project supervisor, Dr. Sonny Irawan who becomes the most important person to teach and guide me in conducting this project. His robust support and willingness to share his knowledge and wide expertise has given me a very important experience in this project study.

Besides that, I would like to thank the lab assistants who had helped me to complete the project especially in conducting the laboratory work and field work. I truly appreciate their help and their sharing of information in order for me to execute the experiment well.

Finally, my deepest appreciation goes to my family and my friends in UTP for their continuous support and inspiration which have enabled me to do my best for this project. I hope that after this project completion, all the findings and knowledge that I have shared through this dissertation would contribution for everyone in the study field.

TABLE OF CONTENT

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Shale can be defined as fine-grained, clastic sedimentary rock which consists of mud that is a mixture of clay minerals and tiny fragments of other minerals such as quartz and calcite. Shale gas is natural gas formed as a result of being trapped within shale formations. Most shales are not commercial sources of natural gas since shale normally have low permeability to allow significant fluid flow to a well bore. Therefore shale gas is one of unconventional sources of natural gas. Shale has low matrix permeability, so gas production in commercial quantities requires fractures to provide permeability. Shale gas has been produced for years from shale with natural fractures. However, the shale gas has started to boom in recent years due to modern technology in hydraulic fracturing (fracking) to create extensive artificial fractures around well bores.

Shale instability problems have perplexed the petroleum industry for many years. Many types of wellbore stability problem such as hole enlargement, hole reduction, drilling fluid loss into the formation, poor hole cleaning, and well control problem can occur in shale formation. All of the mentioned problems could lead to higher drilling cost than what is expected. Therefore in order to address this matter, this project will conduct a study on the use of an improved water-base drilling fluid (WBM) that is simple in formulation and maintenance that shows excellent rheological properties, maintains borehole stability, and a good environmental profile

The main factor which causes shale failure is redistribution of in-situ stress which then surpasses the shear or tensile strength of the rock. Theoretically, before the drilling process, a field has an initial in-situ stress state. When a hole is drilled, this process will alter the stress distribution by removing the rocks that previously filled the borehole. As a result, more stress is concentrated around the wall of the wellbore.

This is when drilling fluids play an important role in order to maintain the stability of the wellbore. The proper weight of the drilling fluids is crucial to balance the hydrostatic pressure inside the borehole with formation pressure.

However wellbore stability problem is not that simple in shale formation. The primary cause of wellbore instability in shale formation is the interaction of water base mud with shales. This will usually cause swelling problem as shale react with water particle. The movement of water and ions into or out of shale can result in large changes in pore pressure in the vicinity of the wellbore, potentially leading to wellbore failure. Therefore an improved design of water base mud is required to reduce the swelling problem and thus maintain borehole stability. In this project, nanoparticle (nanosilica) is used as additive to enhance the water base mud performance in shale formation.

1.2 Problem Statement

The wellbore stability issues have been a main subject in drilling engineering especially in shale formation. Nowadays, shale formation has been known for its instability problem. Wellbore stability problem such as swelling, shrunken hole, stuck pipe, and poor borehole cleaning tend to occur in shale formation. Without a proper analysis for avoiding or minimizing those stability problems, drilling operation in shale formation may cost more than budgeted.

Shale gas is a natural gas produced from shale formations that typically act as both a reservoir and source rock for the natural gas. Precisely, shale is a clastic sedimentary rock that is composed of clay-sized particles. The very fine, sheet-like formation cause several wellbore stability problems especially during drilling operation. When drilling in overbalanced conditions, mud pressure will penetrate progressively into the formation. As shale formation have very low permeability, only small volume of filtrate penetrates into the wellbore. This phenomenon leads to an increase in pore fluid pressure around the wellbore and consequently affects the stability of the wellbore.

Usually, synthetic-base mud (SBM) is frequently preferable system for shale formation due to its stability with the formation and the higher lubricity during horizontal drilling. However, environmental agencies, from EPA to local authorities,

are ensuring that the operators in the gas play areas regulate their drilling operation by adhering to certain protocols and activities. SBM could be targeted next. Therefore, this project will developed a system with goals of low cost, freshwater-base fluids that avoid the use of chlorides (which are considered not environmentally friendly), and improve the stability of wellbore in shale gas well.

When considering the used of water-base mud (WBM) in shale region, wellbore stability problem such as osmotic potential effect is the first thing that comes to mind. Therefore, in order to ensure the successfulness of the project, some problematic questions need to be answered:

- How to design an improved WBM that is simple in formulation and maintenance shows excellent rheological properties, maintain wellbore stability, and good environmental profile.
- How to significantly reduce the invasion of mud filtrate by using WBM system in shale formation and thus reduce the shale stability issues.

1.3 Objectives

The following are the objective of the project that should be achieved:

- To produce an improved WBM that is simple in formulation and maintenance shows excellent rheological properties, maintain wellbore stability, and good environmental profile.
- To determine the optimum concentration of nanosilica in the designed WBM in order to achieved effective physical plugging for shale formation.
- To produce nanosilica that exhibit optimum particle size which is suitable for improving WBM plugging mechanism

1.4 Scope of Study

This research will involve in the study of maintaining wellbore stability in shale formation. It covers understanding on water based mud performance to prevent the wellbore stability problem focusing on shale swelling. This research also encompasses the production and application of nanoparticles (nanosilica) as additive in designing WBM to provide plugging effect in shale formation and thus preventing water adsorption into the shale formation and eventually cause shale swelling. The

study of this project can be broken down three parts which are, production of nanosilica, preparation and rheological analysis of water-based mud (WBM) containing different concentration of nanosilica, and the analysis on plugging performance of designated WBM in shale formation to prevent shale swelling.

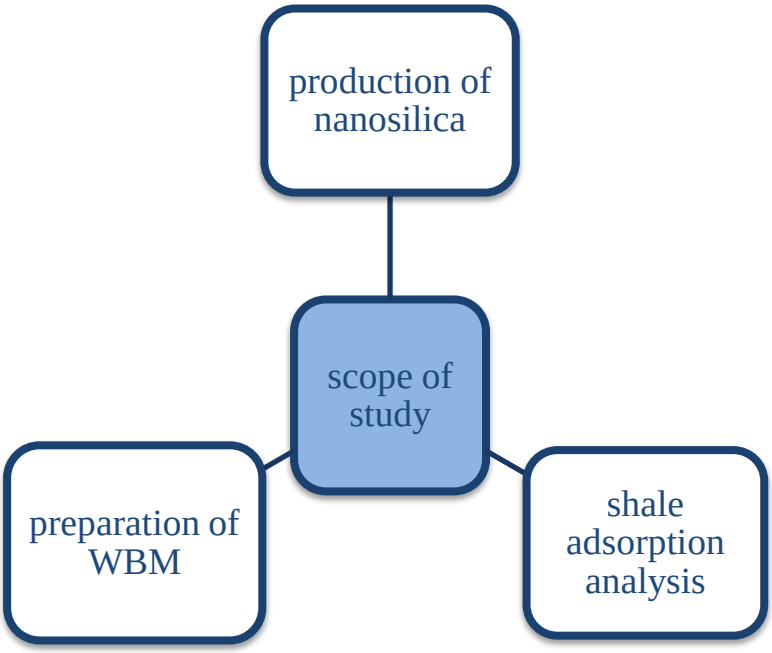


Figure 1: scope of study based on three parts of research planning

CHAPTER 2

LITERATURE REVIEW

2.1 Silica production

According Lazaro (2010), there are two ways to produce silica which is by gel process or pyrogenic silica.

Silica by sol gel process:

In the sol-gel process silica monomers are allowed to condense to colloidal particles. These particles form aggregates, which can age. An important reaction route is the procedure involving waterglass. Waterglass is produced by melting quartz sand with soda. Subsequently the solid waterglass is hydrothermally dissolved in water.

Pyrogenic silica:

The term pyrogenic silica refers to highly dispersed silicas formed from the gas phase at high temperature. Silicon tetrachloride is the usual raw material for flame hydrolysis. It is continually vaporized, mixed with dry air and then with hydrogen, fed to a burner, and hydrolyzed.

2.2 Synthesis of Nanosilica Prepared By Precipitation Method

There are several types of silica, such as fumed silica, precipitated silica, silica gel and colloidal silica, manufactured by different methods. For instance, precipitated silica is prepared by neutralizing a solution of sodium silicate with a sulphuric acid (liquid-liquid procedure) followed by drying of polysilicic acid (Jal et al., 2004).

$$Na2SiO3 + H2SO4 \rightarrow Na2SO4 + H2SiO3$$
$$H2SiO3 \rightarrow SiO2 + H2O$$

The silica particles were generated from the process comprises bringing an alkali metal silicate into contact with sulfuric acid in an aqueous solution. The preparation parameter such as the reaction time affect the final product physical properties, such

as specific surface area, pore size, pore shape and particle morphology as well as chemical properties such as silanol group density.

The concentration of the sand in sodium hydroxide solution is depending on the reaction time. The reaction yield was calculated as the actual weight of precipitate produced, as a percentage of the theoretical quantity possible based on the amount of sand used (Wang et al., 1999).

The specific surface area of silica particles is very high and the aggregation rate decreased when prepared silica in the glycerol system. The resulting spherical silica particles with a very narrow particle size distribution are synthesized. The adsorption method using glycerol gave better results (Vacassy et al., 2000). Method of preparing silica nanoparticles from sand using chemical reaction comprises: removing impurities from the siliceous mudstone which is a raw material by a leaching reaction using sodium hydroxide. Further steps comprise performing heat treatment to remove a water constituent contained in the siliceous mudstone.

2.3 Method of Preparing Nanosilica from Rice Ash Husk (RHA)

One of the alternatives for synthesis of nano silica is extraction from rice husk ash. Rice husk ash (RHA) obtained after burning the rice husk is classified as an industrial waste. Rice husk (RH) consists of about 40% cellulose, 30% lignin group and 20% silica. By burning rice husk at temperature higher than 700°C crystalline silica is formed. The procedure of preparation the nano silica and treated silica (HRHA) is almost the same with using the siliceous sand (Amutha et al., 2010). It is suggested that rice husk ash is an alternative source of amorphous silica. The cost is supposed to be less but with equivalent properties (Amutha et al., 2010).

2.4 Water-Based Muds

Drilling fluid is used to aid the drilling of boreholes into the formation. Drilling fluid is important while drilling oil and natural gas wells and on exploration drilling rigs. Liquid drilling fluid is often called drilling mud. The three main categories of drilling fluids are water-based muds, oil-based mud, and synthetic-based mud.

The main purposes of drilling fluids include providing hydrostatic pressure to avoid formation fluids from entering into the well bore, keeping the drill bit cool and clean

during drilling, carrying out drill cuttings, and suspending the drill cuttings while drilling is paused and when the drilling assembly is transported in and out of the hole. Maintaining stability in vicinity of wellbore is also among the important function of drilling mud.

Most basic water-based mud systems begin with water followed by clays and other chemicals are added into the water to produce a homogenous blend. The clay is usually bentonite, frequently referred to in the oilfield as "gel". Gel likely makes reference to the fact that while the fluid is being pumped, it can be very thin and free-flowing, though when pumping is stopped, the static fluid builds a "gel" structure that resists flow. When an adequate pumping force is applied to "break the gel", flow resumes and the fluid returns to its previously free-flowing state. Many other chemicals are added to a WBM system to achieve various effects, including: viscosity control, shale stability, enhance drilling rate of penetration, cooling and lubricating of equipment.

2.5 Water-Based Mud (WBM) Rheological Study

According to (Benjamin herzhaft, 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 equipment.

Rheology is the study of the deformation and flow matter (ASME Shale Shaker Committee, 2005). Rheology of fluids in the well is the relationship between the flow rate and the pressure required to maintain the flow rate (either in pipe or annulus). The relationships between these properties will affect circulating pressures, surge and swab pressures and hole cleaning ability. In this project, the rheological study comprises of plastic viscosity, yield point, electric stability and gel strength. Each study is so significant to choose a better base fluid.

Viscosity is a measure of the resistance of a fluid which is being deformed by either shear stress or tensile stress. In everyday terms (and for fluids only), viscosity is "thickness" or "internal friction". Thus, water is "thin", having a lower viscosity,

while honey is "thick", having a higher viscosity. If the fluid is less viscous, the movement of the fluid will become easier (Keith, S. 1971). Plastic viscosity relates to the resistance to flow due to interparticle friction. The friction is affected by the amount of solids in the mud, the size and shape of those solids and the viscosity of the continuous liquid phase.

Readings are taken from viscometer. Using the formula below to get Plastic Viscosity;

Plastic Viscosity, PV= [600rpm Reading] – [300 rpm Reading]

Unit: centipoises, cp

Normally, the higher the mud weight, the higher the PV will be. (Jetjongjit, R. 2010). However, if you have an increasing trend of PV without mud weight change, it means that there is an increase in ultra-fine drill solid content in the mud system. Moreover, if you use oil base mud, please keep in mind that emulsified water in oil base drilling fluid will act like a solid, and it will increase the PV dramatically

Yield point estimates the portion of the total viscosity that comes from attractive forces between particles suspended in the mud.

Yield Point, YP = [300rpm Reading] – PV

The gel strength is the shear stress of drilling mud that is measured at low shear rate after the drilling mud is static for a certain period of time (Jetjongjit, R. 2010) but they routinely measured after 10 seconds (initial gel strength) and 10 minutes. Gel strength are determined in two-speed direct-indicating viscometer by slowly turning the driving wheel on top of the instrument by hand and observing the maximum deflection before the gel breaks. Gel strength also can be measured with a rheometer or shearometer (canon, F. 1999).

Specification value;

Gel 10sec: 10 – 20 lb/100ft2

Gel 10min: 20 – 40 lb/100ft2

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 general unit that represents the fluid is centipoise. A centipoise 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 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 cm^3) 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.

Filtration control is one of the main factors considered essential in drilling. Filtration measures the relative amount of fluid loss 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.

2.6 Shale Gas Well Instability

Shale becomes unstable if the effective stress near wellbore exceeds the strength of the hole. It is complicated as shale also sensitive certain drilling constituents, such as water. Stress is altered at and near the borehole walls as shale is replaced by drilling fluid of a certain density. Besides, interaction of drilling fluid with shale alters its strength as well as pore pressure near wellbore. This will cause decrease in shale strength and increases in pore pressure as fluid enters the shale. (Fjaer, et al. 2008)

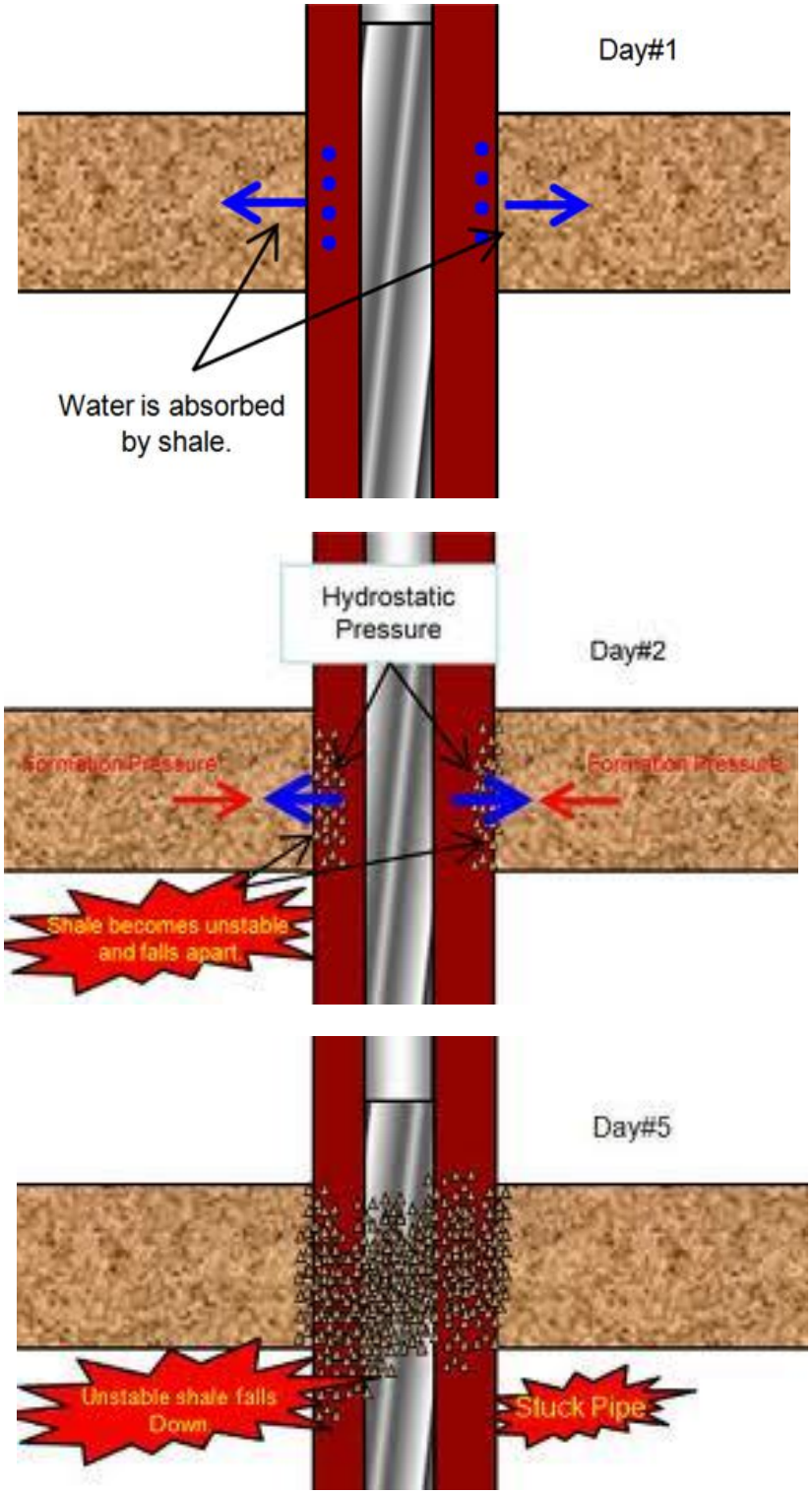


Figure 2: illustration of wellbore instability in shale gas well

Retrieved from www.drillingformulas.com

2.7 Effect of Osmotic Pressure on Shale Stability

Osmotic pressure is also believed to be a contributing factor affecting wellbore stability (Mody and Hale, 1993; Chenevert and Pernot, 1998). When shales are exposed to different drilling fluid for a period of time, swelling pressure can be observed. Moreover, the shale strength is varied with exposure time as process of hydration or dehydration happen. Chenevert, (1970) stated that osmotic pressure can be treated as hydraulic potential that drives water into or out of shale formation. Therefore, exposure of drilling fluid to wall of the wellbore cause the contacted formation being exposed to both hydraulic and osmotic potentials.

2.8 Interaction between Water and Shale (Shale Swelling)

It is believed that the prime factor of shale instability come from unfavorable interaction between the water-based muds and shale formations (Chenevert, 1970; Bol, 1992; Van Oort, 2003). Shale instability is generally caused by pore pressure changes and mechanical property alterations around the wellbore, induced by both chemical and hydraulic effects. These alterations are caused by water and ion movement into or out of the shale formations. Chenevert (1970) discovered that differences in water activity could cause an osmotic flux of water into or out of the shale. Ballard et al (1992) constructed an experimental technique using radioactive tracers to control ion and water movement in shale and discover it to be a diffusion dominated process under zero applied pressure. Concentration gradient is the driving forces for the movement of ion and water into and out of shale. Van Oort (1997) showed pore pressure can fluctuates due to the flux of water and ions into or out of shale.

There are numbers of research concerning moisture adsorption in shale and clays has shown that the adsorbed water leads to an expansion of some of the clay layers (swelling) and a corresponding decrease in the interlayer-bonding and shale strength. This decrease in shale strength associated with water adsorption results in eventual material failure. Bol (1986) found that the difference ib water activity between the drilling mud and shale formation induced water movement which changes the pore pressure distribution. Moreover, the movement of water and ions also affect the mechanical properties of shale, such as strength and modulus.

When describing wellbore stability problems in shale formation, one of the most widely studied problems is shale swelling (Chenevert, 1970; Steiger, 1993). Osmotic effect is believed to be the prime cause of shale swelling. Santarelli (1995) and Carminati (1997) postulated that swelling of shale is induced by capillary effects from shale dehydration. On the other hand, other researcher such as Norrish (1954) believed that shale swelling is caused by the physiochemical interactions of drilling fluids with reactive components present in shale such as montmorillonite.

CHAPTER 3

METHODOLOGY

3.1 Project Planning

A set of methodology has been designed in order to achieve the objectives of the projects. Figure below shows the project planning.

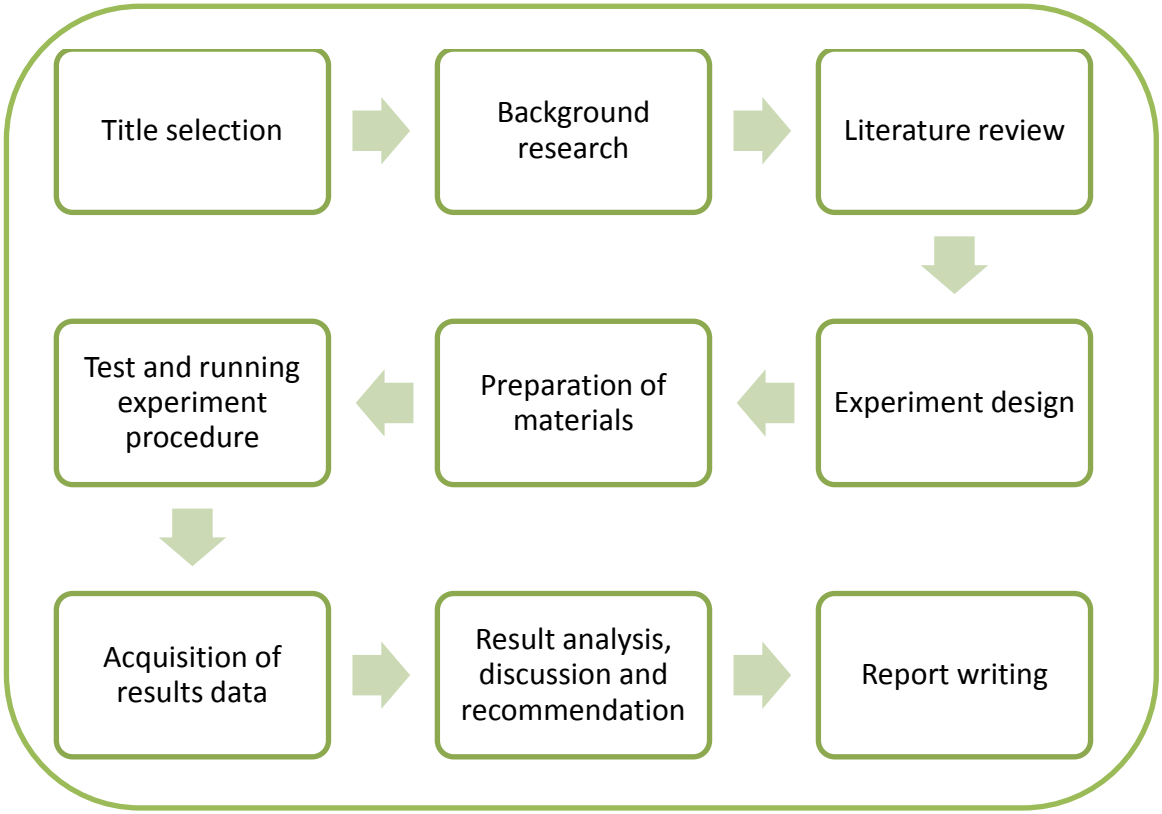


Figure 3: project workflow

Table 1: objective with respective methodology

objectives	methodology
• To determine the optimum concentration of nanosilica in the designed WBM in order to achieved effective physical plugging for shale formation	• Conduct experimental procedure by using shale adsoption test equipment. This test will analyze the plugging performance of the tested fluid on shale sample.
• To produce an improved WBM that is simple in formulation and maintenance shows excellent rheological properties, maintain wellbore stability, and good environmental profile.	• Produce WBM samples with different concentration of nanosilica in laboratory • Analyze the rheological properties of each sample using laboratory equipment such as viscometer and filter press equipment.
• To produce nanosilica that exhibit optimum particle size which is suitable for improved nanosilica WBM	• Run experimental procedure to produce nanosilica from siliceous sand in laboratory. • Conduct analysis on nanosilica performance and particle size analysis by using equipment such as XDS and SEM.

The table above shows the methodology for each objective to be successfully obtained. The description of each method will be explained in detail in the next section of this chapter.

3.2 Experiment / Laboratory Activities Procedure

3.2.1 Shale Sample Preparation

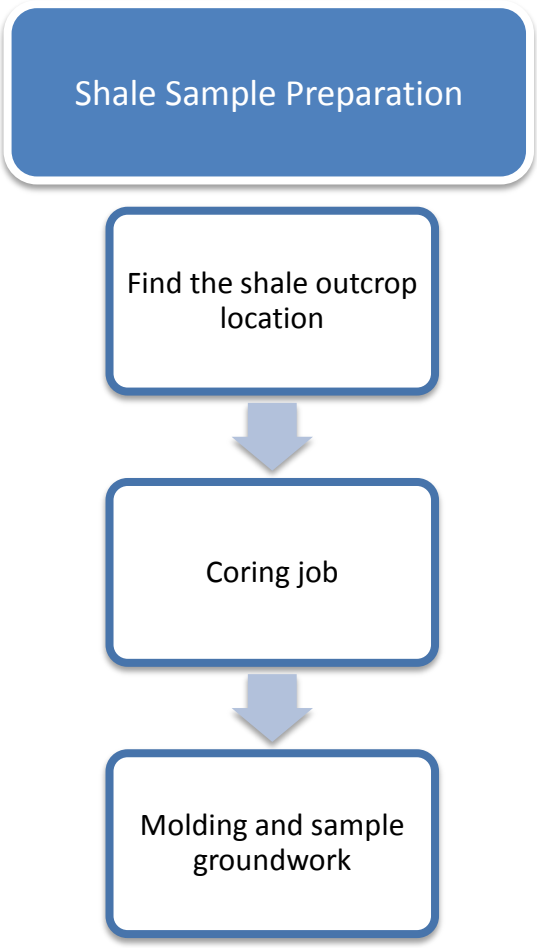


Figure 4: flow chart of shale sample preparation

From the above figure, the first task to prepare the shale sample is to find the suitable location of shale outcrop for coring job. After conducting location survey, Seri iskandar Shale outcrop has been chosen as location for conducting the coring job. The shale rocks outcropping at seri iskandar are part of the Paleozoic sedimentary deposits in kinta valley. The area is easily accessible which located beside the local main road. Figure 3 below show the location of the coring area. The next step is to conduct coring job at the location. Coring job is done by using coring machine by using 2 inch coring bit. The operation of coring job is done individually with assistance from coring technician. This coring job is conducted for two days and total of 9 shale samples with length of 70cm on the average is obtained.

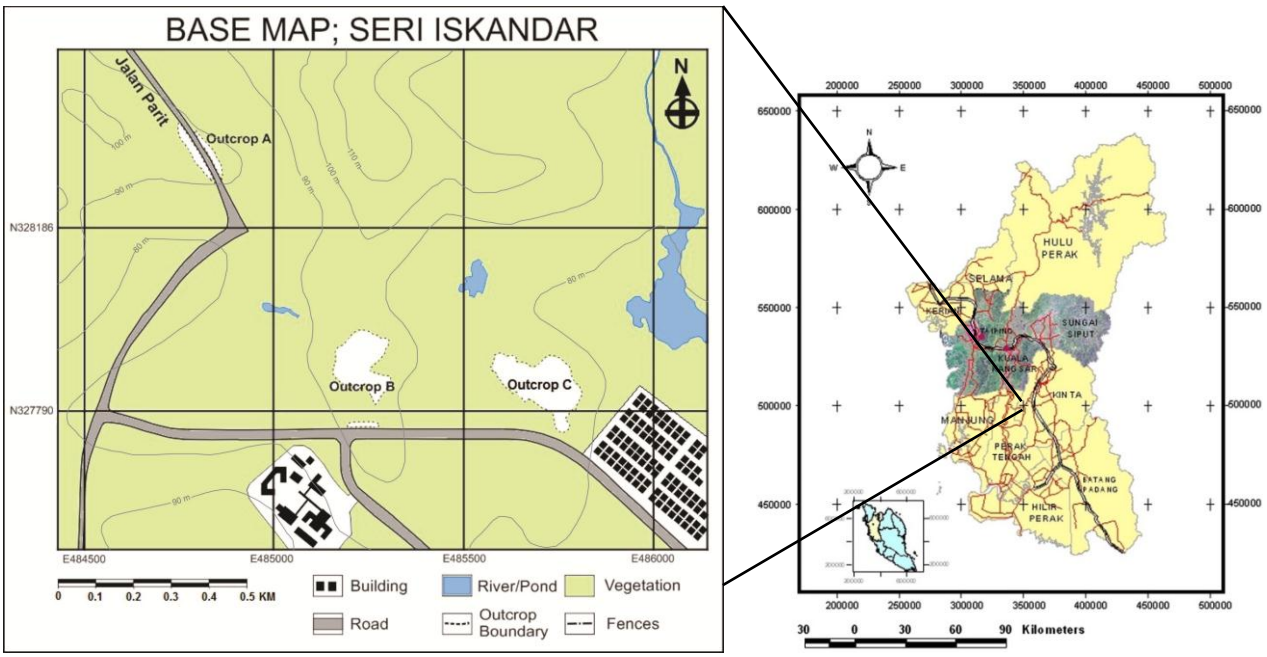


Figure 5: base map of the coring location, Seri iskandar, Perak

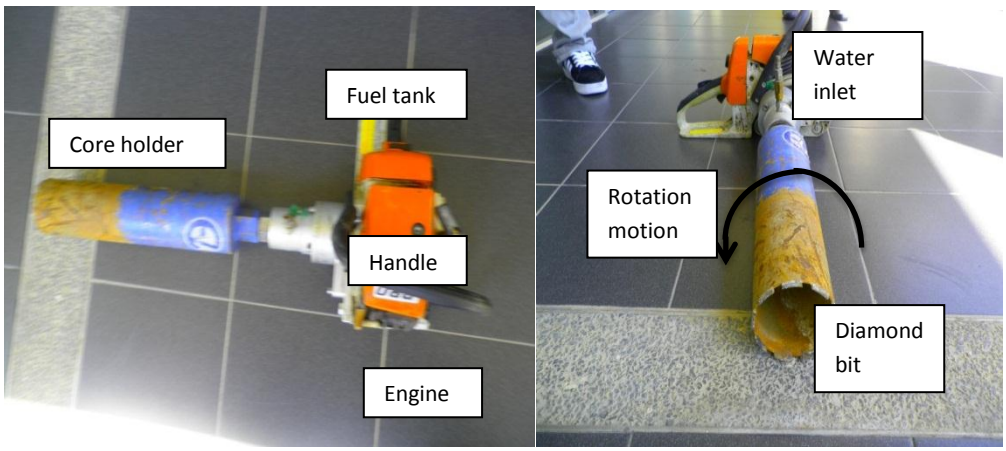


Figure 6: coring machine (2 inch bit)

Figure 7: coring job, 1) core drilling 2) prepare machine 3) obtained core samples

After the samples have been obtained from the coring job, sample molding is conducted to provide support for the weak shale. Shale sample is cemented to protect it from fracture and collapse. Figure below show the core samples molding activities.

Figure 8: molding activities

3.2.2 Nanosilica Production

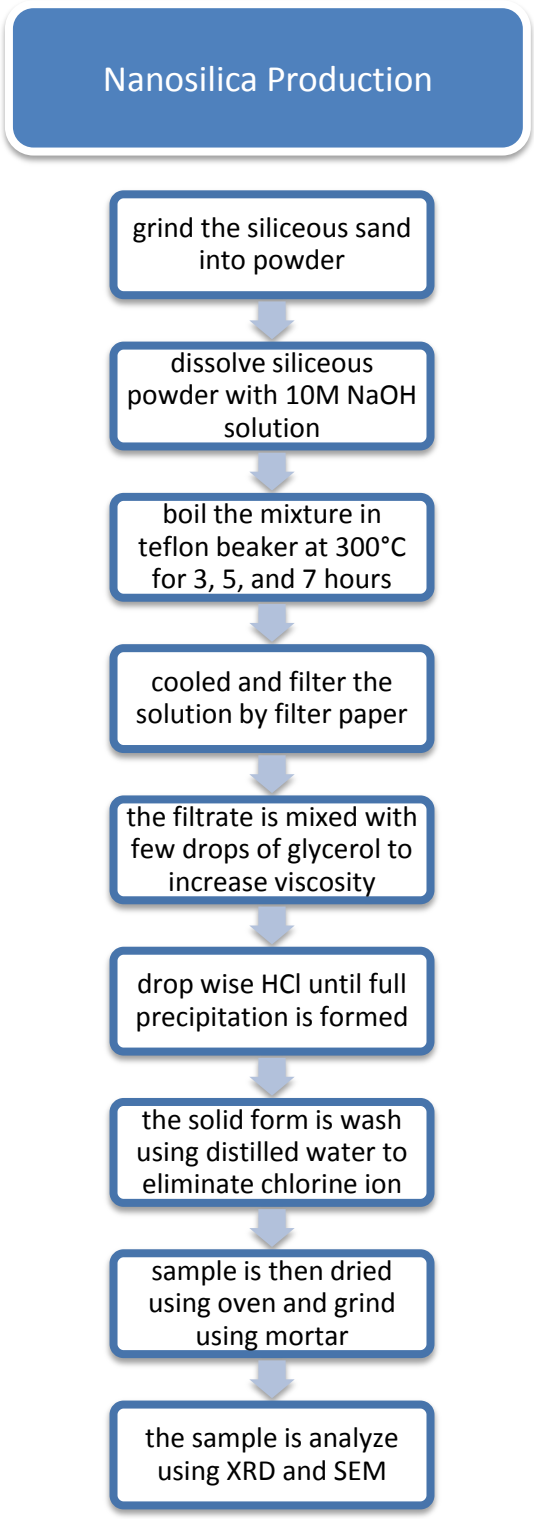


Figure 9: flow chart of nanosilica production

The siliceous sand which is silica mineral sources is grinding using grinder. As the result, powder form silica sand is getting with large surface area. The powder form silica sand is characterized using available equipment such as XRD and SEM. Nano

silica is prepared from siliceous sand using a chemical reaction by precipitation method. First, sand is heated with 10M sodium. The reaction occurred is as follows:

$$\underset{\text{(Sand)}}{SiO_2} + \underset{\text{(Sodium hydroxide)}}{2NaOH} \rightarrow \underset{\text{(Sodium silicate)}}{Na_2SiO_3} + \underset{\text{(Water)}}{H_2O} + \underset{\text{(Solid)}}{Impurities}$$

The reaction is taking place inside the Teflon beaker and is placed on the hot plate with temperature 300°C. The sample of the solution is taken for each 30 minutes and few drops of hydrochloride acid are drop into the sample. The process is repeated until white precipitation can be observed. The reaction time has to be repeated for several times to observe the effect of reaction time with the yield. The reaction times are manipulated at 3, 5 and 7 hours. The graph of yield versus reaction time is plotted to study the effect of reaction time on sodium silicate yield at constant temperature.

The solution is then filtered to separate the unreacted sand and sodium silicate. The samples are then divided into two portions where one is without the glycerol and one with glycerol. Glycerol is added into the solution to increase the viscosity and prevent agglomeration among particles. Next, the sodium silicate which is distillate is added with acidic acid until full white precipitation formed. The process named precipitation method and being controlled by controlling the pH hydrochloric acid is added until the solution having pH less than 3. The reaction is as follows:

$$Na_2SiO_3 + 2HCl \rightarrow H_2SiO_3 + 2NaCl$$

Samples are then washed repeatedly in distilled water under identical conditions until no more chlorine found in the solution. Water from H2SiO3 is removed by drying in oven and follows by grounding so that amorphous silica is formed. The silica then is characterized using SEM and XRD to study their properties. Table below show the equipment and apparatus used in this experiment.

Table 2: List of chemical, apparatus and equipment

Chemical	Amount
Hydrochloric acid (HCL)	1L
Sodium Silicate (Na2SiO3)	1L
glycerol	20mL
Sodium hydroxide (NaOH)	500mL
Siliceous sand	2.5kg
Apparatus	
Teflon beaker	Hot plate
Glass beaker	Centrifuge
Plastic bottles	Mortar and pestle
Portable ph meter	Oven
Filter paper	Magnetic stirrer
Equipment	
XRD	
SEM	

3.2.3 Preparation Of Water Based Mud (WBM)

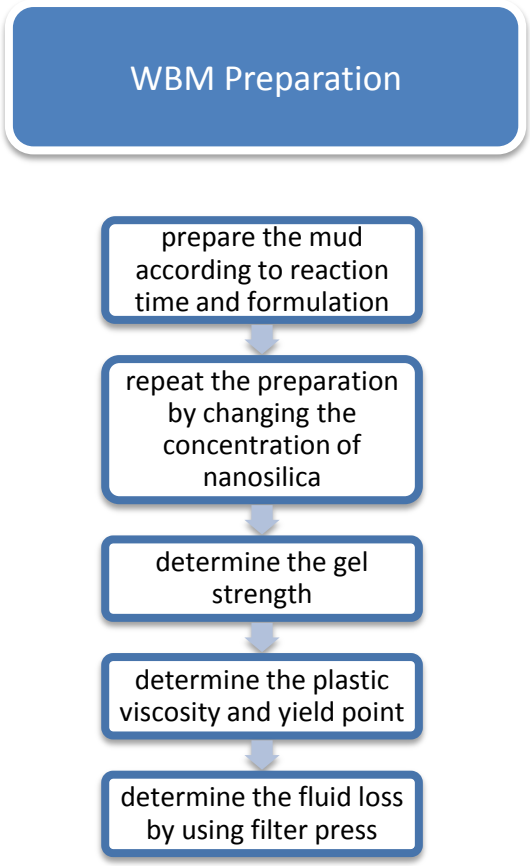


Figure 10: flow chart of preparing water based mud with different concentration of nanosilica

Table 3: data of mud mixing flow according to its component

Mud mixing flow	
Mixing time (min)	**Base and additives**
0	Fresh water
2	Soda ash
2	Potassium chloride
5	HYDRO-PAC LV
5	HYDRO ZAN
2	DRILL-BAR
2	Caustic soda
2	Nanosilica
23	Mixing complete

Table 4: data of WBM formulation

Mud formulation				
sample	1	2	3	4
Density, ppg	11.5	11.5	11.5	11.5
Formulations	**0% nanosilica**	**5% nanosilica**	**10% nanosilica**	**15% nanosilica**
Fresh water, ml	330	330	330	330
Soda Ash, g	0.3	0.3	0.3	0.3
Potassium Chloride, g	45	45	45	45
HYDRO-PAC LV, g	2.5	2.5	2.5	2.5
HYDRO-ZAN, g	1.25	1.25	1.25	1.25
DRILL-BAR, g	62	55	49	38
Caustic soda, g	0.2	0.2	0.2	0.2
Nanosilica, g	0	30	60	90

Water based mud (WBM) was prepared in mud laboratory according to its formulation and mixing flow from table above. The complete formulated WBM is then stored in sealed container at atmospheric temperature to prevent evaporation. Each sample will be used in rheological analysis to identify mud density, plastic viscosity, yield point, gel strength and fluid loss using filter press. The samples will also be used in shale adsorption test to study the plugging effect of nanosilica.

A procedure is given for determining the density. The density of drilling fluid is expressed as grams per cubic centimeter, kilograms per cubic meter, pounds per gallon or pounds per cubic foot. Equipment used to measure the mud density is called MUD BALANCE

Figure 11: tools used to determine mud density (mud balance)

Procedure:

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

2) Measure the temperature of the drilling fluid and record.

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) 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.

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 centerline.

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.

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

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

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

Figure 12: tool used to determine viscosity (viscometer)

Procedure (Determination of viscosity using the Marsh funnel):

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.

2) Remove finger and start the stopwatch. Measure the time taken for mud to fill in the cup.

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

Procedure (Determination of viscosity and/or gel strength using a direct-indicating viscometer):

1) Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 100 cm3) 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 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.

2) Heat (or cool) the sample to the selected temperature. Use intermittent or constant shear at 600r/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.

3) With the sleeve rotating at 600rpm, 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 R600 in Pascal for 600rpm

4) Reduce the rotor speed to 300 rpm and wait for the dial reading to reach steady value. Record the dial reading R300 in Pascal for 300 rpm.

5) Stir the drilling fluid sample for 10 s at 600 rpm.

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 rpm speed, the maximum reading attained after starting rotation at 3 rpm is the initial gel strength. Record the initial gel strength (10-second gel) in pounds per 100 square feet.

7) Re-stir the drilling fluid sample at 600 rpm for 10 s and then allow the drilling fluid to stand undisturbed for 10 min. repeat the measurements as in 6 and report the maximum reading as the 10- minute gel in Pasc

3.2.4 Design of Shale Adsorption Equipment

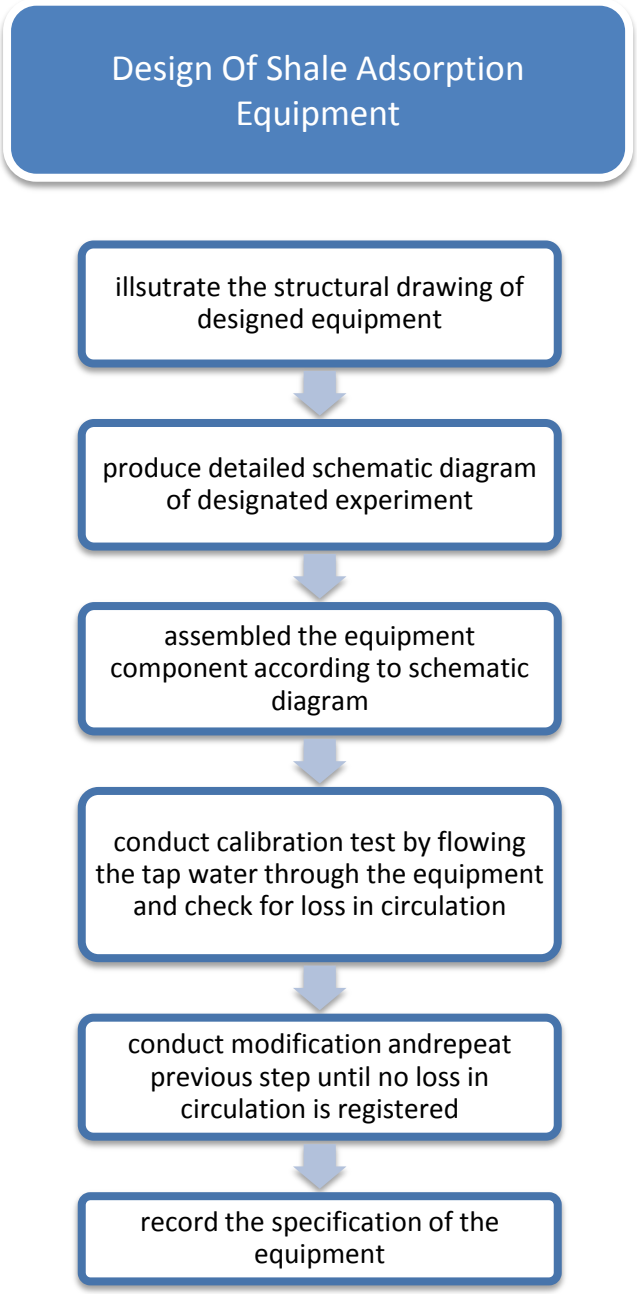


Figure 13: flow chart of shale adsorption equipment design

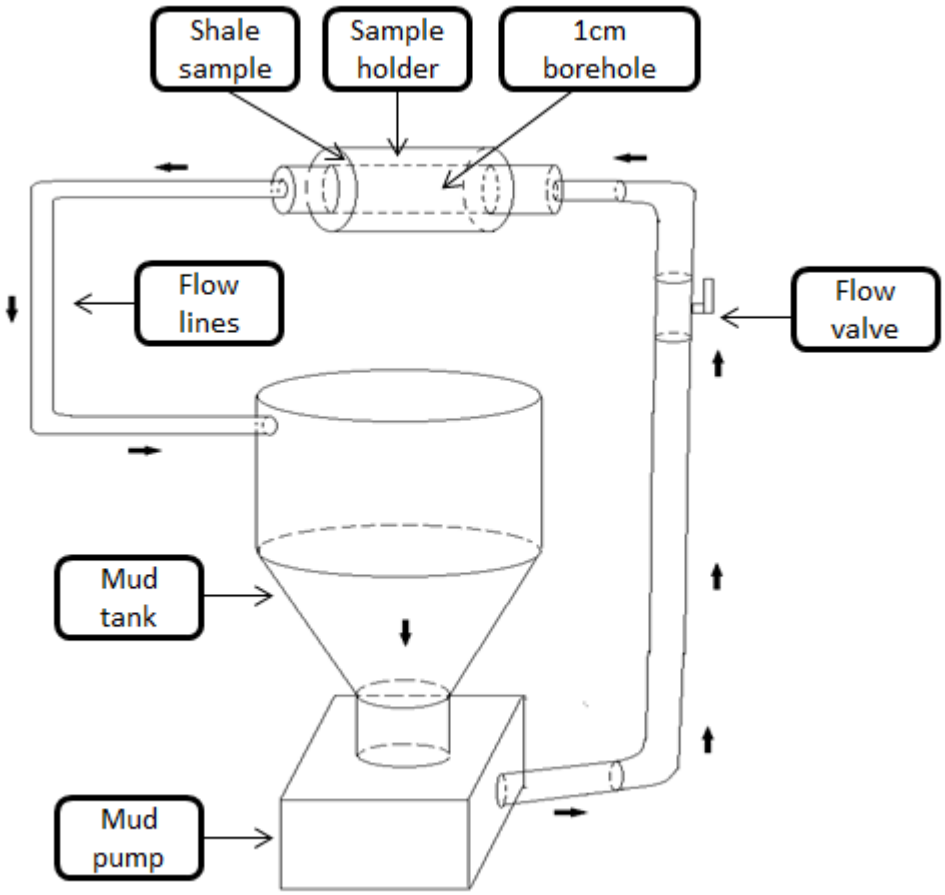


Figure 14: structural drawing of shale adsorption equipment

Figure 9 and 10 shows the flow chart of designing and structural drawing of shale adsorption equipment. The purpose of this equipment is to analyze the mass of fluid adsorbed by shale sample when fluid is flow through the sample. The mechanism of this equipment is to let the designated mud to flow through one inch cylindrical borehole of the shale sample. As mud flow through the sample, the change in weight of the sample is recorded. The change in weight of the sample is assumed due to absorption of fluid from the mud into the shale sample. From this data, a pattern of adsorption rate of each sample can be analyzed. Table below shows the description of each component of the shale adsorption equipment.

Table 5: component description

Components	Specification	Function
1)Flow lines	• Steel pipe • 1cm diameter • Leak proof	• Flow the fluid
2)sample holder	• PVC material • 5cm diameter • 10cm length	• To hold shale sample
3)Mud tank	• Aluminum material • 2 liter volumes	• To store the fluid
4)Mud pump	• 240 – 280 volt • 95 Watt • 0.3A • 2000-2300 L/H	• To pump the fluid through the equipment
5)electronic balance	• Up to 1200g • 0.00 sensitivity	• To measure the change in weight of shale sample

Reliability test has been run for this equipment to make sure that the equipment meets the expectation of the experiment objectives. During the reliability test, one liter of freshwater is flow through the equipment for 3 consecutive days. Loss in freshwater volumes is determined for each day. Table below show the results of the reliability test

Table 6: result of reliability test

Number of days	Freshwater loss in volumes (ml)
1	3ml
2	0ml
3	1ml

From the table, it can be observed the reliability of the equipment is dependable for the purpose of the experiment. The equipment only exhibit error of 0.4% for operation period of 72 hours.

3.2.5 Shale Adsorption Test

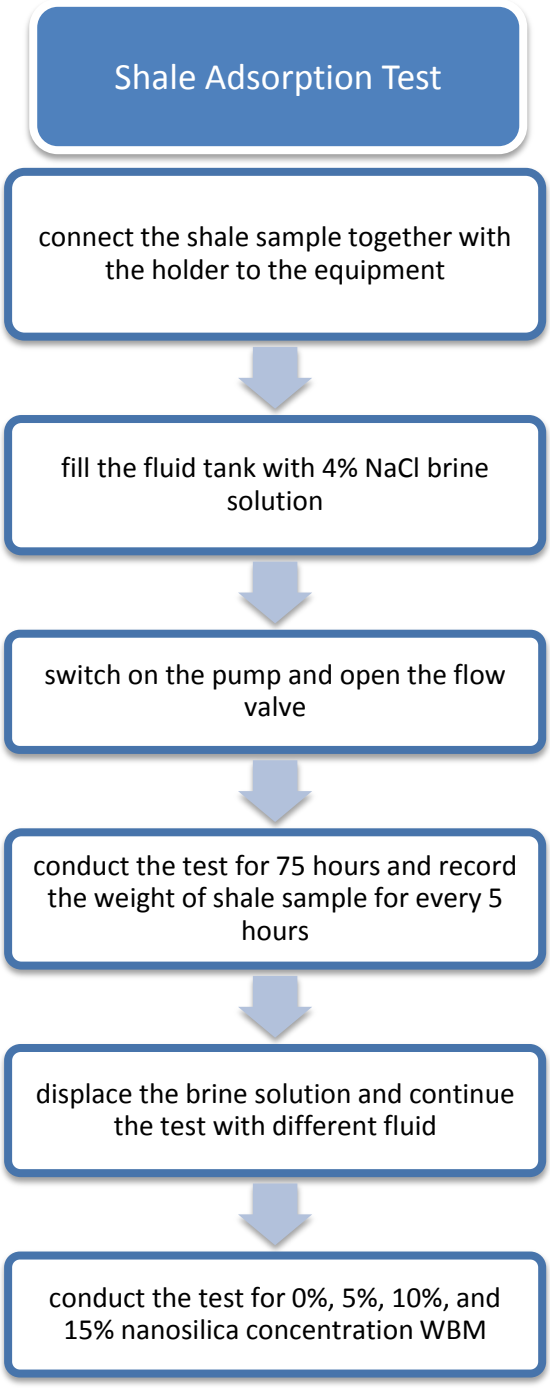


Figure 15: flow chart of shale adsorption test

Figure above show the procedure of shale adsorption test. The mechanism of this test is to measure the change in weight of shale sample. Shale sample is assumed to gain its mass from fluid adsorption process from the tested liquid (water-based mud and brine). Therefore, the change in weight pattern of each sample can be observed.

From those patterns, the optimum concentration of nanosilica which is required by WBM to effectively plug the pore space can be obtained.

By assuming change in weight of the shale sample is equal to weight of water adsorbed by the shale sample, the rate of adsorption is therefore can be explain as follows;

$$W_{st} - W_{si} = W_{awt}$$

Where;

$W_{st} = weight\ of\ shale\ sample\ at\ t\ time$

$W_{si} = initial\ weight\ of\ shale\ sample$

$W_{awt} = weight\ of\ water\ adsorbed\ by\ shale\ sample\ at\ t\ time$

Therefore;

$$rate\ of\ adsorption, \qquad A_w = \frac{W_{awt}}{t}$$

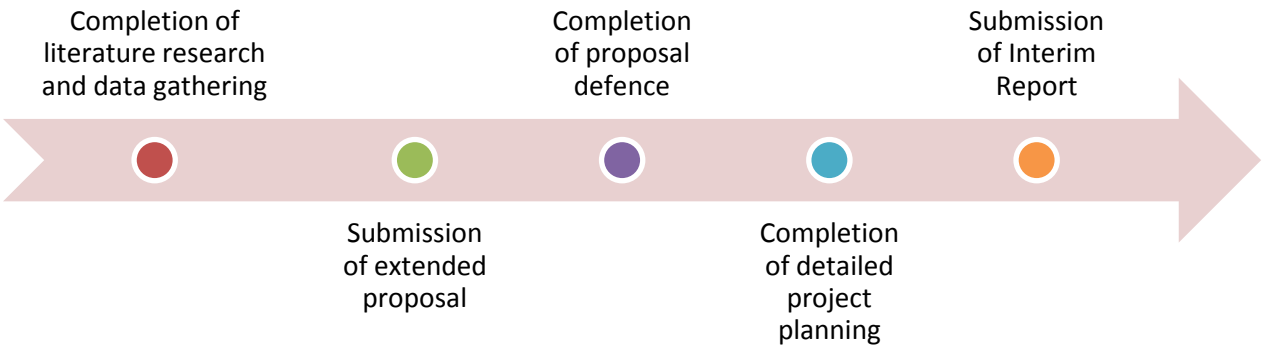
Thus, rate of change of shale sample weight is equal to rate of water adsorption

3.3 Gantt Chart

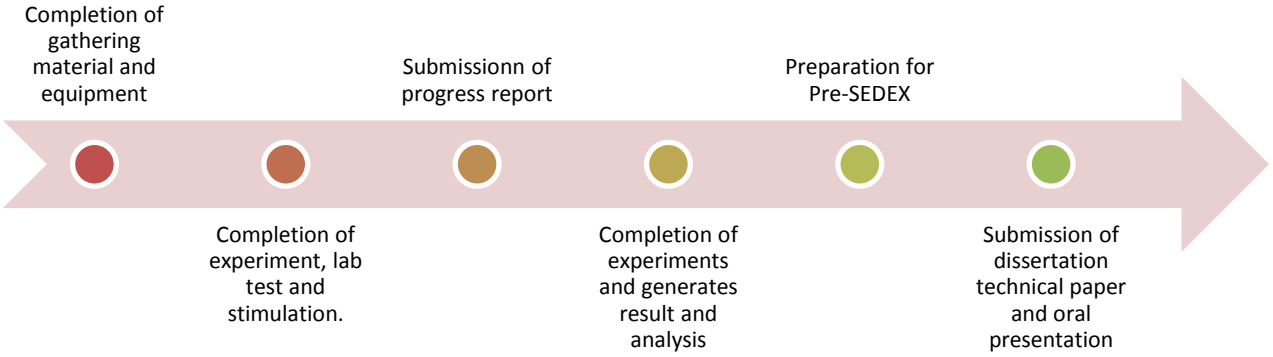
PROJECT ACTIVITIES	WEEKS																										
	Final Year Project 1														Final Year Project 2												
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Topic Selection and Proposal																											
Preliminary Research Work																											
Submission of Extended Proposal																											
Identify material and equipment																											
Proposal Defense																											
Project Work Detailed Planning																											
Submission of Interim Draft Report																											
Submission of Interim Report																											
Gathering Materials																											
Conducting Experiments and Lab Tests																											
Submission of Progress Report																											
Result Analysis and Discussion																											
Pre-SEDEX Preparation																											
Submission of Draft Report																											
Submission of Dissertation (soft bound)																											
Submission of Technical paper																											
Oral presentation																											
Submission of Project Dissertation (hard bound)																											

3.4 Key Milestone and Project Activities

FYP 1



FYP 2



CHAPTER 4

RESULTS AND DISCUSSION

4.1 Production of Nanosilica Analysis

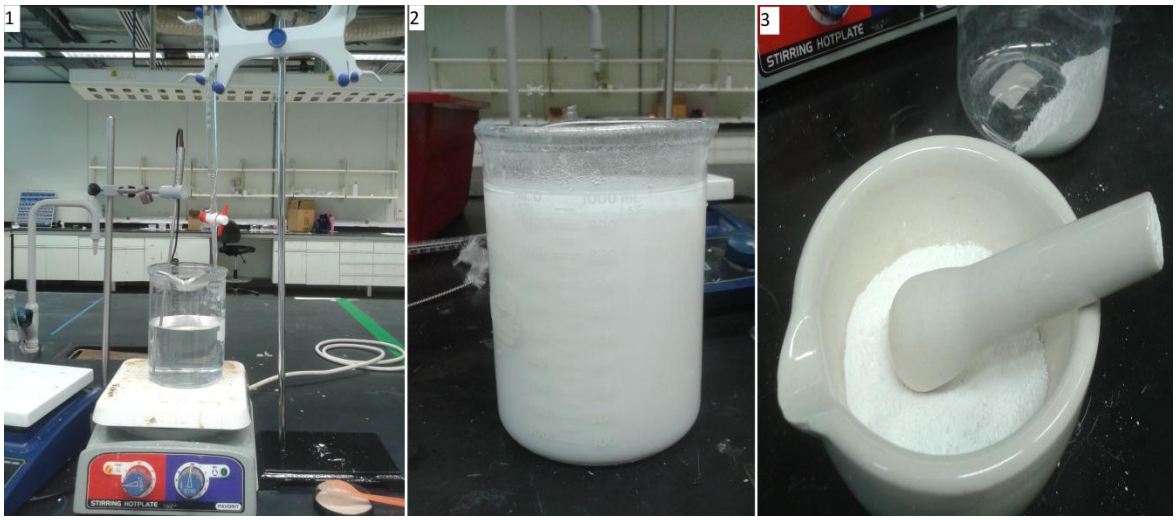


Figure 16: 1) acid titration process to sodium sillicate to produce nanosilica, 2) precipitated nanosilica, 3) grinding process by using mortar

Figure 17: 1) sieving process to collect nano sized silica, 2) collected nanosilica

Figure 15 and 16 show the result from the process of producing nanosilica. From this figure, the amount of obtained nanosilica is enough for the purpose of mixing with

WBM and producing overall 3 WBM sample containing different concentration of nanosilica. A total of 880g of nanosilica powder is produced from this process. This nanosilica is then taken to the lab for analysis. The next section will explain the characterization and analysis on nanosilica sample.

4.1.1 Effect of Reaction Time On Yielding Sodium Silicate

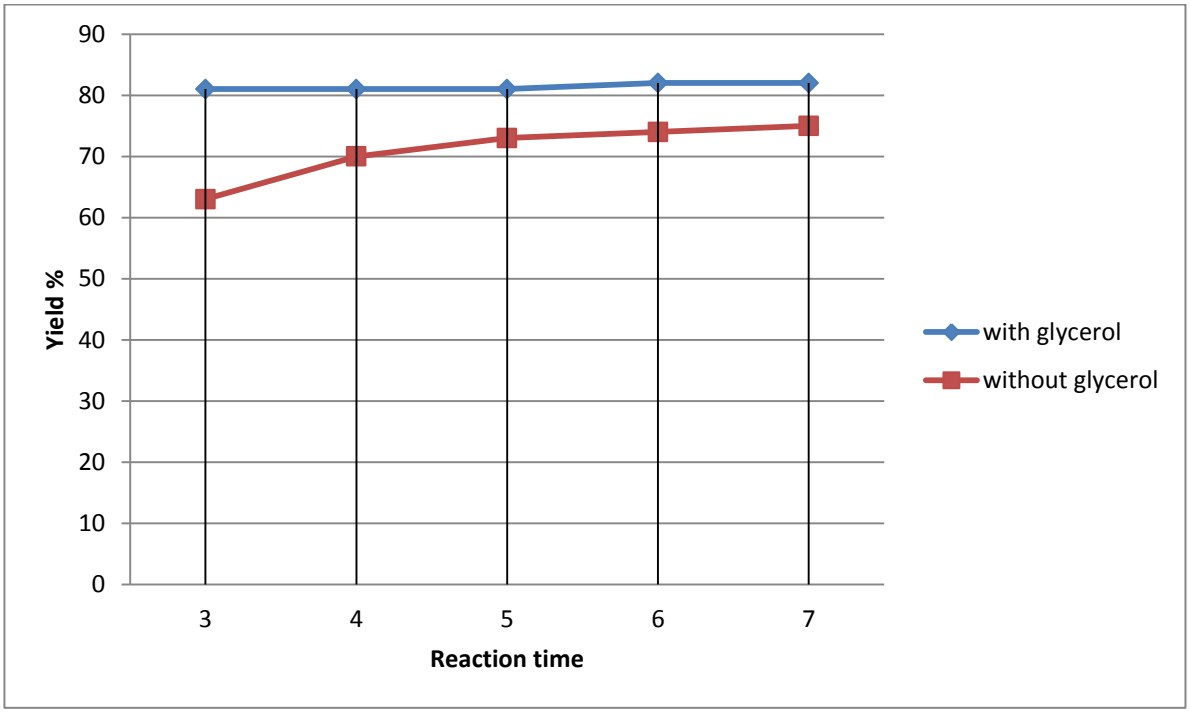


Figure 18: Graph of Yield vs. reaction time

Figure 18 shows the yield of sodium silicate solution by solving the siliceous sand into sodium hydroxide aqueous solution with increasing reaction time. The reaction temperature is kept constant at 300°C and reaction time increase from 3 hr to 7 hr. For this experiment, some of heat is assumed lose to surrounding atmosphere and also absorbed by Teflon beaker. Two sample of sodium hydroxide aqueous solution is prepared where one sample with glycerol and another one is without glycerol. The purpose of glycerol is to avoid agglomeration formation and hence control particle size of nanosilica. The equation below shows the reaction that taking place:

$$\underset{\text{(Sand)}}{SiO_2} + \underset{\text{(Sodium hydroxide)}}{2NaOH} \rightarrow \underset{\text{(Sodium silicate)}}{Na_2SiO_3} + \underset{\text{(Water)}}{H_2O} + \underset{\text{(Solid)}}{Impurities}$$

From the above graph in figure 18, for both curves, the yield efficiency is increasing with increasing reaction time. Though, the yield efficiency with existence of glycerol shows almost consistent at 81%. For reaction without glycerol on the other hand shows rapid increases in yield efficiency and rate slowed down after reaching reaction time of 5 hours. The final yield without glycerol remains constant at 76% after reacted for 7 hours. This observation of data suggests the yield of sodium silicate solution is higher with presence of glycerol.

4.1.2 Particle Size Analysis

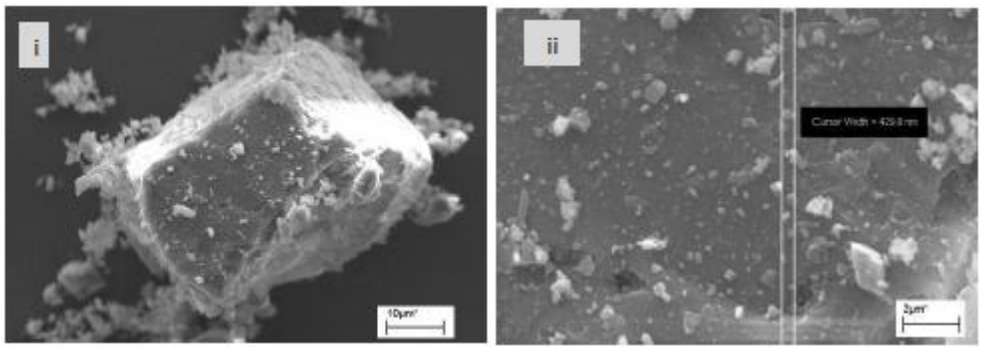


Figure 19: SEM micrographs of siliceous sand i) 1000 magnified and ii) 5000 magnified

The above figure shows the image of SEM micrograph of siliceous sand. From the image, it can be observed that the shapes of the siliceous sand particles consist of uniformly crystal with flat surface area. It is obvious the mean particle size of siliceous sand is larger than that of precipitated nanosilica. The average size of particle is measured around 531nm for siliceous sand.

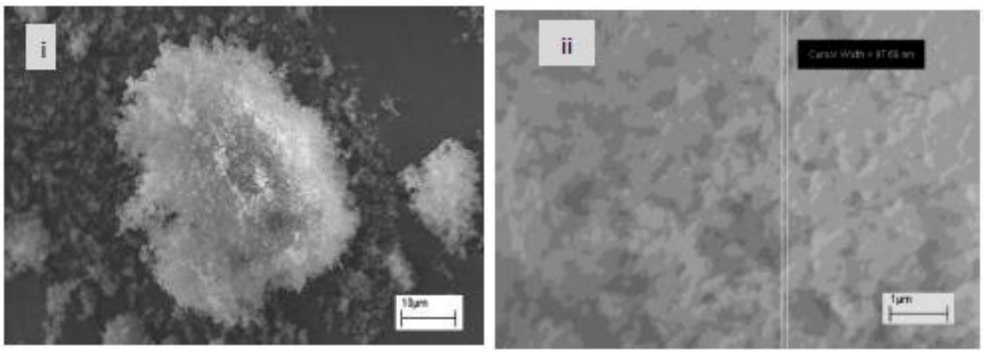


Figure 20: SEM micrograph of precipitated nanosilica i) 1000 magnified and ii) 5000 magnified

The above figure shows the image of SEM micrograph for precipitated nanosilica. From the image of SEM, the particle size of precipitated nanosilica is far smaller when compared with that of siliceous sand. It has been measured the average particle size of precipitated nanosilica is around 78nm and particles size for siliceous sand is 531 nm. Besides, the shape of precipitated nanosilica is also different than siliceous sand. The shape of precipitated nanosilica exhibits an indefinite or spherical shape. The microscopic observation reveals that the particles comprise of amorphous silica particle with extremely fine particles.

Compared to previous study, the existence of glycerol in this experiment helps to reduce agglomeration of particles. The agglomeration mechanism influences the particle size and morphology of the samples. For the purpose of this study, a nanosilica sample with less agglomeration is better in order to increase the efficiency of plugging effect. Therefore, when plugging effect is optimum, the amount of water particles from WBM entering the shale formation can be significantly reduced.

4.2 WBM Rheological Test Result

Table 7: rheological test result summarization

WBM samples with different nanosilica concentration		Nanosilica 0%wt	Nanosilica 5%wt	Nanosilica 10%wt	Nanosilica 15%wt
Viscometer reading	600 rpm	105	108	114	120
	300 rpm	68	74	81	91
	Plastic viscosity	37	34	33	29
	Yield point	31	40	48	62
Gel strength	10 seconds	11	10	10	11
	10 minutes	14	12	13	15

Table 8: filtration loss tabulation for WBM samples with 0%wt and 5%wt nanosilica concentration

sample	filtration loss	time	sample	filtration loss	time
0%		0	5%		0
	4.2	5		3.8	5
	6.6	10		6	10
	7.5	15		7.1	15
	8.2	20		7.9	20
	8.8	25		8.5	25
	9	30		8.6	30

Table 9: filtration loss tabulation for WBM samples with 10%wt and 15%wt nanosilica concentration

sample	filtration loss	time	sample	filtration loss	time
10%		0	15%		0
	3.1	5		2.9	5
	5.1	10		4.2	10
	6.6	15		5.1	15
	6.9	20		5.6	20
	7.4	25		6.1	25
	7.9	30		6.7	30

The table 7, table 8, and table 9 shows the result on the rheological test that have been conducted for each WBM samples. A total of four WBM sample has been prepared according to its respective mud formulation. The four WBM samples exhibit nanosilica concentration of 0%wt, 5%wt, and 10%wt respectively. The next section of this topic will analyze on each rheological properties that have been determined.

4.2.1 Plastic Viscosity Analysis

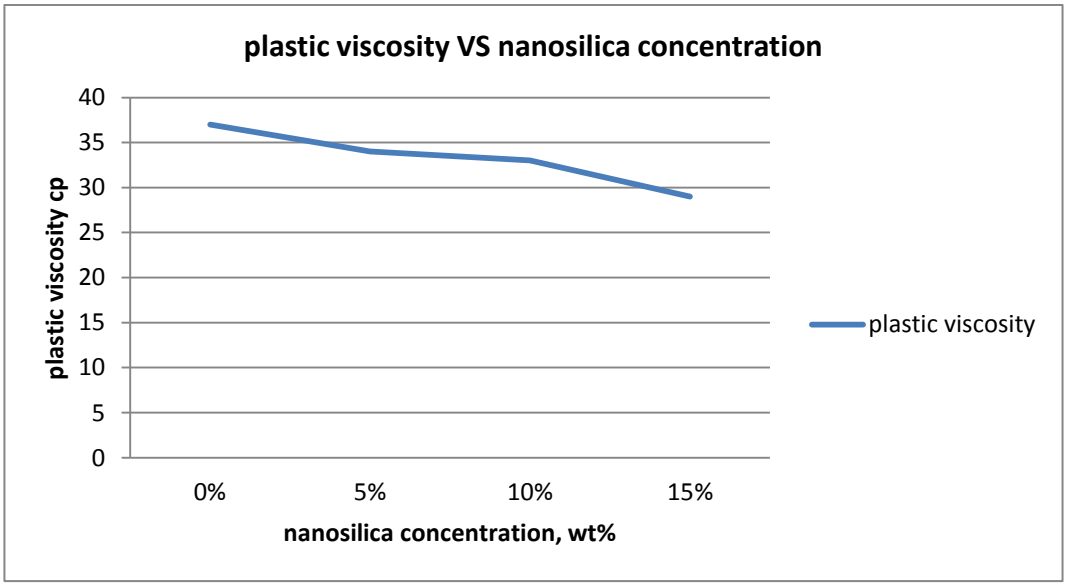


Figure 21: plastic viscosity trend respective to increase in nanosilica concentration in WBM

Viscosity is a measure of the resistance of a fluid which is being deformed by either shear stress or tensile stress. Plastic viscosity is related to the resistance to flow due to interparticle friction. The friction is affected by the amount of solids in the mud,

the size and shape of those solids and the viscosity of the continuous liquid phase. Normally, the higher the mud weight, the higher the PV will be. From figure 20, it can be observed that the trend of plastic viscosity is decreasing although the samples exhibit almost similar mud density. However, Plastic viscosity is a function of solids concentration and shape and therefore as the amounts of nanosilica are increased, the values of plastic viscosity decrease. Nevertheless, the range of plastic viscosity from figure 20 only shows small variation in its value which means the concentration of nanosilica in WBM do not significantly affect the plastic viscosity trend.

4.2.2 Yield Point Analysis

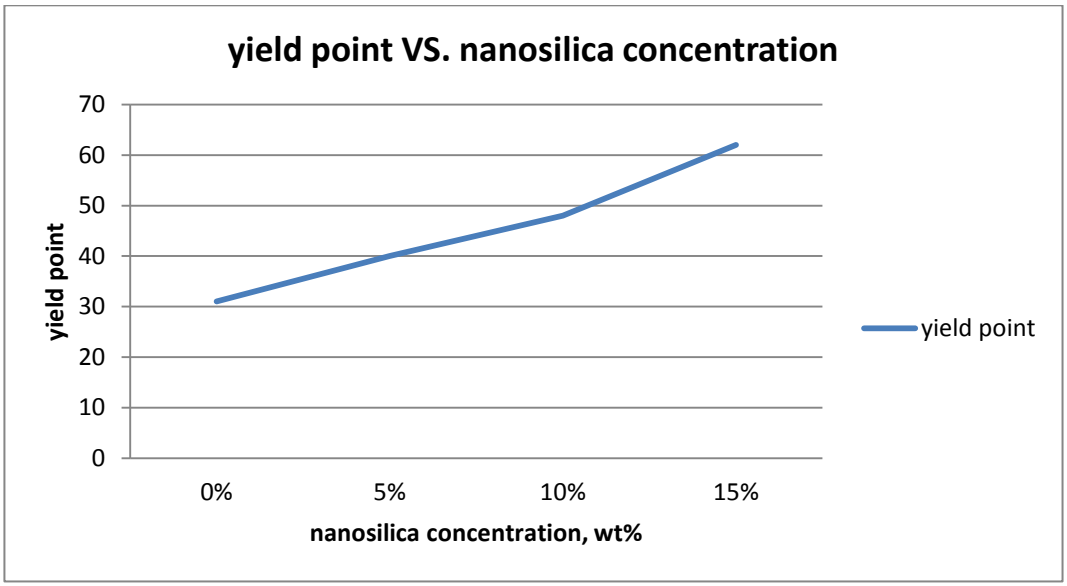


Figure 22: yield point trend respective to increase in nanosilica concentration in WBM

Yield point estimates the portion of the total viscosity that comes from attractive forces between particles suspended in the mud. From figure 21, it can be perceived the value of yield point tend to increase as the concentration of nanosilica in WBM is increased. Therefore, it can be concluded that the presence of nanosilica would likely to enhance the attractive forces between particles suspended in the mud. In oil and gas industry, yield point is used to evaluate the ability of a mud to lift cuttings out of the annulus. A high yield point implies a non-Newtonian fluid, one that carries cuttings better than a fluid of similar density but lower yield point. However, too high yield point will cause high pressure loss when mud is circulated. From this analysis, obviously the increase of nanosilica concentration would tend to boost the ability of WBM to lift cutting from annulus to the surface. Nonetheless, too much

nanosilica will increase the yield point and cause high pressure loss during circulation which is not good for drilling operation. Therefore an optimum amount of nanosilica is important such that it will maintain the WBM performance.

4.2.3 Gel Strength Analysis

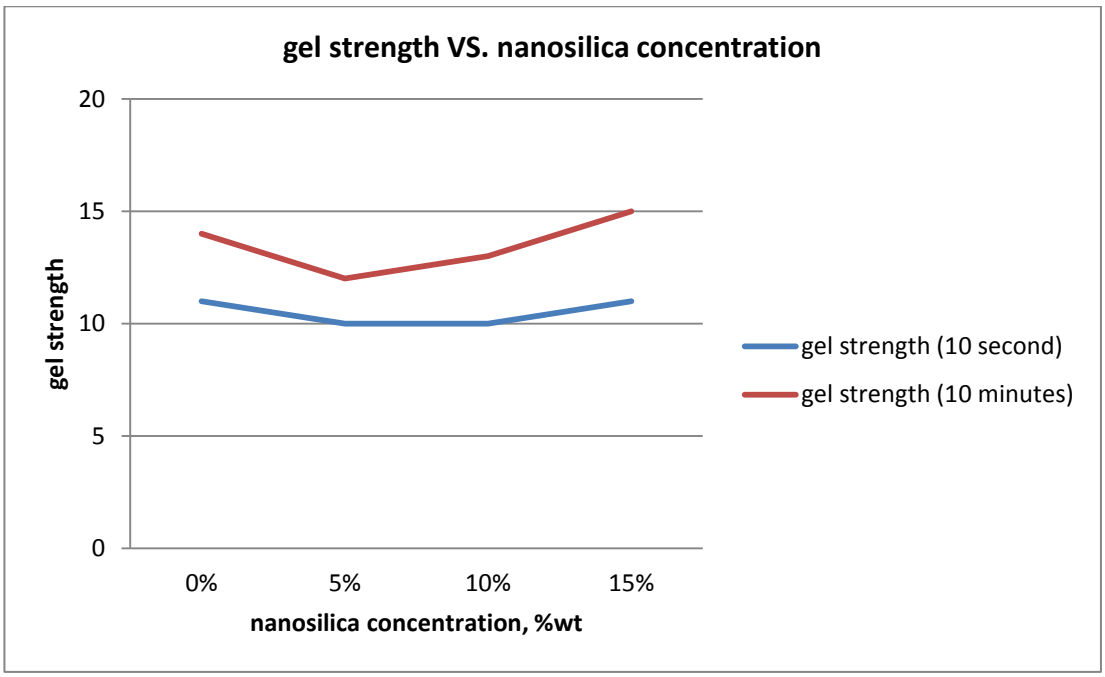


Figure 23: Gel strength trend respective to increase in nanosilica concentration in WBM

Gel strength is the ability to suspend cuttings when the mud is stationary. Based on figure 22, the pattern of gel strength can be analyzed. According to the observation on the gel strength pattern, the increment of gel strength is not significant as the nanosilica concentration is increased because nanosilica does not change the chemical bonding in the drilling fluid. This is due to nature properties of silica which is not strongly reactive to disturb the molecular bonding between substances in water based mud.

4.2.4 Fluid Loss

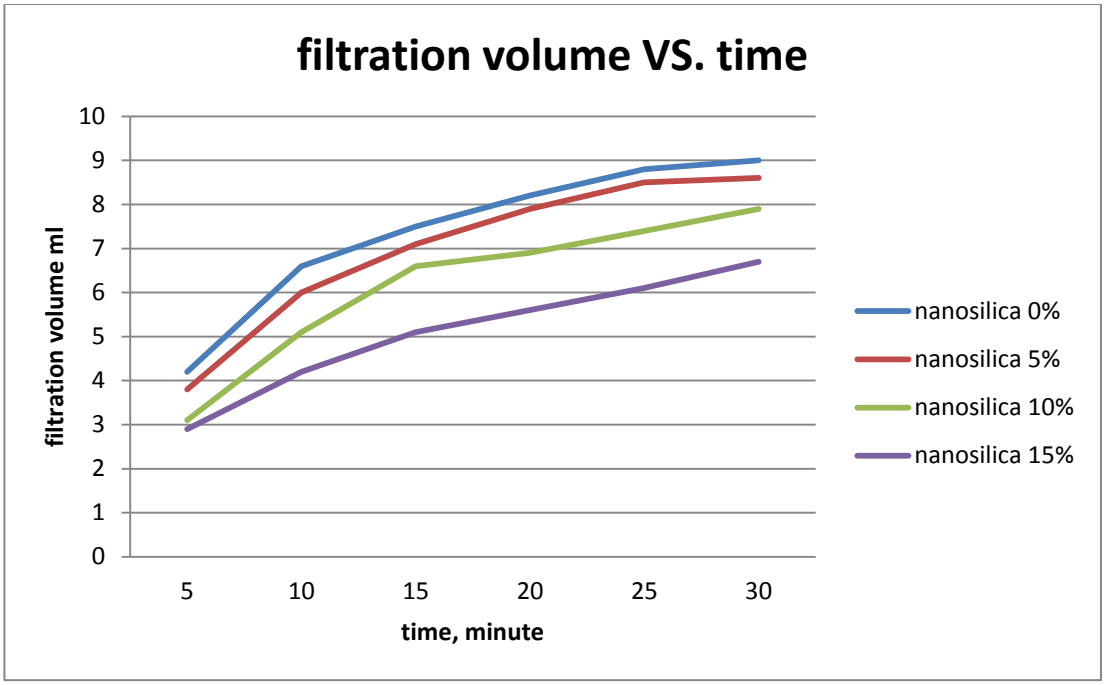


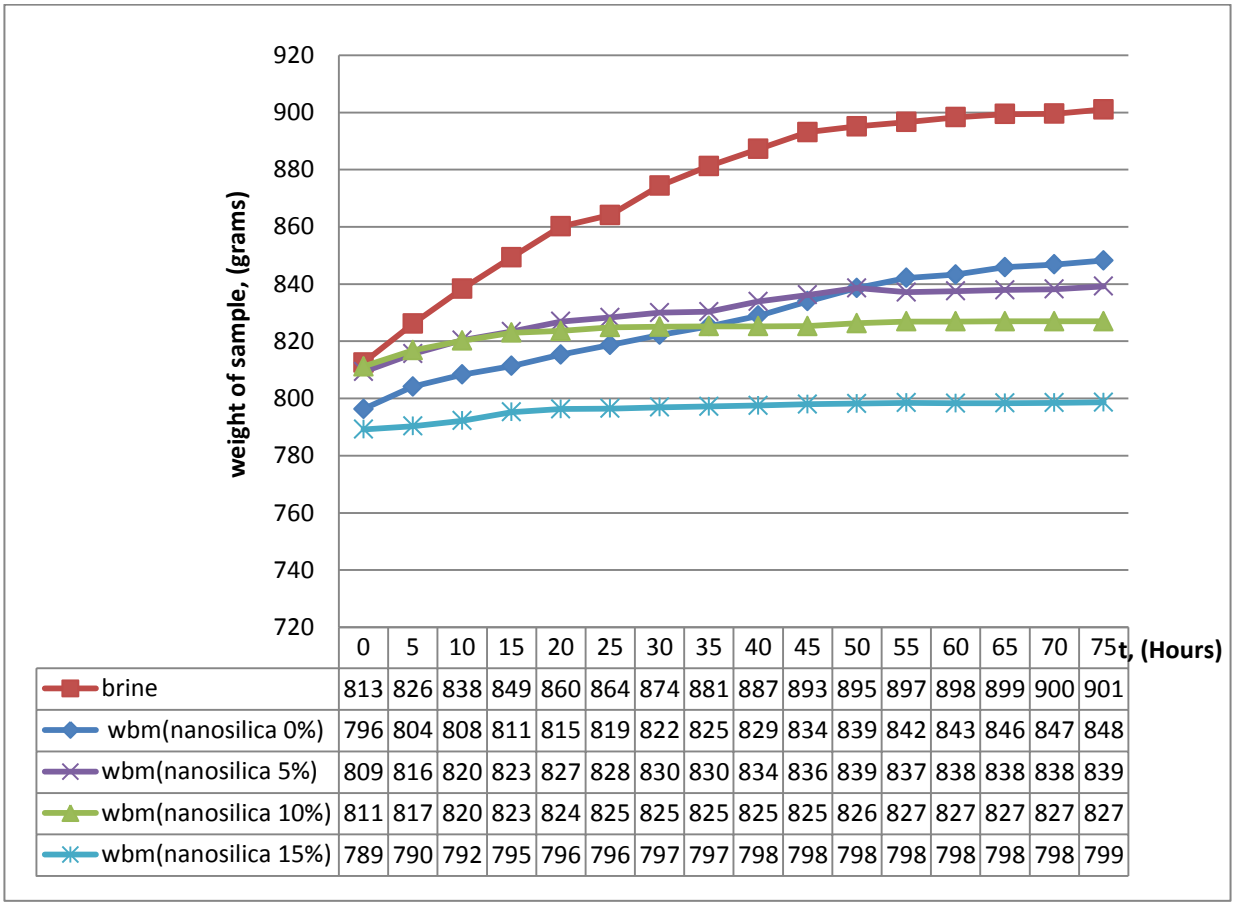
Figure 24: Filtration loss trend with increase in nanosilica concentration in WBM respective to time.

Fluid loss is the amount of fluid lost into the formation. In filtration loss test, filter paper is used as the membrane to filtrate the WBM containing different amount of nanosilica. Therefore, the filtration volume in this test is incapable to reflect the condition in real shale formation because the pore size of filter paper does not represent the pore size of shale formation. The pore size of filter paper is in micron scale which is relative large compared to usually nano-scale pore size found in shale formation. Theoretically, nanosilica can pass through this micro-pore in filter paper and thus the plugging performance is not very effective for this test. From figure 23, as the amount of nanosilica is increased, the total filtration volume will be less. Based on the figure 23 also, the trend or curve shape of filtration volume is almost the same for all samples. This indicates that the nanosilica takes a while to plug the pore throat and effectively prevent the filtration loss.

4.3 Shale Adsorption Test Analyses

Shale expansion or swelling occurs when there is adsorption of water particle by the clay layers which result in increases in mass and decreases in shale strength. The purpose of this test is to analyze the plugging effect of different nanosilica concentration in water based mud (WBM). The effectiveness of nanosilica particle to prevent invasion of water component into the shale core samples can be obtained from this test.

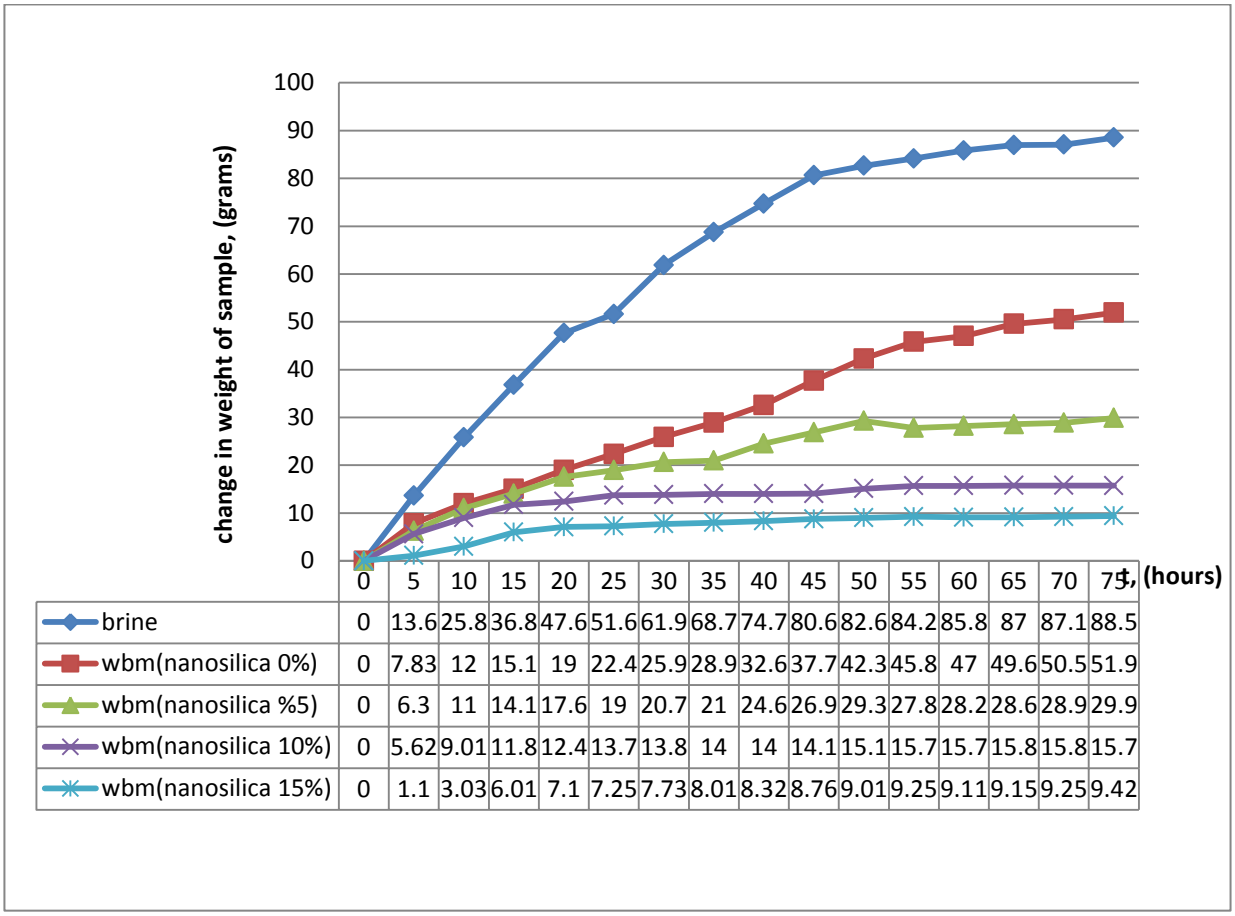
Five Seri Iskandar shale samples that have been prepared beforehand are used in this test. The samples exhibit more or less similar petrophysical properties. The samples are then tested with brine and four samples of WBM with nanosilica concentration of 0%, 5%, 10%, and 15% respectively. Each test is run for 75 hours and the mass of the sample is weight for every five hours. Figure below shows the result of the test.



t, (Hours)	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75
brine	813	826	838	849	860	864	874	881	887	893	895	897	898	899	900	901
wbm(nanosilica 0%)	796	804	808	811	815	819	822	825	829	834	839	842	843	846	847	848
wbm(nanosilica 5%)	809	816	820	823	827	828	830	830	834	836	839	837	838	838	838	839
wbm(nanosilica 10%)	811	817	820	823	824	825	825	825	825	825	826	827	827	827	827	827
wbm(nanosilica 15%)	789	790	792	795	796	796	797	797	798	798	798	798	798	798	798	799

Figure 25: plugging effect with different concentration of nanosilica

Figure below show the change in mass of each test with different concentration of nanosilica.



	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75
brine	0	13.6	25.8	36.8	47.6	51.6	61.9	68.7	74.7	80.6	82.6	84.2	85.8	87	87.1	88.5
wbm(nanosilica 0%)	0	7.83	12	15.1	19	22.4	25.9	28.9	32.6	37.7	42.3	45.8	47	49.6	50.5	51.9
wbm(nanosilica %5)	0	6.3	11	14.1	17.6	19	20.7	21	24.6	26.9	29.3	27.8	28.2	28.6	28.9	29.9
wbm(nanosilica 10%)	0	5.62	9.01	11.8	12.4	13.7	13.8	14	14	14.1	15.1	15.7	15.7	15.8	15.8	15.7
wbm(nanosilica 15%)	0	1.1	3.03	6.01	7.1	7.25	7.73	8.01	8.32	8.76	9.01	9.25	9.11	9.15	9.25	9.42

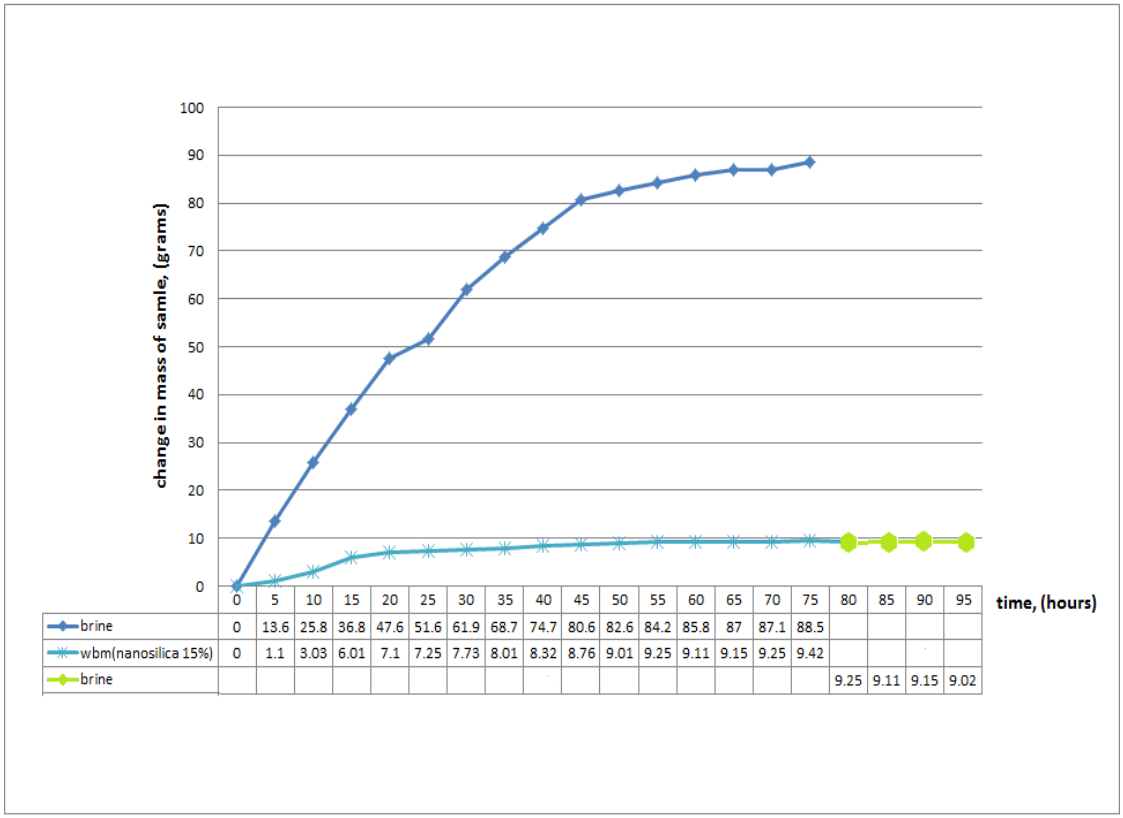
Figure 26: plugging effect with different concentration of nanosilica

As the tested liquid passes through the shale samples, some component of water particle is been adsorb. This will cause the shale to swell and increase the weight of the samples. Therefore, the change of mass of the samples indicates the rate of the adsorption of water particle

During the first test, brine with 4% NaCl concentration is used as tested liquid. The brine solution was tested with shale samples for 75 hours. For every 5 hours, the valve is closed to stop the flow for weight of the sample to be taken. From both figure 25 and 26, it can analyze, from the beginning, the increment in sample weight occurs rapidly which indicates adsorption of water particle occurs very fast. However, after flowing the brine solution for 65 hours, rate of change in weight of the samples started to become low. This could possibly indicate that the osmotic pressure differential is no longer high enough to force the water to be adsorbed by shale samples.

After completing the first test, brine was displaced with formulated drilling fluid (WBM) containing different concentration of nanosilica. From both figure, it can be observed as the concentration of nanosilica was increased from 5% to 15%, an increase in plugging properties was observed thus reducing the adsorption of water particle. With only addition of 5% concentration of nanosilica to WBM, it has shown significant reduction in increment of shale sample weight compare to WBM without nanosilica. However, the amount of nanosilica for 5% concentration is far too less in or to sustainably plugs the shale sample. Due to that, some increment shale sample weight can be observed even after 75 hours because the sample is not properly sealed by small amount of nanosilica. On the other hand, it is also noticed that after 50 hours, both WBM with concentration of 10% and 15% did not registered any increment in changes of sample weight. This suggests that the shale sample has been properly sealed. The comparison between 10% and 15% nanosilica concentration is that the time taken for nanosilica to sealed the shale sample properly. It can examine from figure 1 and 2, shale sample for 10% nanosilica concentration registered high rate of changes in weight in the beginning compare to 15% nanosilica concentration. This meant that concentration of nanosilica also affect the time for plugging effect occurs. From the test, WBM with nanosilica concentration of 15% shows the most effective plugging performance. However, such dosage of 15% would not be practical in oil and gas fields.

After successfully run the test for formulated WBM with nanosilica concentration of 15%, the same sample is continued to be tested by replacing the WBM with brine solution. Figure 3 below show the result of the test.



	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95
brine	0	13.6	25.8	36.8	47.6	51.6	61.9	68.7	74.7	80.6	82.6	84.2	85.8	87	87.1	88.5				
wbm(nanosilica 15%)	0	1.1	3.03	6.01	7.1	7.25	7.73	8.01	8.32	8.76	9.01	9.25	9.11	9.15	9.25	9.42				
brine																	9.25	9.11	9.15	9.02

Figure 27: sustainable plugging mechanism of nanosilica

From figure 27, there was almost no change in mass that registered even after the WBM is replaced with brine solution. This meant that the shale sample had been physically plugged and that the plugging effect was sustainable.

Table 10: change in weight of shale sample reduction

Test	Flowing fluid	Change in weight of sample after 75 hours, grams(g)	Change in weight of sample after 75 hours reduction, %
1	4% NaCl Brine	88.5	-
2	WBM (0% nanosilica)	51.9	41.4%
3	WBM (5% nanosilica)	29.9	66.2%
4	WBM (10% nanosilica)	15.7	82.3%
5	WBM (15% nanosilica)	9.42	89.4%

Table 10 shows the reduction in rate of change in shale sample weight after 75 hours. From the calculated result, 89.4% reduction was achieved using the formulation under study as showed in table 1. This significant reduction in rate of change of sample weight reflects substantial reduction in permeability of shale sample due to excellent plugging with the WBM. With this improved designated WBM, very little water particle manage to invades into the shale, preventing problems such as shale swelling in the field. Therefore, Shale stability can be effectively maintained during drilling operation with this WBM.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The primary objective of this project is to produce an improved WBM that is simple in formulation and maintenance shows excellent rheological properties, maintain wellbore stability, and good environmental profile has been successfully studied. A combination of conventional and economically affordable material is combined with new nanoparticle material which is produced from common source such as siliceous sand is used to achieve desired rheological properties and wellbore stability in shale formation.

This project has also effectively achieved the secondary objective which is to produced nanosilica that exhibit optimum particle size which is suitable for improved WBM plugging mechanism in shale formation. A proper size of nanosilica has been produced from very common source such as siliceous sand. This suggests that a mass production of nanosilica for industry application is achievable.

From this project also, the effect of nanosilica concentration on rheological properties has been studied. The results show that tha WBM exhibit excellent rheological properties and suitable for shale formation drilling operation.

The existence of nanosilica in water-based mud has proved to provide effective plugging effect in shale formation. The designated WBM significantly reduce the adsorption of water by shale formation. This major improvement will prevent the event of shale swelling during drilling operation and thus successfully maintain the wellbore stability in shale formation.

5.2 Recommendation

In order to improve the results and possible outcomes of this study, some recommendations need to be done. This recommendation will help to increase the depth of understanding in wellbores stability problems in shale formation faced by drilling engineers. In this project, many important parameters such as temperature and drilling mud pressure have been neglected. Therefore, in future works, perhaps by taking account those parameters; an improvement in this matter can be obtained.

To tell the truth, there are many limitations when conducting this project. Obstacles such as malfunction of crucial equipment will give huge impact on project outcome. The initial plan on this project is to used formation damage tester which capable of analyzing the relationship between fluid pressure and fluid loss into formation. However a change has to be made as the equipment from the given facilities was failed to function properly. By solving this matter in future, more accurate and various data can be obtained to improve the project outcome.

Others recommendation also include testing different type of nanoparticle and analyze its plugging effect in shale formation. There are many ways also to produce nanosilica besides from siliceous sand. For instance, nanosilica can also be produced from reaction of olivine rocks with sulfuric acid. In future study, it will be good to compare the characterization of this nanosilica from different type source and analyze its plugging performance in shale formation.

CHAPTER 6

REFERENCES

1. Chen, G., et al., A study of wellbore stability in shales including poroelastic, chemical, and thermal effects. Journal of Petroleum Science and Engineering, 2003. 38(3): p. 167-176.
2. A. Lázaro, H. J. H. Brouwers (2010), Nano-silica production by a sustainable process, 8th fib PhD Symposium in Kgs. Lyngby, Denmark
3. Belyakova, L. A.& Varvarin, A. M. (1999). Surfaces properties of silica gels modified with hydrophobic groups. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 154(3), 285-294.
4. Amutha, K., Ravibaskar, R.& Sivakumar, G. (2010). Extraction, synthesis and characterization of nanosilica from rice husk ash. Journal of Nanotechnology and Applications, 4(1), 61-66.
5. Jal, P. K., Sudarshan, M., Saha, A., Patel, S.& Mishra, B. K. (2004). Synthesis and characterization of nanosilica prepared by precipitation method. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 240(1-3), 173-178.
6. Al-Bazali, T.M., Zhang, J., Chenevert, M.E., Sharma, M.M.:" Factors Controlling the membrane efficiency of shales when interacting with water-based and oil based Muds." SPE 100735, the 2006 SPE international oil &Gas conference and exhibition in China, Beijing, China, 5-7 December 2006.
7. Bol G. M. et al, "Borehole stability in shales", SPE 24975 presented at European Petroleum Conference held in Cannes, France, 16-18, November 1992.
8. Chenevert M. E: "shale alteration by water adsorption", JPT (sept 1970), pp 1141-1147.
9. Van Oort, Eric: "On the physical and chemical stability of shales', Journal of Petroleum Science & Engineering, 38 (2003) 213-235.
10. Ballard T. J. Beare S. P. and Lawless T. A.: "Fundamental of Shale Stabilization: Water Transport Through Shales", IADC/SPE24974, presented at the European Petroleum Conference held in Cannes, France, 16-18 November, 1992.

11. Van Oort, E.: "physico-chemical Stabilization of Shales", SPE paper 37263 presented at 1997 International Symposium on Colloid Chemistry, Houston, Texas, USA, Feb. 18-21.
12. Hale, A. H., Mody, F. K. and Salisbury, D. P.: "Experimental Investigation of the Influence of Chemical Potential on Wellbore Stability", IADC/SPE paper 23885, presented at the 1992 IADC/SPE Drilling Conference in New Orleans, Lousiana, Feb. 18-21, 1992.
13. Steiger, R. P. "Advance Triaxial Swelling Test On Preserved Shales Cores", presented at the 34th U. S. Symposium On Rock Mechanics (June 27-30, 1993) and to be published in the Int. J. Rock Mech. Min. & Geomech. Abstr. (1993)
14. Santarelli, F. J. and Carminati, S.: "Do Shale Swell? A Critical Review Of Avaible Evidence", SPE/IADC 29421, presented at the 1995 SPE/IADC Drilling Conference held in Amsterdam, 26 February- 2 March 1995.
15. Carminati, S., Brignoli, M., Marco Di A. and Santarelli, F. J.: "The Activity Concept Applied To Shales: Consequence For Oil, Tunneling And Civil Engineering Operation", Int. J. Rock Mech. & min. Sci. 34, paper no. 038. 1997.
16. Norrish K.: "The Swelling of Montmorillonite", Disc. Of Faraday Soc., V.18, P.120, 1954.
17. Benjamin herzhaft, Y. P. (2001). Rheological Properties of Drilling Muds in Deep Offshore Conditions. Amsterdam, The Netherlands: SPE/IADC Drilling Conference.
18. ASME Shale Shaker Committee, (2005) Drilling Fluid processing handbook
19. Keith, S. (1971). Mechanics (Third Ed.). Addison-Wesley. ISBN 0-201-07392-7
20. Jetjongjit, R. (2010). Plastic Viscosity (PV). http://www.drillingahead.com/profiles/blogs/plastic-viscosity-pv
21. Canon, F. (1999). Drill Master: Drilling fluids Properties