Experimental Investigation of the Effects of Size and Concentration of Nanoparticles on Shale Stability

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Dissertation submitted in partial fulfilment of the requirements for the Bachelor of Engineering (Hons) (Petroleum Engineering)

September 2013

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CERTIFICATION OF APPROVAL

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BACHELOR OF ENGINEERING (HONS)

(PETROLEUM ENGINEERING)

Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH,PERAK

September 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contain herein have not been undertaken or done by unspecified sources or persons.

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(NUR FATHIN MUNIRAH BT MOHD AZMAN)

ABSTRACT

This paper presents the experimental investigation of the effects of size and concentration of nanoparticles on shale instability. Nanoparticles are added to conventional water-based drilling fluid and are tested on shale to test for its stability. Stability here means the shale will not disintegrate into pieces when in contact with water as the cuttings will cause downhole completion to stuck. Nanoparticles with different sizes and concentrations are used in this experiment to see their effects in providing the best nano-based drilling fluid to enhance shale stability.

Shale dispersion is a process by which shale cuttings disintegrate into smaller size usually described as fines. Dispersion of shale cuttings occur when it is in contact with water. It contributes to the wellbore instability as the cuttings have tendency to cause downhole completion stuck. Shale's dispersive abilities vary depending on the fluid in contact with it. A fluid will successfully inhibit dispersion by preventing the shale from breaking into smaller pieces. In this project, dispersion test were performed to investigate the inhibitive properties of the prepared drilling fluid with the shale samples. The drilling fluid's ability to maintain the cuttings integrity was analyzed. Thus, at the end of this project, a sample of drilling fluid with good inhibitive property will be concluded.

To come out with the correct fluid, the author has used nanoparticle as the inhibitive agent. This is due to its small size, which is said to be effective in plugging the shale pore throat. When nanoparticle successfully plugged the pore, the shale will regain its strength and have fewer tendencies to disintegrate into smaller size. Two different sizes of nanoparticle will be tested in the experiment to check which size is more suitable to plug the shale pore throat. Different concentration of nano-based fluid also been used to determine the correct concentration for the inhibitive properties to be effective.

Hot rolled dispersion test has been conducted for this project. Six drilling fluid formulations with different concentration added with different size of nanoparticle have been used. The shale cuttings was hot rolled together with the drilling fluid in a roller oven for 16 hours at 66^{o} C. Based from the hypothesis, shale stability could be reduced to the maximum if it is being hot rolled in 10 wt % and 10-15 nm nanoparticle added nano-based fluid. From the experiment, it is proved that shale dispersion could be reduced in higher nano-based concentration. However, the result does not give convincing value to test for the size variations.

ACKNOWLEDGEMENT

I would like to take this opportunity to express my profound gratitude and deep regards to my supervisor, Dr. Masoud Rashidi for his guidance, monitoring, and constant encouragement throughout the course. The help and guidance given by him time to time shall carry me a long way in the journey of life on which I am about to embark.

I also want to take this opportunity to express a deep sense of gratitude to laboratory technicians for their support, valuable information and guidance, which helped me in completing this task through various stages. I am grateful for their cooperation during the period of my assignment.

Lastly, I would like to thank my parents and friends for their constant encouragement without which this project would not be possible.

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CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

About 75 % of reservoir formation account for shale and 90 % of shale are responsible for wellbore instability problem. Wellbore instability problem originated from shale is a common phenomenon among drilling engineers. Shale which exhibit low permeability has caused various kind of damage that lead to wellbore becomes instable. Water invasion from wellbore into shale formation can cause swelling and loss of water from shale can result in hydration. These two problems are significant when shale is exposed to the water-based drilling fluid.

To avoid interaction between shale and water, of course, oil-based drilling fluid has superior result rather than water-based drilling fluid. However, the concern for oil-based drilling mud as a replacement over water based fluid has generated many negative impacts. Oil-based drilling fluid has been proved to give higher cost and more pollution towards environment. Because of that reasons, water-based drilling fluid is still preferable to be used as drilling mud. Little modifications on water based drilling fluid might help engineers to encounter problems arise.

Among the ways developed in order to modify water-based drilling fluid to be used effectively in drilling activity may include adding some additives. Carminati et. al. (2000) showed that the most effective additives in controlling the pore-fluid pressure in the formation and shale hardness, thus preventing shale instability is the silicates. As long as the pore pressure in the formation is kept at higher level, there will be no invasion of fluid from wellbore into shale. Cheng Fa Lu once stated in his research paper that by adding KCL in water-based drilling fluid, hydration of clay particles can be reduced. Moreover, sodium ion in montmorillonite shale, the type of clay particle which absorb plenty of water can be replaced by potassium ions which later can result in tight, hard shale.

Due to small pore throat and extremely low permeability exhibit by shale, none of this specially engineered drilling fluid added with additives seems to be of help. Range of shale pore size does not seem compatible with the size of conventional additives particle for them to form mudcake when in contact. Mud cake is important as it act like a filter to prevent excessive water invasion into the formation. Al Bazali et al. (2005) found that average pore throat sizes of a variety of shale range from 10 to 30 nm, which is way smaller than commonly used drilling fluid additives particle diameters which is in the range of 100 to 10000 nm. To form mudcake, shale pore throat has to be plugged by smaller particles.

Researchers have come out with a solution to this problem. It is said that nanoparticle can be the best option in order to effectively plug the pore throat and form bridging across the shale. Nanotechnology represents the development and application of materials, methods, and devices in which the critical length scale is on the order of 1 to 100 nm. The use of nanoparticles in drilling fluid turns out to be the first potential large scale application of nanoparticles in the oil and gas industry. Experts believe that nanotechnology will someday bring huge earnings to the industry if they are developed and invested properly (Pourafshary et al. 2009).

There are ranges of nanoparticle size that need to be identified to see its compatibility in becoming a good plugging agent. Abrams (1977) proposed that for the particle to effectively plug the pore throat, it should be equal to or slightly greater than one-third of the median pore size of the formation, plus the concentration of bridging size solids must be at least 5% by volume of the solids in the final mud mix. A laboratory experiment was conducted to prove this theory.

In this laboratory experiment, the effects of SiO_2 nanoparticle mixed with water-based drilling fluid on shale stability were investigated. Due to the easy access and abundant amount of silica nanoparticle, it was chosen as the main particle needed to plug in the shale pore throat. Though nanoparticle has been proved technically as a small particle, due to wide range distribution of shale pore size in nanoscale, the size of nanoparticle is taken as the manipulative parameter in this experiment. The concentration of nanoparticle added into water-based drilling mud also varies. Both, the size of nanoparticle and its concentration are considered as important parameter in choosing the best nano-based drilling fluid to block the pore throat.

SiO_2 nanoparticle in the range of 5-15 nm and 20-30 nm with the concentration of 2, 5, and 10 wt % were used. Hot rolled test was conducted to see and checked the stability of shale cutting after it was rolled with nano-based drilling fluid.

1.2 PROBLEM STATEMENT

Shale was proven as a major contributor towards wellbore instability. There are controllable as well as uncontrollable factors of wellbore instability which physico-chemical interaction of shale and drilling fluid falls under the controllable factor. Many researchers have come out with various attempts to solve this problem as the loss from wellbore instability gives huge effect towards production.

Basically, shale stability problem arise from the imbalance shale stresses with its strength. As shale buried deep down the formation, it is subjected to in situ stresses and pore pressure. At this time, equilibrium exists between the stress and strength. When drilled, altered stress environment affect the shale condition. Shale also exposed to the sudden drilling fluid which could induced the equilibrium possessed by shale strength and stress. The imbalance between strength and stress of shale can be due to the density difference own by the drilling fluid. When shale and fluid interacts, strength is altered as well as pore pressure adjacent to the borehole wall. As fluid enters shale, pore pressure increase while shale strength decreases.

An analysis of the available data obtained from O'Brien-Goins-Simpson Associates, fluid-shale interaction indeed causes and alters the shale strength and pore pressure near the bore hole. The result of the analysis shows that activity imbalance causes the movement of fluid inflow and outflow from the shale, how the content of the drilling fluid (amount of additives added) may affect the inflow/outflow of the fluid and how fluid flow into shale results in swelling pressure.

Instability develops by shale come to existence when stress exceeds the strength. Obviously, one needs to work out on balancing the new stresses and strength featured by the shale. Attention need to be subjected to the development of drilling fluid. Supposedly, the interaction between the drilling fluid and shale should be minimize to prevent the alteration of stresses but oil-based drilling fluid, the most suitable fluid to be used have many concerns for its usage. As a matter of fact, though oil-based fluid may excel in minimizing the interaction, it is restricted by many parties due to environmental regulation and economic concern. By any means, water-based fluid has to be used instead of oil-base fluid. To accomplish the objective of reducing the interaction between drilling fluid and shale, methods in altering water-base drilling mud has now become a crucial experiment among the researchers.

The modification of water-base drilling fluid includes adding additives to enhance the rheology of fluid. Polymers are added to adjust mud rheology and to perform bridging purposes between particles in shale. In drilling activity carried out at sand formation, drilling fluid will filtrate into formation pores and gradually plugged the pores with its particle.

Mudcake will form in this condition and act like a filter towards the filtrate coming in. Filtration can be reduced with the presence of mudcake. As in the shale formation, with its low permeability attribute, mudcake is form with ultra-low filtration. This happen because shale requires the match between the pore throat size and the plugging material. Small pore throat size exhibit by shale makes it difficult for bigger particle in conventional water-base fluid to plug the pore and form mudcake. Preventing filtration in this case will be harder.

The development of nanoparticle saves the invention. With its small size range, nanoparticles are chosen as the most suitable material to plug the shale pore throat. Nanoparticle will plug the tiny shale pore throat and formed tight mudcake. When the pore throat has been plugged, the inner strength of the shale will improve. Thus, the shale will not disintegrate into smaller pieces. Apart from testing the shale swelling, due to many constraints, this experiment will test the rate of shale dispersion by using hot rolled dispersion test. In this experiment, nanoparticle with different size and concentration will be added into water-base fluid to study its affect in shale stability. It is proved by the lab experiment that nanoparticle with small size and high concentration is effective in improving shale stability.

1.3 OBJECTIVE AND SCOPE OF STUDY

Main objectives of this project can be found as below;

i. To examine how nanoparticle-based drilling fluid can reduce shale dispersion.
ii. To show that wide range of nanoparticle size can affect the result of experiment.
iii. To find the correct nanoparticle concentration in plugging shale pore throat thus could reduce the shale dispersion.

The project will be conducted fully in laboratory. The focus will be on how to examine the shale dispersion after it is soaked with drilling nanoparticle based drilling fluid. The procedure of the experiment was based on the steps enclosed by American Association of Drilling Engineers. Hot Rolled Test or Dispersion Test was conducted to test the effectiveness of nano-based fluid on maintaining shale instability.

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CHAPTER 2: LITERATURE REVIEW

2.1 WELLBORE INSTABILITY

Wellbore instability is one of the major problems faced by drilling engineers in oil and gas industry. Engineers recognized this problem when there is a dissimilar hole diameter down hole with drill bit size. Other direct indicators of wellbore instability instead of oversize hole can be excessive volume of cuttings received at the surface, cavings at surface, and excess cement volume required.

Wellbore instability is caused by various reasons and usually these reasons are divided into mechanical effects and chemical effects. Mechanical effect can be either failure of the rock around the hole because of high stress, low rock strength, or inappropriate drilling practices, while chemical effects arise from damaging interaction between the rock (generally shale) and drilling fluid.

Uncontrollable factors that give to wellbore instability commonly originated from the formation problems. Naturally faulted or fractured formations contain loose pieces in which if the pieces fall into wellbore, it can jam the string in the hole. Tectonically stressed formations and high in-situ stresses also can result to wellbore instability. When a hole is drilled in an area with high stresses, the rock around the wellbore will collapse into the wellbore and produce splintery cavings. The bottom line of the problem is forces acting in the formation can push the wall of the hole inward. The hole will collapse if there are no precautions taken immediately.

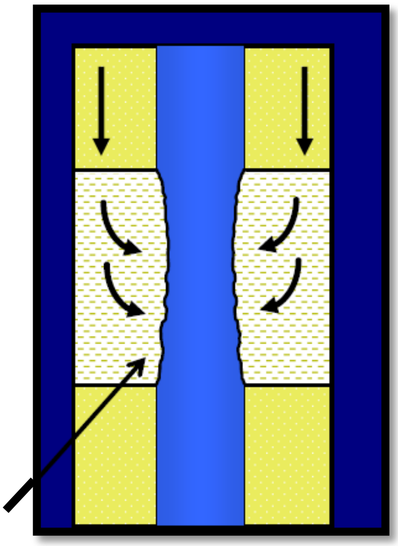


Figure 1: Forces acting on the wellbore wall

2.2 SHALE INSTABILITY

2.2.1 Poroelasticity and thermal effects

Shale failure primarily caused by the redistribution of in situ stress which exceeds the shear or tensile strength of the rock. Poroelastic effects are discussed under the condition of undrained and drained situation (Chen G. et. al., 2003). Shale which exhibit low permeability attribute is said to face short exposure times under undrained condition. The formation can "feel" the boundary effects and transient penetration will occur between the formation and the wellbore. Heat conduction dominates the heat transfer process in a low permeable porous medium like shale (Wang and Papamichos, 1994). But then the effect of heat convection is neglected because of the extremely low fluid velocity in shale. Charlez, 1997 come out with the finding where rock stresses are also affected by thermal diffusion between the drilling mud and the formation.

These two effects; poroelasticity and thermal are then being incorporated into an equation. Kurashige (1998) merged heat transport into Biot's poroelastic theory and come out with thermoporoeleastic theory for fluid-filled porous materials. Study from Guizhong Chen et. al. later shows that pore pressure can be partially decoupled from temperature for shale formation. Wang and Papamichos (1994) presented solutions for partially decoupled equations for temperature and pore pressure. Under appropriate initial and boundary conditions, the partially decoupled equations can be solved analytically.

2.2.2 Physical-chemical fluid rock interaction

Wellbore instability also can be caused by physical/chemical fluid-rock interaction. There are many physical/chemical fluid-rock interaction phenomena which can modify near-wellbore rock strength or stresses (Pasic et. al., 2007). These include hydration, osmotic pressures, swelling, rock softening, and strength changes and dispersion. It is proved that shale; common sedimentary rocks which make up 75% of drilled section contributes to 90% of wellbore instability problems.

Theory developed by Ghassemi A. and Diek A. considers chemical and poroelastic processes and couples ion transfer in the mud/shale system to formation stresses and pore pressure. They are using linear, Biot-like isotropic poroelastic theory to derive the field equations needed. The solution they have developed considered studying the impact of solute transfer on stress and pore pressure fields. The result of the analyses shows that solute transfer causes the chemical-osmosis to become time-dependent. The transfer of ions causes osmotic pressure dissipation and re-establishment of a pore pressure regime characteristic of hydraulic flow. On top of that, it is noticed that the rock can experience radial stresses resulted by the physicochemical interaction when in contact with drilling fluid.

2.2.3 Total aqueous potential vs. swelling effect

Total aqueous potential (pressure and chemical potential) of the pore fluid increase when pore pressure increase. Pore pressure can be increased due to mud pressure penetration and osmotic inflow. Osmotic inflow (flow from wellbore into the formation) takes place when the activity of drilling fluid is higher (low salt concentration) than the shale activity. Mud pressure would penetrate progressively into the formation when drilling activity was conducted under overbalance condition.

Due to the increase in total aqueous potential of the pore fluid, water will be absorbed into the clay platelets. If the platelets are free to move, the absorption will cause the platelets to move further apart and thus result in swelling. If the platelet movement is restricted or constrained, hydrational stress will be generated. Forces in clay which involve the well known van de Waals forces and double layer repulsion also give rise to hydration pressure. Both, the increase in pore pressure and a reduction in mud support will lead to less stable wellbore condition.

2.2.4 Tests conducted on shale

2.2.4.1 Water distribution analyses using X-ray diffraction

.Water distribution is important in shale stability as it affects the degree of clay swelling. X-ray diffraction technique is used to establish the composition and swelling behavior of the shale. In a paper develop by Perez A.D. et.al, seven samples of shale from different oil field were tested for their composition and swelling behavior which later result with this conclusion; the role of activity in the swelling behavior of shale only take place at lower temperature. Thermal gravimetric (TG) analyses also aid in this research. Below is the diagram of TG analyses made to the shale sample.

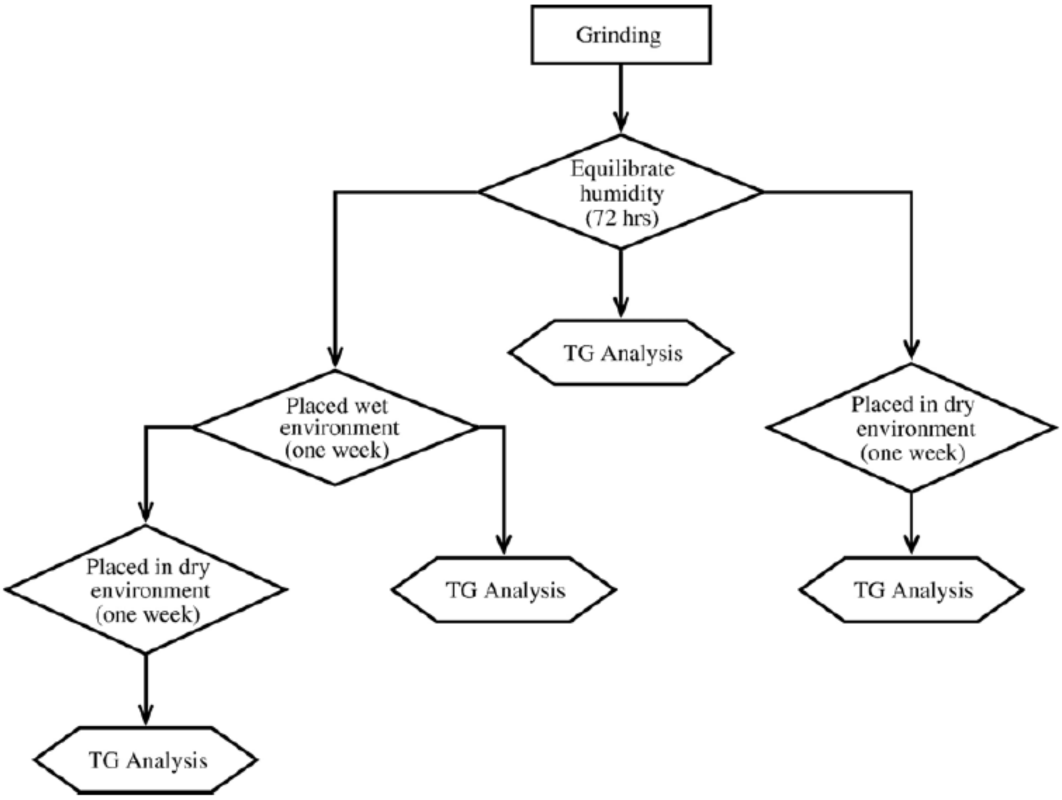


Figure 2:TG Analyses

There are two groups of shale preparation procedure for the X-ray diffraction test. One in which the goal is to obtain perfectly random grain orientation and the other one in which perfect orientation of the clay minerals flakes parallel to the substrate is the goal. The goal in this study is to obtain the oriented one. Natural clays have hydrophilic surfaces that can absorb water and some ionic particles. Due to this, when shale is in contact with water based drilling fluid, swelling takes place. Results of the analyses show that the identification and quantification of groups and subgroups of smectite play an important role in swelling behavior.

2.2.4.2 Cations Exchange Capacity (CEC)

Cations are positively charged ions such as calcium (Ca_{2+}), magnesium (Mg_{2+}) and potassium (K_{+}). CEC is the capacity of the soil to hold on to these cations (Ketterings et. al.). The cations are held together by the negatively charged clay and organic particles in the soil through electrostatic forces. CEC contain in soil because clay particles and organic matter in the soil tends to be negatively charged. The mineral would not have any charge when it contains only silica and oxide.

Soils in New York contain aluminums as well as silica. They have negative charge because of the substitution of silica by aluminums in the mineral structure of the clay. This substitution result in clays with negative surface charge. The negative charge of the clay is balanced by the positive charge of the cations in the soil.

CEC test as per recommended by API, uses methylene blue to test for the CEC value. The higher the CEC the more clay or organic matter present in the soil. It also indicate that high CEC (clay) soils have a greater water holding than low CEC (sandy) soils.

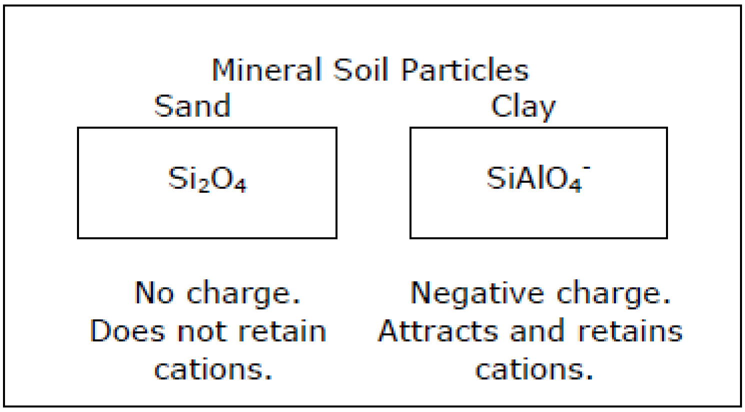


Figure 3: Substitution of silica by aluminum in soil clay particles causes clays to have a negative charge. Because of this negative charge, the soil can hold on to positively charged cations such as calcium (Ca2+), magnesium (Mg2+) and potassium (K+).

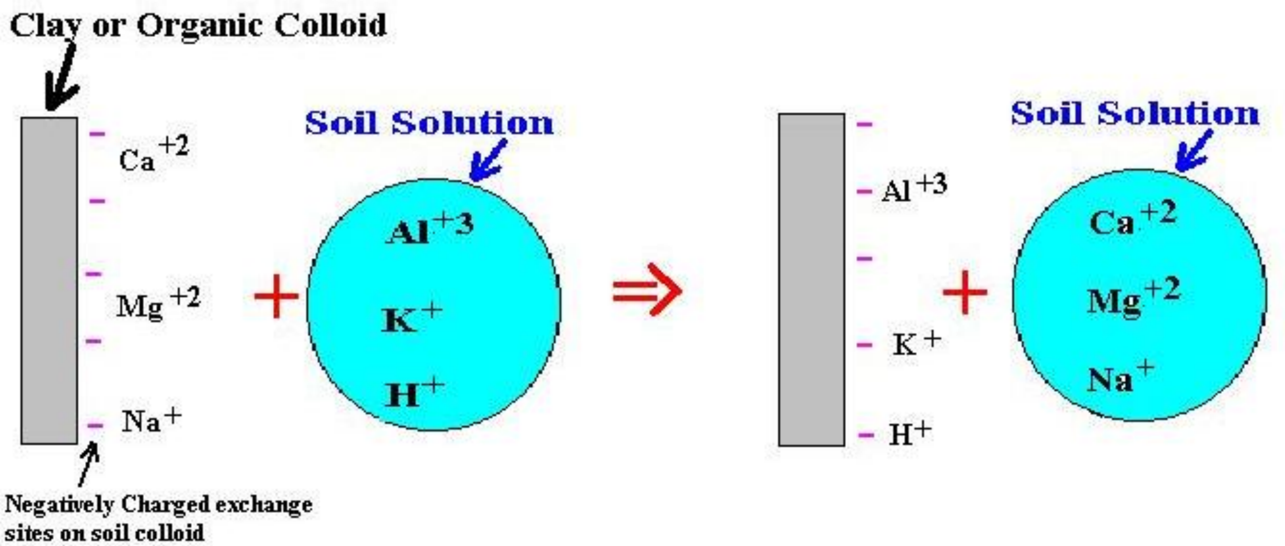


Figure 4: Cation Exchange Capacity mechanism

2.3 METHODS TO CORRECT SHALE INSTABILITY

The presence of semi-permeable membrane at the interface between drilling fluid and shale act as a controller to the ion movement. The membrane inhibits the movement of ions from one component to the other. From the research conducted by Chenevert (1970), certain oil mud does provide good inhibitive properties for the ionic movement.

To induce the interaction between shale and water, oil-based drilling fluid has been used instead of water-based. But due to the environmental problem and economical consideration, oil-based drilling fluid has been restricted by the responsible party. Thus, it is preferred to use the existing water-based drilling fluid with low interaction between the fluid and the shale. Modifications and alterations toward the drilling fluids are very much needed in order to minimize the interaction.

Bridging purposes is when the solids are added to a drilling fluid to bridge across the pore throat thereby building a filter cake to prevent loss of whole mud or excessive filtration. As shale exhibit low permeability, filtration rate (rate of fluid invasion) will be lower. In this situation, mud cake is more difficult to be formed. When filtration continues, pore pressure will continually increase because there is no mud cake to reduce filtration. Eventually, pore pressure will be equal to the hydrostatic pressure provided by drilling fluid column, which suppose to support borehole. When there is lack in support, borehole become instable. Hence, it is to be noted that bridging is important in enhancing drilling fluid.

Researchers come out with various methods to correct shale instability problems. Among the resolving methods would be increasing drilling fluid inhibition, improving the quality of drilling mud cake, and sealing the shale pore throat. Adding polymer in drilling fluid can adjust the mud rheology and is one of the most used methods to improve drilling fluid. In one paper presented by Cheng Fa Lu for SPE publication, KCL/ polymer mud are designed specifically to minimize the instability problem. Potassium ions in KCL can replace the sodium ions contain in sodium montmorillonite shale through cationic exchange and result in tight, hard shale. Polymers too can form polymeric bridging between particles in shale which also important in shale inhibition.

A journal paper developed by Ismail I. and Huang A.P from Universiti Teknologi Malaysia has formulated Methyl Glucoside (MEG) drilling fluid as shale inhibitor. MEG is introduced into sodium chloride mud to control shale hydration and dispersion. This formula is said to possess similar performance with oil-based drilling mud. Based on the experimental results obtained, it is proved that MEG fluid can improve shale stability with regards to different concentration react with different shale reactivity and clay content in shale.

Different concentration of drilling fluid can become the main factor in determine whether the shale will be dehydrated or swell. Osmosis is the flow of solvent (water) from a solution containing low concentration of solute (salinity) into a solution of higher solute concentration through a membrane that is permeable to the solvent, not to the solute (Tan P.C. et. al. 1997). Osmotic inflow is the term used to describe the flow from the wellbore to formation while osmotic backflow is the flow from formation to wellbore. When the activity of drilling fluid is higher (low salt concentration), osmotic inflow will occur. Water will penetrate from the wellbore into the formation thus cause clay swelling. That is why in most experiments, in order to prevent water invasion from wellbore (from water drilling based fluid) to shale, managing its fluid concentration is considered as one of the important factor that will affect the result.

In a research journal published in 1995 written by Stamakis E. et al, a new application of unique organic materials (OCM) has been proven to efficiently inhibit swelling of shale. The former cationic polymer system turns out to be the most successful inhibitor approaching the inhibition depicted by oil- based drilling fluid. Inhibitive cationic polymer works in a mechanism of exchangeable cations in clay mineral lattices replace by the cationic additives. The reaction electrostatiscally binds the clay platelet together, reducing the tendency of water absorption by platelets and thus can reduce swelling in shale. OCM have excellent inhibiting characteristics where it is water soluble, have very low toxicity, biodegradable, functioning in all common drilling fluids pH levels, compatible with all common drilling fluid additives, and are stable to temperature in excess of 400 deg celcius. These properties make OCM as an excellent inhibitor in improving shale stability.

2.4 NANOPARTICLE TECHNOLOGY

Nanoparticle is widely used in numerous area including material sciences, medical, cosmetic, sports, and recreational markets. Nanoparticle increasing development can be seen by the number of publications depicted by the Graph 1 (Rao J.P. and Geckeler K.E.,2011) The evolution of nanoparticle in oil industry, specifically in drilling fluid modification is somewhat new and fresh. Past years has showed mud engineers been struggling in modifying the drilling fluid with the help of bentonite and other colloidal solids but in the end nanotechnology has found to be the best solution to be added into drilling fluid.

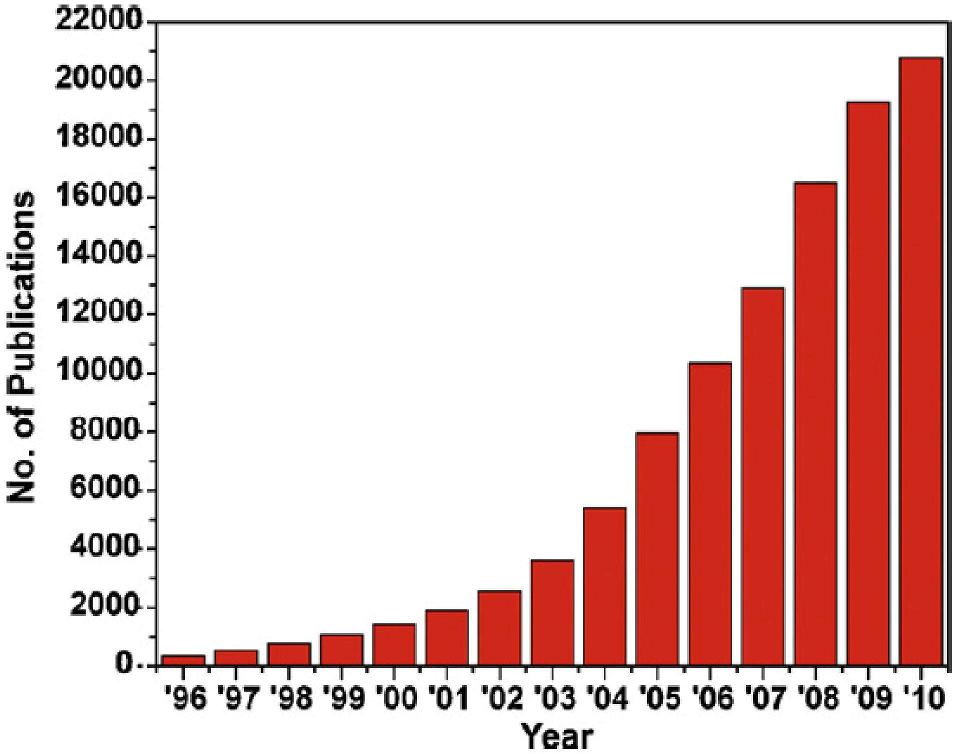


Figure 5: The number of publications cited in Scopus® database on polymer nanoparticles during the period of 1996–2010

2.4.1 Preparation techniques

Nanoparticle used as an additive in drilling fluid. It is said to bring benefits in the area of rheology, fluid loss or shale stability in drilling fluids. Polymer nanoparticle is a term refers to any type of nanoparticle but specifically for nanosphere and nanocapsules. Nanosphere are particles whose entire mass is solid and molecules may be absorbed at the sphere surface or encapsulated within the particle. Nanocapsules are vesicular systems, acting as a kind of reservoir, in which the entrapped substances are confined to a cavity consisting of a liquid core (either oil or water) surrounded by a solid material shell (Rao J.P. and Geckeler K.E., 2011). A schematic representation of polymer nanoparticles is shown below;

Figure 6: Ilustration of classification of polymer nanoparticles nanospheres (a), nanocapsules containing oil (b), and water (c)

Polymers nanoparticle can be prepared either by two ways; from preformed polymers or by direct polymerization of monomers using classical polymerization.Solvent evaporation,salting-out,dialysis and supercritical fluid technology are the methods that can be utilized for the preparation of nanoparticles from preformed polymers.On the other hand,by direct polymerization of monomers techniques,nanoparticles can be directly synthesize by the polymerization of monomers using micro-emulsion, mini-emulsion,surfactant-free emulsion and interfacial polymerization technique.Type of polymeric system, area of application, size requirement are the factors affecting the choice of preparation method. An illustration of different preparation techniques for polymer nanoparticles is given as below;

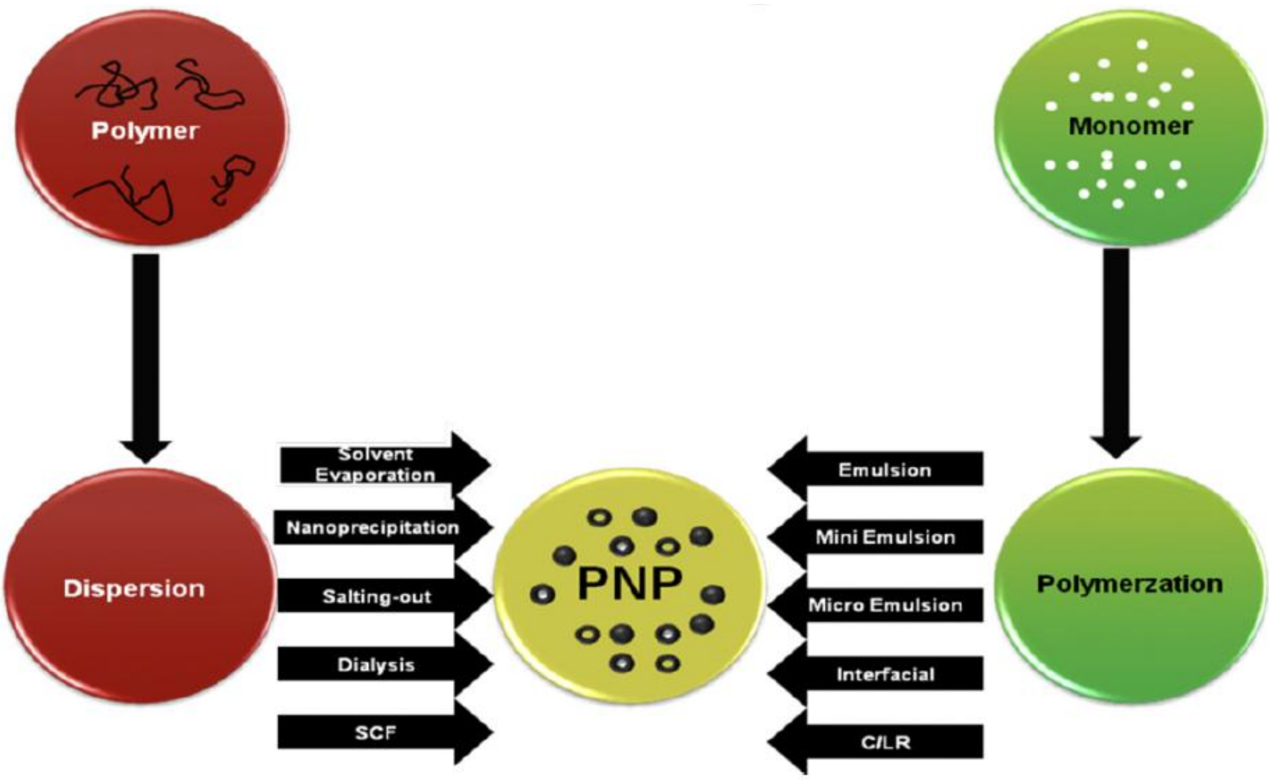


Figure 7: Schematic representation of various techniques for the preparation of polymer nanoparticles

2.4.2 Synthesis of Nanosilica

Three commonly used methods can be employed to synthesize silica nanoparticle. They can be produce through reverse micro emulsion, flame synthesis, and widely utilized sol-gel. In reverse micro emulsion, the formation of spherical micelles by surfactants molecules dissolved in organic solvents is the main point. In the presence of water, the polar head groups organize themselves to form microcavities containing water, which is often called reverse micelles. In synthesis of silica nanoparticles, the nanoparticles can be grown inside the microcavities by controlling the addition of silicon alkoxides and catalyst into the medium containing reverse micelles. High cost and difficulties in removal of surfactant in the final products are the major drawbacks of this method. Another method used in silica nanoparticle synthesizing is high temperature flame decomposition of metal-organic precursors. In this process, silica nanoparticles are produced by reacting silicon tetrachloride with hydrogen and oxygen. The main disadvantage of this technique is difficulties in controlling the particle size, morphology, and phase composition. Still, this is the prominent method that has been used to produce silica nanoparticle in powder form.

Other than the two techniques, sol gel process also can be employ to synthesize silica nanoparticle (Rahman I.and Padavettan V., 2012). The process involves hydrolysis and condensation of metal alkoxides (Si(OR)4) such as tetraethylorthosilicate (TEOS, Si(OC2H5)4) or inorganic salts such as sodium silicate (Na2SiO3) in the presence of mineral acid (e.g., HCl) or base (e.g., NH3) as catalyst. A general flow chart for sol-gel process which leads to the production silica using alkoxides is shown below ;

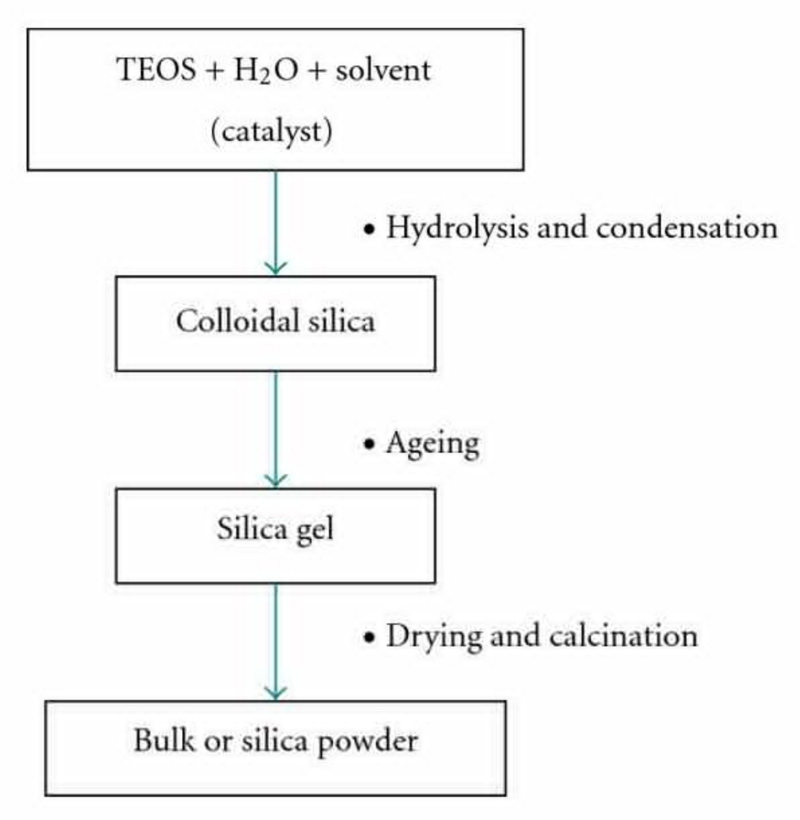


Figure 8 : Sol Gel process

2.4.3 Nanotechnology in drilling fluid

Sealing shale pore throat also gives significant effect towards improving shale stability. Because of the small pore exhibit by shale, size of particle needed in order to seal the pore should be as small as possible. In this case, researchers have been eyeing on the usage of nanoparticle to facilitate the drilling fluid used for drilling in shale section. Nanoparticle is the development and application of materials, methods, and devices in which critical length is on the order of 1-100 nm (Li G. et.al. 2012). Due to a wide variety of potential applications in biomedical, optical and electronic fields, research on nanoparticle is currently exceeding the others.

Nanoparticle has been an ideal choice because of its size range where it can fit into the small pore throat of shale. Larger particles in conventional drilling fluid cannot form mud cake because they cannot fit in the pore of the shale. Average pore throat size of common shale range from 10 to 30 nm while range for particle diameter for commonly used drilling fluid is 100 to 10 000 nm, which is far too large. When in contact with shale, it does no form mud cake and will not stop the fluid invasion. When drilling fluid filled with nanoparticles come in contact with shale, mud cake will form and bridging can take place. As being discussed earlier, bridging can help in reducing the rate of water invasion from wellbore into the formation thus keep borehole wall stable.

Other than its contribution in reducing shale instability, nanoparticle has also taking a role in controlling migrating fines particle. Problems regarding fines migration in reservoir have given a serious headache towards field engineers. Migration of fine particles mostly from sand can affect hydrocarbon production and can lead to uneconomical output. In order to control the fines, rendering formation fines immobile by attaching them to the formation matrix is the best option found (Ogolo N., et. al. 2012). An investigation has been conducted and it is revealed that nanoparticle has substantial effect towards making the fines bond tightly to the formation matrix.

A group of researchers from Nigeria published a paper aimed on investigating the fines trapping capacity of nine nanoparticles in sand packs. They examined the ability of nano treated sands to retain clays they contain at high flow rates of low salinity water capable of moving fines. From the experimental results, they found out that dispersing the correct type of nanoparticles in reservoir formations can control migrating fines caused by unconsolidated reservoir formations. In addition, instead of reducing permeability, nanoparticles, through several reported laboratory work and field applications, seems to improve the permeability of formation. This is a big reward to the researchers who have been looking for an ideal technique to control migrating fines problem.

In a paper written by Cai J. et al on “Decreasing Water Invasion into Atoka Shale Using Nonmodified Silica Nanoparticle”, concentration of nano-based drilling fluid and size of nanoparticle itself has become an important parameter in keeping the borehole stable. Their paper presents laboratory data showing the positive effect of adding nonmodified silica nanoparticles, size varying from 5 to 22 nm to water based drilling mud and their effect on water invasion into shale. Two types of common water based drilling mud; a bentonite and a low-solids mud were tested with and without the addition of 10 wt % nanoparticle.

The experimental results show that there is a huge permeability reduction when shale is in contact with water based mud when it is added with nanoparticle. For bentonite mud, the permeability decreased by 57.72 to 99.33%, and, for low-solids mud, the permeability decreased by 45.67 to 87.63%. They also found that nanoparticles of 10 wt % with size ranging from 7 to 15 nm are shown to be effective at reducing shale permeability, thereby reducing the interaction between shale and water based drilling fluid. As the average pore throat sizes of a variety of shale ranging from 10 to 30 nm, it seems like particles that falls into the size range between 7 to 15 nm is the most suitable in plugging the pore throat. The result from Pore Penetration Test shows that, 10 wt % nanoparticle concentrations gives superior outcomes than 5 wt % nanoparticle concentration.

CHAPTER 3: METHODOLOGY

The method to be consider in this project are divided into two; the preparation of nano-based drilling fluid and the other one is the preparation of shale sample.

3.1 RESEARCH METHODOLOGY

3.1.1 Preparation of Nano-Based Drilling Fluid

The nano-based drilling fluid is prepared by adding the nanoparticle (with different sizes and concentration) into the fresh water drilling mud. The experiment highlights the usage of water-based drilling fluid instead of oil-based fluid because of the economic and environmental issue given by oil-based fluid. The nanoparticle was added with different amount to produce different concentration into 250 ml gram of fresh water drilling mud. The concentrations needed are 2 wt %, 5 wt %, and 10 wt %. Nanoparticle with size average 30 nm and 15-20 nm are used for this experiment.

3.1.2 Preparation of Shale Sample

Technically, shale sourced from the oil well gives better result rather than the outcrop. But due to the time constraint and complex procedures of getting shale from the oil companies, the only alternative left is to source shale from outcrop. Shale from Batu Gajah was taken as the sample used for this experiment. The shale were then grounded into small pieces and dried in the oven to remove all their water. Dried shale sample were sent for mineralogy identification .These tests can provide important information on the reactivity of the shale. The shale samples used were ground to 4-8 Tyler Equivalent Mesh size as this represents the cuttings observed on drilling field. 10 grams of shale cuttings of 4-8 Tyler Equivalent Mesh sizes were used per experiment. There are six experiments involved where each experiment uses different concentration with different size of silica dioxide nanoparticle;

- Fluid 1: 2 wt % of 30 nm nano-based fluid
- Fluid 2: 5 wt % of 30 nm nano-based fluid
- Fluid 3: 10 wt % of 30 nm nano-based fluid
- Fluid 4: 2 wt % of 15-20 nm nano-based fluid
- Fluid 5: 5 wt % of 15-20 nm nano-based fluid
- Fluid 6: 10 wt % of 15-20 nm nano-based fluid

3.2 PROJECT ACTIVITIES

3.2.1Hot rolled dispersion test

Shale dispersion is where the shale cuttings disintegrate into size usually described as fines. Shale's dispersive abilities vary depending on the fluid in contact with it. The fluid will successfully inhibit dispersion by preventing the shale from breaking into smaller pieces. Hot rolled dispersion test were carried out to investigate the inhibitive properties of the prepared drilling fluids with the shale samples. A greater inhibitive property could be defined by calculating the percent recovery of the shale after the experiment. The higher the percent recovery, the higher the inhibitive property of the fluid, thus is more suitable to be the fluid which can prevent the shale disintegration.

A laboratory test was carried out to study the effect of nanoparticle in water-based drilling mud in preventing shale hydration and dispersion through hot rolling dispersion test. The inhibitive properties of the nano-based fluid were evaluated. It is believed that nanoparticle with high concentration and small size in water-based drilling mud has the highest inhibitive properties.

Dispersion test was carried out using a roller oven and 500 ml high temperature aging cells made from stainless steel. Shale sample will be rolled together with nano-based fluid at 66 deg celcius for 16 hours long. Hot rolled oven was used to simulate the actual downhole situation where rolling is one of the action involved. A schematic of rolling process is shown below;

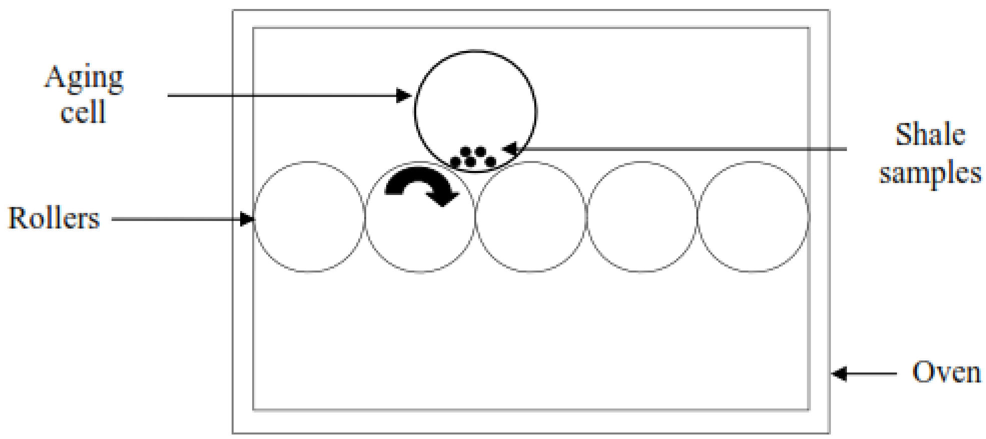
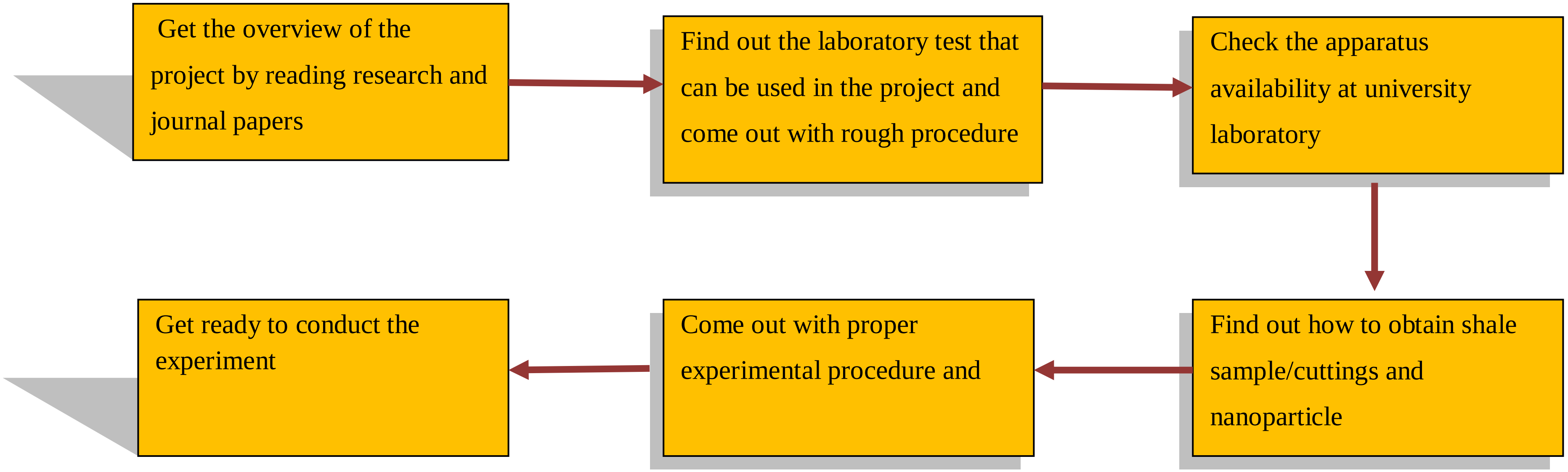


Figure 9: Shale in hot-roller oven

3.3 KEY MILESTONE



3.4 GANTT CHART

3.4.1 Project Gantt chart

Week / Detail	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Project work continues															
Submission of Progress Report															
Project work continues															
Pre-EDX															
Submission of draft report															
Submission of dissertation (soft bound)															
Submission of Technical Paper															

Oral Presentation															
Submission of Project Dissertation (Hard Bound)															

- Process

- Suggested Milestone

3.4.2 Procedure Gantt chart

Detail \ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Literature review	■	■	■	■											
Shale preparation					■										
Purchasing nanoparticle					■	■									
First experiment using 30nm nanoparticle							■								
2nd experiment using 15-20nm nanoparticle								■							
Result review								■	■						
Experiment continues (in case of error found in previous experiment)									■	■	■	■			

Oral presentation preparation															
Dissertation preparation															

3.5 Tools

Apparatus	Material
• Hot rolled oven • 500 ml stainless steel aging cell • 4-8 Tyler Equivalent mesh screen • Drying oven • Weight balance • XRD Chamber • Rheology tester	• Shale cuttings • 30 nm and 15-20 nm silica dioxide nanoparticle • Fresh water • Bentonite

CHAPTER 4: RESULT AND DISCUSSION

4.1 DATA GATHERING AND DATA ANALYSIS

4.1.1 Shale Reactivity

To know how the drilling fluid will react with the formation, knowing the mineral composition of a formation to be drilled is crucial. X-ray diffraction test was conducted to determine the mineralogical composition of shale or cuttings. A sample of shale was exposed to X-ray diffraction and was compare to the resultant diffraction pattern to know standards to determine which minerals are present in the sample. Shale reactivity is a function of types and amount of clay minerals present in the system. In this experiment, shale sample obtained from Batu Gajah was sent for X-ray diffraction (XRD) test to determine its mineralogy. From the peak reading of the XRD result, the sample is showing a portion of quartz, mica, and kaolinite.

4.1.2 Hot rolled dispersion test

The inhibitive properties of silica dioxide (SiO_2) nano-based drilling fluid of different concentrations were evaluated by carrying out hot rolled dispersion test with different sizes of SiO_2 nanoparticle. 10 grams of shale cutting was added into the aging cell together with nano-based fluid and was hot rolled at 66^{o} C for 16 hours. After 16 hours, the undispersed sample was recovered and weighed. Shale percent recovery was calculated by taking the weight of undispersed shale divided by the original weight of shale. The experiment continues with different concentration of nano-based fluid with different SiO_2 nanoparticle size.

- Experiment 1: 2 wt % of 30 nm nano-based fluid
- Experiment 2: 5 wt % of 30 nm nano-based fluid
- Experiment 3: 10 wt % of 30 nm nano-based fluid
- Experiment 4: 2 wt % of 15-20 nm nano-based fluid
- Experiment 5: 5 wt % of 15-20 nm nano-based fluid
- Experiment 6: 10 wt % of 15-20 nm nano-based fluid

Base fluid : Fresh water + 4% Bentonite		
Mud system	Size of nanoparticle (nm)	% Recovery
Bentonite slurry	Base fluid (without nanoparticle)	21.7
Bentonite slurry of 2 wt%	15	36.3
	30	35.2
Bentonite slurry of 5 wt%	15	55.5
	30	49.6
Bentonite slurry of 10 wt%	15	62
	30	66.7

4.1.3 Calculation

To calculate the percent of shale recovery, the equation used is as per below;

$$P\ (\%) = \frac{Wu}{Ws} \times 100$$

Where,

P = percent recovery (%)

Wu = weight of undispersed shale/ weight after being hot rolled (g)

Ws = original weight of shale sample (g)

4.2 EXPERIMENTATION PROCEDURE AND PROTOTYPE

1- Nanoparticle powder with different volume concentration was added into the fresh water drilling fluid. Fresh water and bentonite acts as a base fluid.

2- Nanoparticle was dispersed into the base fluid using ultra sonic agitation for about 30 minutes.

3- Place into the aging cell

4- Weight approximate 10 grams, Ws, of the clean sample sized 4-8 Tyler Equivalent Mesh size.

Figure 10 : Shale cuttings

5- Place into the aging cell

6- Roll in the oven for 16 hours at 66 ^{o} C.

7- Shale being cooled down to room temperature after being hot rolled.

8- Pour the fluid pass through 2.38 mm (8 Tyler Equivalent Mesh size) screen.

Figure 11 : Undispersed shale after hot rolled

9- Wash and weight the sample, Wu

Figure 12: Dried shale after hot rolled

4.3 DISCUSSION / FINDINGS

Below is the shale percent recovery obtained after the experiment.

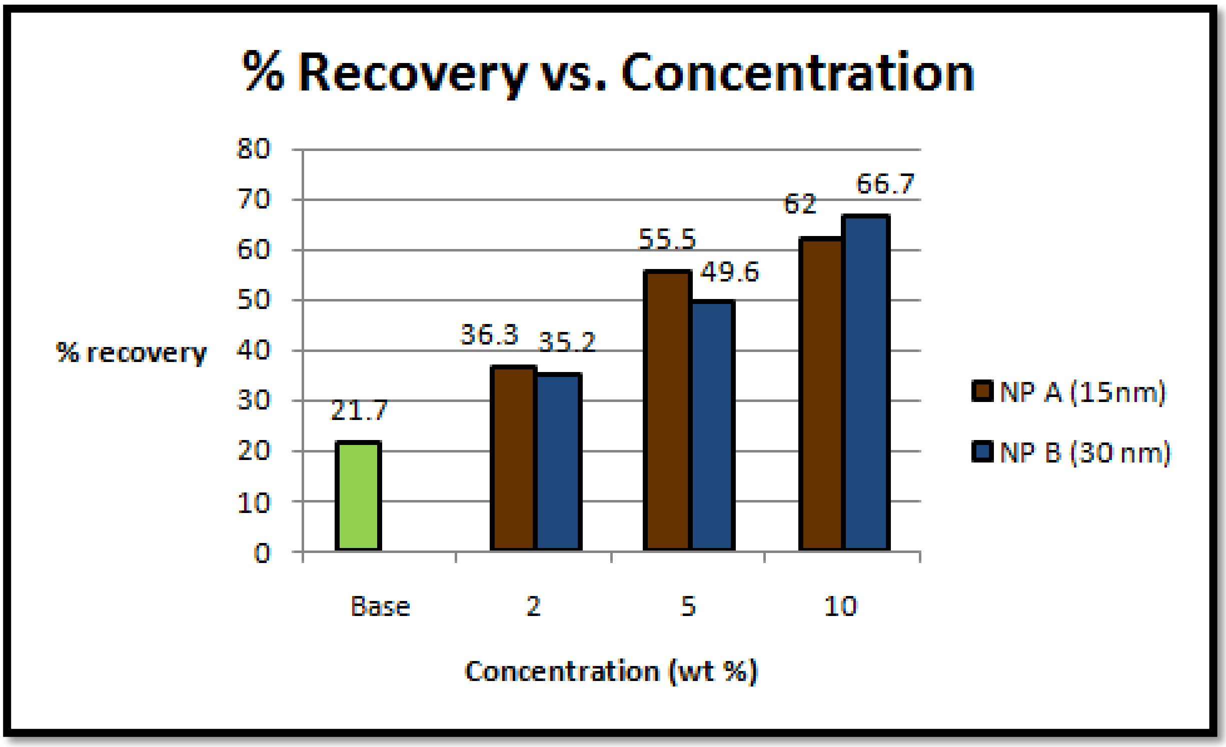


Figure 13: Shale percent recovery vs. Concentration

From the experiment result, it can be seen that the percent recovery is higher in concentrated nano-based fluid. The percent recovery in 2 wt % is 35-36 % while in 10 wt % is in the range of 62-66 %. This shows concentrated nano-based fluid has greater inhibitive performance than less concentrated fluid. The reason behind this is related to the water activity of the fluid. The presence of high concentration of nanoparticle in water-based mud form lower water activity in mud system. Once the mud activity is lower than shale, the tendency of water to be absorbed by shale is reduced. However, different size of nanoparticle gives insignificant result for the percent recovery. At 2 wt% and 5 wt%, the smaller size nanoparticle gives better reading than bigger size nanoparticle, which correctly justifies the hypothesis. But in 10 wt %, the bigger size nanoparticle gives better recovery than the smaller one. This experiment failed to show the superior result of 15 nm nanoparticles plugging the shale pore throat than the 30 nm nanoparticles, as being stated in the hypothesis.

The author believed that the failure is mainly due to the wrong choice of experiment. The dispersion test can only measure how the concentration of the fluid affects the shale hydration. To measure the plugging properties, which is related to the size of nanoparticle needs other laboratory experiment, the swelling test which cannot be done here in UTP due to time constraint, lack of materials and apparatus. Besides that, to conduct the experiment, the author may need a shale sample abundant in montmorillonite because this mineral has good swelling property. However, this kind of shale is very difficult to be obtained. Thus, it is quite impossible to carry out the swelling test.

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

5.1 RELEVANCY TO THE OBJECTIVE

Wellbore instability has been a nightmare to almost all drilling engineers around the world. To avoid uneconomical production and loss of productivity, wellbore instability problem has to be avoided as sooner as it can. It has been verified by researchers that wellbore instability mostly occurs at shale region. Shale contains high clay particles which one of the particles called smectite is very absorbent to water. As water-based drilling fluid being flowed downhole, water invasion from wellbore into shale formation will take place. This may result in swelling effect in the shale. Swelling effect can be explained by two major incidents; one is by the osmotic inflow and the other one is by the mud penetration. If there is less salt concentration (or high activity) in drilling fluid, osmotic inflow will take place. Osmotic inflow is the flow from the wellbore into the formation. Osmotic inflow will then increase the pore pressure and then lead to absorption of water into the shale formation. The latter case explains the loss of initial wellbore strength by mud column. When well is drilled, the mud column which supports the wellbore will lose its integrity. In the meantime, mud can penetrate into the formation and cause the formation to become instable. If the clay platelets in shale formation are free to move, swelling effect will take place but if they are restricted, hydrational effect will occur.

One method to avoid water invasion into the shale is by plugging its small pore throat. Particles in conventional drilling fluid are too big and cannot form bridging effect. Bridging is important because with the presence of mudcake, water can be blocked from entering the shale pore throat. Thus, water invasion can be reduced as well as the swelling effect in shale. Other than aiding in forming the bridging effect, the particles have to plug the pore throats leaving no empty space for water inside the shale. To achieve this, many have suggested using nanoparticle as the additives added into conventional water-based drilling fluid. Nanoparticle is the most suitable particle to be used as plugging material because of its smallest particle size range. Nanoparticle will be added into conventional water-based drilling fluid forming nano-based fluid. This fluid will be tested on shale to check for the stability enhancement. In this project, the size and concentration of nanoparticles are varied to see their effect in giving the best engineered drilling fluid to increase the shale stability.

In this project, the author has modified the procedure to determine the shale stability. Rather than to undergo the swelling test for the shale, the author has taken the initiative to check and analyze on the inhibitive properties of the drilling fluid. This experiment has taken the principle of the osmotic flow mentioned above. The inhibitive properties of the fluid is said to be significant if the concentration of nano-based fluid is higher. The presence of high concentration of nanoparticle in water-based mud can be considered as one of the best solute which is able to demonstrate the desirable characteristics to form lower activity (or high concentration) in mud system. Once the mud activity is lower than shale, the tendency of water to be absorbed by shale could be reduce thus preventing shale dispersion.

The inhibitive property of the fluid is measured by the shale percent recovery. The higher the recovery, the less dispersed the shale is. Thus, the fluid in which the recovery was higher is the best fluid (with highest inhibitive property) to prevent shale dispersion. Based on the graph, the recovery is increasing with the increase of concentration. At 10 wt%, the recovery was the highest. However, for different size of nanoparticle, the result is quite irrelevant. The recovery was supposed to be higher in fluid mixed with smaller nanoparticle but at 10 wt%, the recovery for bigger nanoparticle is greater than the recovery for smaller nanoparticle. The author believed that the error might be due to the wrong choice of experiment. To check for the effectiveness of nanoparticle size in preventing shale dispersion, one must use other experiment i.e. swelling test and pressure penetration test.

5.2 SUGGESTED FUTURE WORK FOR EXPANSION AND CONTINUATION

5.2.1 Scanning Electron Microscope (SEM)

Rather than using XRD test alone, shale can be analysed in more accurate manner through SEM. SEM is a tool for performing high-magnification analyses of shale. By using SEM technology, three dimensional observations of micro-fractures and cavities in the shale can be obtained. On top of that, SEM also offers the result for the texture and orientation of the shale, its degree of compaction, and the presence of imbedded minerals and pores. SEM also can demonstrate any invasion of drilling fluid into shale microscopic pores.

SEM needs either cavings, cores or any other large cutting to be conducted. The sample needs to be large enough to be cut or broken to expose a fresh surface for analyses. The analyses can be quite time consuming, depending on the expertise and what is being observed. Due to its expensive instruments, most laboratories do not have the SEM apparatus. SEM procedure also needs a real trained analyst to conduct the procedure.

5.2.2 Swelling Test

Due to the unavailability of the linear swelling tester in the laboratory, this project cannot be continued using this test. By using the linear swelling tester, free swelling of a reconstituted shale pellet after the shale has been in contact with a drilling fluid can be measured easily. The tester consists of a fluid reservoir, a shale chamber, a linear variable differential transformer (LVDT), A/D converter, and computer. Though the procedure is a bit complicated, but this is the best method to test the shale reactivity onto the fluid sample being tested. The more swelling observed with water, the higher water-sensitive the clay is. This test can be performed on cuttings, cavings, sidewall core, and full-diameter core. Formation samples with massive structures and homogeneous composition are most suitable for this test.

5.2.3 Bulk Hardness Test

This test is designed to test the hardness of the shale sample after it is being exposed to the fluids. After some interactions with fluids, shale will become softer due to the absorption of water, swelling,and dispersion of fine particles. The method of this test also using the hot roller oven. After hot rolling, the shale pieces are recovered on a 50-mesh sieve and place into the bulk hardness tester. The shale hardness will be represented by its torque reading. Harder and more competent shale pieces will give higher torque readings. Cuttings are the most suitable sample to be used in this test.

5.2.4 Shale Membrane Test (SMT) / Pressure Penetration Test

This test might be the best test to study the effect of physical plugging on shales with the nanoparticle. The SMT apparatus is illustrated as per below. A test fluid is pumped to flow across the top surface of a shale sample at a constant pressure (P2,P3), while measuring the pressure buildup in the bottom reservoir with a tiny constant volume. Shale sample permeability for the test fluid can be interpreted from the bottom pressure buildup (P1). Permeability changes of the same shale sample, with respect to various test fluids (brine and the WBM), are treated as an indicator of physical plugging (also shale stability) by solids and nanoparticles in water-based mud. The more significant the permeability reduction between the WBM and initial brine, the better the physical plugging by the solids and nanoparticles.

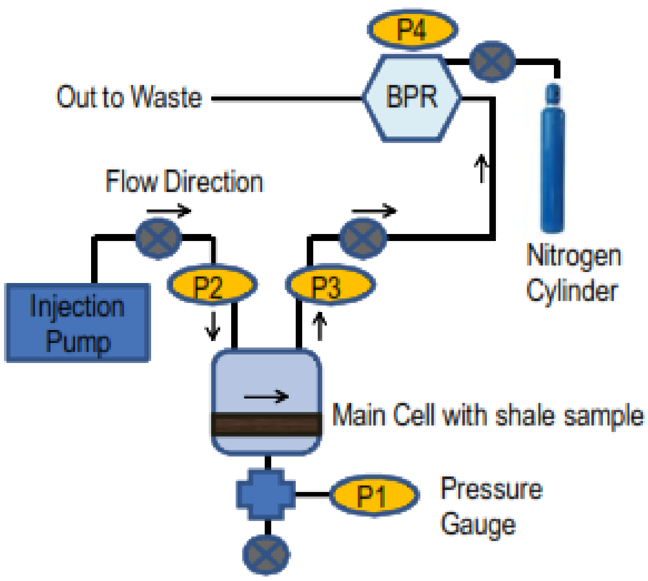


Figure 14: The SMT apparatus

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