

CERTIFICATION OF APPROVAL

**Investigation on Bentonite and Its Compatibility with Cationic Surfactant for  
an Effective Permeability Modifier**

by

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16680

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May 2015

## CERTIFICATION OF ORIGINALITY

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

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## ABSTRACT

Natural swelling ability and low cost have made bentonite preferable material for improved oil recovery such forming viscous water suspension. In Improved oil recovery (IOR) projects, poor volume sweep efficiency in heterogeneous reservoir limits its success. One of the IOR methods which of recent interest is permeability modifier method. Conventional blocking agent such as bright water and micro gel based on polymer cannot be applied at high temperature. Therefore, bentonite is proposed as a blocking agent because it can withstand high temperature conditions. However, bentonite has tendency to coagulating before it could penetrate the wellbore when in contact with water. Thus, cationic surfactant such as n-dodecyltrimethylammonium bromide (DTAB) is proposed as an additive in bentonite solution to overcome this problem. Laboratory experiments has been conducted in this study to determine the most stable modified bentonite solution before it is analysed in core flooding test. This study found that, the most stable bentonite solution is 20 $\mu$ m nanoclay with 13.7 $\times 10^{-3}$ mmol/kg of DTAB. It is able to reduce the liquid permeability of the core in about 64% from original permeability. Indeed, modified bentonite solution has the potential to be used as a blocking agent in a permeability modifier method.

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## ABBREVIATION

BPS	Benchtop Permeability System
CDG	Colloidal Dispersion Gels
CMC	Critical Micelle Concentration
DTAB	N-Dodecyltrimethylammonium Bromide
EOR	Enhanced Oil Recovery
HCl	Hydrochloric Acid
NaCl	Sodium Chloride
NaOH	Sodium Hydroxide
NTU	Sodium Chloride
PV	Plastic Viscosity
SDS	Sodium Dodecyl Sulfate
YP	Yield Point

# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Bentonite minerals could offer a huge business potential in this era as it is proven to give a wide range of application and benefits to many industries. Bentonite can be defined as absorbent impure clay which forms in a microscopic crystal altered from glassy igneous material such as volcanic ash or tuff (Onal, 2006). Industrial bentonite predominantly contain  $\text{Ca}^{2+}$  and  $\text{Na}^+$  to form Ca-bentonite and Na-bentonite due to the counterbalanced of exchangeable cations in the interlayer of bentonite particles (Yunfei *et al.*, 2006, Onal, 2006). Moreover, the swelling property of bentonite to form a viscous water suspension makes it reliable for many industries. Some of the applications are paint, paper, plastics, decolorization, chemical carriers, liquid barriers, foundry bondants, drilling fluids, desiccants, sealants, cosmetics, adhesive and catalyst (Onal, 2006). Bentonite has been applied in industries since early 1848 by Knight found near Fort Benton, Wyoming which it occurring a highly plastic clay mineral (Grim, 1953, p.361).

There are many applications of bentonite in the oil and gas industry such as in drilling process and well plugging. In the process of drilling, bentonite is the main component in the drilling fluid or so called water base mud (Al-Homadhi, 2007). Whereas, according to Towler (2008) sodium bentonite is used in well plugging activity because it has the swelling property, useful to act as a sealant to form an impermeable layer when it is in contact with water.

Permeability modifier is one of the approaches in enhanced oil recovery where its objective is to plug the main path or high permeability region in a reservoir. This leads to a situation where low permeability region appears more favourable to water flood and allows the remaining oil flow into the production well from these zones. Moreover, early water breakthrough is discovered during water flooding activity due

to the water more favourable and easy to flow in a high permeability region thus causing in a huge impact in production planning. Many different materials have been proposed as a blocking agent and as concluded by Seright *et al.*, (1995) the materials are gels, particulates, precipitate, microorganisms, foams and emulsions.

In this study, bentonite is proposed as a permeability reducing agent and will evaluate its performance and effectiveness by laboratory experiments approaches. However, bentonite alone is not compatible to be injected in the reservoir as it could solidify before it could reach the ‘thief’ zones. According to Lu (1988), cationic surfactant has a better performance in clay swelling inhibition compared to other available additives. Thus, in this study cationic surfactant will be used as swelling inhibitor for bentonite and its compatibility to obtain an effective permeability modifier in the formation will be evaluated. Lastly, the performance of modified-bentonite materials will be compared with other conventional blocking agent for permeability modification.

## **1.2 Problem Statement**

Most of the unrecovered oil in the heterogeneous reservoir was trapped in the pore because of its low permeability due to the geological layering, fractures and rocks inter-layer. These factors result in poor volume sweep efficiency of oil which limit the success of chemical EOR field projects. Eventually, the early breakthrough of water will be found in the production well once the oil from favourably reached pores has been recovered. Extra studies and precautions need to be taken when exploring heterogeneous reservoir to prevent uneconomical decision. It is important to have favourable volumetric sweep efficiency especially at low permeable region in order to obtain optimum results of chemical enhanced oil recovery.

Many studies have been done in improving the conformance control in the heterogeneous reservoir and one of the inventions is permeability modifier approach. There are many suggestions on selecting the type of chemical used as blocking agents and one of the chemical that received intention is preformed polymer gels or conventionally known as colloidal dispersion gels (CDG). However, CDG could not be implemented at high temperature reservoir over 100 degree Celcius (Caili *et. al*, 2012). CDG will result in thermal degradation and low-cross-linking intensity when exposed at high temperature surrounding (Xiaofen, 2004 and Caili, 2010).

Alternatively, bentonite clays is proposed as a permeability modifier because it could withstand at high temperature condition and high stability.

Generally, smectites is the dominant clay mineral in bentonites which consist of montmorillonite, beidellite, saponite, nontronite and hectorite (Grim, 1968). Montmorillonite and mixed montmorillonite-illite are strong sensitive clay minerals and it swells when in contact with water (Lu, 1988). Ideally, bentonite is injected and penetrated into the reservoir and flow to the high permeable zone to block that particular area in order for water or gas flooding to flow through the low permeable region and swept the oil. The purpose in this study is to determine the effectiveness of the permeability modifier by using bentonite. However, by contacting only water with bentonite, it has a tendency to accumulate and solidify in the wellbore before it penetrates through the reservoir. Thus, cationic surfactant will be used in this study to determine its compatibility with bentonite and act as a blocking agent in the reservoir.

### **1.3 Objectives**

In this project, bentonite has been selected as a material for the permeability modifier which will be modified by using cationic surfactant for possible improvement in the sweep efficiency. The main objectives are:

- To identify the optimum stability of bentonite and cationic surfactant for permeability modifier application.
- To compare the performance of blocking agent against bentonite with anionic surfactant and grafted polymer.
- To evaluate the performance of the blocking agent

### **1.4 Scope of Study**

The Scope of this study is mainly to determine the optimum stability of colloidal dispersion of modified bentonite solution. There are several parameters concern such as the size of bentonite particles, the pH of the solution and the concentration of the cationic surfactant. This project requires laboratory experiment and analytical evaluation based on the results obtained. Extensive literature review is required in order to strengthen the analysis of the results. The effectiveness of the stabilize solution is determined by conducting core flooding test. Finally, the author will come out with a summary of the results as well as recommendations.

## **1.5 Relevancy and Feasibility of Project**

This study is relevant to student as well as the oil and gas industry especially in EOR method because conformance problem in heterogeneous reservoir reduce the effectiveness of EOR application due to the observation of an early water breakthrough. There are extensive studies have been made in order to improve the conformance of heterogeneous reservoir and many of the studies used chemical based material as a blocking agent. However, there are many restrictions when applying chemicals into the reservoir and this can be encountered by substituting chemical based material to bentonite. Applying bentonite as a blocking agent is feasible to the EOR projects where it is require less cost and restriction. On the other hand, this project is feasible to carry out by considering the capability of a final year student and time constraint with the assistance of supervisor. The analytical evaluation can be done by the accessibility to journals, books and other sources. The project can be conducted and completed within the timeframe.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Reservoir Conformance**

Conformance is defined as a measure of the volumetric sweep efficiency during an oil-recovery flood or process being conducted in an oil reservoir (Robert *et al.*, 2011). Reservoir conformance is taking into consideration when external fluid drive is injected into the reservoir to increase the sweep efficiency of oil to the production well. A perfect conformance drive occur when injected fluid is able to sweep the remaining oil uniformly across the reservoir meanwhile the imperfect conformance left the remaining oil un-swept and causing the fluid in early breakthrough.

Homogeneous reservoir is mostly and favourably open for exploration as it has same rock properties across the reservoir thus it is easier for oil production planning until its abandonment. However, in heterogeneous reservoir the rock properties are varies across the reservoir thus it is difficult to estimate the oil recovery performance. Non-uniformities of vertical and horizontal rock in heterogeneous reservoir makes the conformance inefficient as injected fluid will flow to the favourable region which is high permeability region and left the remaining oil at low permeability areas.

Enhanced oil recovery (EOR) projects are applied when water injection is no longer sufficient for sweep the oil to the production well. Chemical EOR is applied to the reservoir in order to increase the sweep efficiency of remaining oil. However, chemical EOR oil projects have a limits impact when there are reservoir conformance problems. This problem could delay the oil production pattern due to the slow fluid flow in the reservoir. Poor sweep efficiency can be determine by the excessive water production which leading to early well abandonment with unrecoverable hydrocarbons.

Injection profiles helps showing the fluid flow determination across the reservoir through the wellbore.

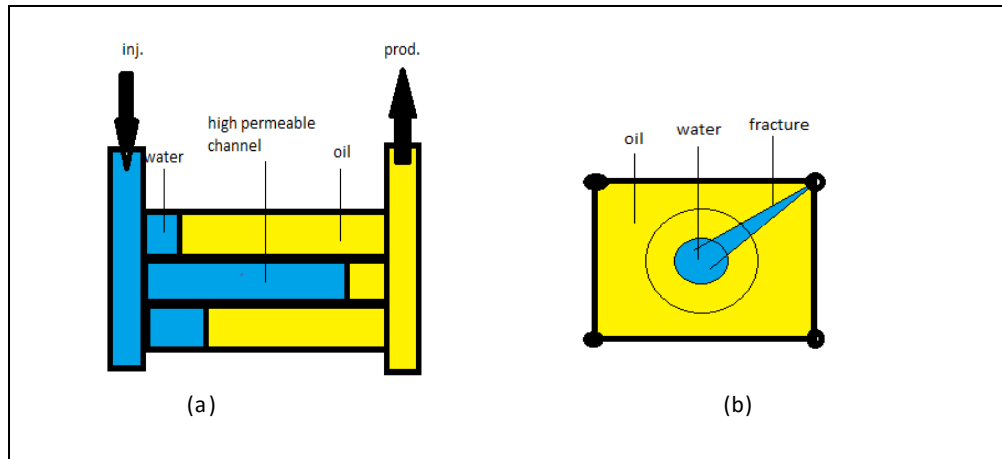


Figure 2.1. (a) Vertical conformance problem and (b) Areal conformance problem due to fracture (Sydansk *et al.*, 2011)

Usually, the vertical and horizontal non-uniformities, due to geological factor make the oil left unrecoverable by the early water breakthrough in production well. Clear understanding of conformance problem can be visualized from the Figure 2.1. Figure 2.1(a) shows high permeability channel only available at the midway path within the matrix rock reservoir. Water is shown to flow more in the high permeability channel compare to the low permeable channel and cause in early breakthrough of drive-fluid. In Figure 2.1(b), areal conformance problem is shown by the existence of a fracture across the reservoir that extends from injection well through one of the production well. This cause the water is easily flow to that particular well and cause early breakthrough compare to other wells.

## 2.2 Conformance Improvement Methods

The major reason of petroleum-reservoir conformance problem is because of the variation of fluid- flow capacity in the heterogeneous formation (Robert *et al.*, 2011). There are many other typical conformance problem which are high-permeability channel with and without cross-flow, directional trends, fingering due to difference in viscosity, water coning and many more. This problems will results in early water breakthrough of injected fluids as well as low oil production. Channelling or fingering of fluids brought unsuccessful EOR field test in the 1970s and 1980s

because most of the injected EOR fluids will just flow through the high permeable region without contacting overall reservoir's area (Brattekas, 2014).

There are two conformance-improvement techniques that can be applied within the oil-reservoir rock itself ((Robert *et al.*, 2011). The first technique is to increase the viscosity of displacing fluid whereas the second technique is using blocking agent material in high permeable channels. According to Sydansk and Romero-Zeron (2011), a successful technique of conformance-improvement results in several contribution such as:

- Sweep efficiency is reduced
- Oil recovery is increased
- Oil recovery rates is accelerated
- Reduced in recycling of drive fluids which lead to reducing in EOR expenses.
- Environmental liabilities is reduced as well as environmental benefits is increased.

Reducing permeability or totally plugging high-permeability flow channels and/or anomalies is the second most widely applied method to promote conformance improvement within the oil reservoir itself (Robert *et al.*, 2011). The objective of this method is to enable the fluid-drive to flow favourably in low permeability channel thus sweep the hydrocarbon in those channels. Moreover, this method will slow down the fluid-drive production when the high permeable channels is completely plugging. Thus, the conformance improvement is held within the reservoir itself instead of applied at the wellbore area. Ideally, the blocking agent should reduce the permeability at high channelling area and wash out all the remaining water in the pore without damaging the oil productivity (Seright *et al.*, 1995).

Conformance improvement such as permeability-reducing method gives positive attraction as it results in efficiently increases oil sweep efficiency when there is a vertical and areal conformance problems in the heterogeneous reservoir. The material used as the blocking agent must be studied thoroughly in order to prevent the change in oil properties in the reservoir as well as the rock itself. Many chemicals materials have been proposed to reduce the channelling in reservoir which cause an early water breakthrough (Seright *et al.*, 1995). Some of the materials that widely been



tested for future industrial application are gels, particulates, precipitates, microorganism, foams and emulsions. However, these materials have its own advantages and drawbacks that have to consider before applied it in the industry.

Gel is the most popular material that is used in the industry as a blocking agent in high permeability channels or in fractures (Brattekas, 2014). The gel enable increased of differential pressure gradients across matrix block during fluid flooding process. Many types of gel system has been tested for the purpose of oilfield conformance such as bulk gels and microgels. Moreover, according to Sydask and Romeo-Zeron (2011) polymer gels is the most widely applied conformance-improvement technology in petroleum industry. Polymer gels could flow at high flowrate, yet rigid and may be placed deep in the reservoir. However, polymer gels could also penetrate to a significant degree into all open zones which means it may flow through the low permeable zone and can be seriously damaged the zones (Seright *et. al*, 1995). An effective gel placement is easier to achieve in fractured wells because of the linear flow of the fractures and large permeability contrast between pore of formations and fracture (Seright *et al.*, 1995)

### **2.3 Bentonite Applications**

Bentonite is clay type material which exists as discrete deposits and created by the volcanic eruptions when volcanic ash settled in shallow seas. Bentonite have been used widely in the industry for numerous application such as palletizing iron ores, foundry bond clay, ceramic, drilling mud, sealent, animal feed bond, bleaching clay, agricultural carrier, cat box adsorbent, adhesive, catalyst support, desiccant, emulsion stabilizer, cosmetic, paint, pharmaceutical, civil engineering, pillared clay organoclay and polymer-clay nanocomposites (Onal, 2006). There are several types of bentonite available and widely used which is Na-bentonite, K-bentonite and Ca-bentonite. These types of bentonite have different characteristics and properties which differentiate it applications in the industries.

It is important to understand the nature of bentonite. The dominant clay minerals in bentonite are smectites which consist of montmorillonite, beidellite, saponite, nontronite, and hectronite (Grim, 1968). Bentonite consist of clay and non-clay minerals as well as some organic impurities. A small portion of non-clay minerals are usually silica polymorphis, zeolites, feldspar, carbonates, sulphites, sulphides,

sulfates, oxides and hydroxides (Moore *et al.*, 1997). Montmorillonite is the smectites which cause the bentonite to swell when in contact with water. Montmorillonite can be described as a hydrous aluminium silicate containing small amounts of alkali and alkaline-earth metals (Krumrin *et al.*, 2014).

In addition, it is important to understand the unique chemical structure of the bentonite in order to determine the chemical bonded when it is in contact with other materials for instance in this study is cationic surfactant. Montmorillonite is made of two basic building blocks which are aluminium octahedral sheet and silica tetrahedral sheet. Every two silica tetrahedral (T) sheets bonded with one central alumina octahedral (O) sheet as seen in Figure 2.2. The montmorillonite lattice is negatively charge and bonded to form 2:1 layers due to the isomorphous substitution in the octahedral sheets of  $\text{Fe}^{2+}$  and  $\text{Mg}^{2+}$  for  $\text{Al}^{3+}$  and in tetrahedral sheets of  $\text{Al}^{3+}$  for  $\text{Si}^{4+}$  (Onal, 2006). This negative charge is balanced by cations such as  $\text{Na}^+$  and  $\text{Ca}^{2+}$  which form Na-bentonite and Ca-bentonite located between layers and around the edges (Onal, 2006).

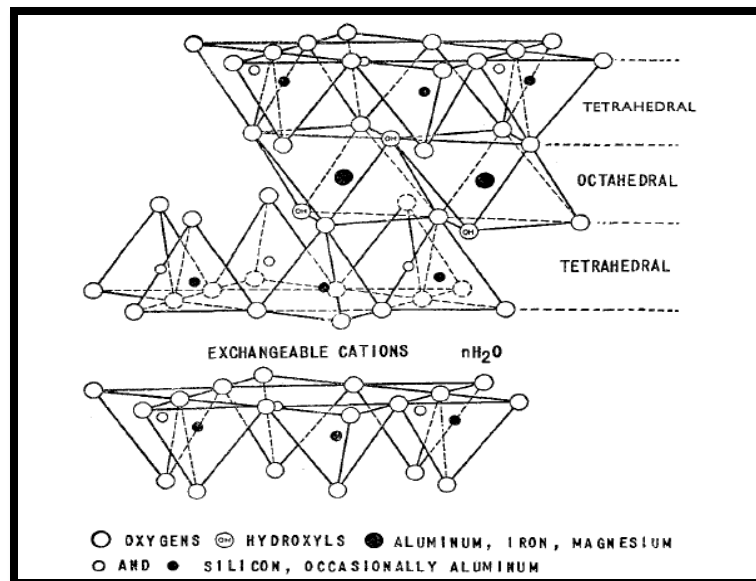


Figure 2.2. Diagrammatic sketch of montmorillonite structure (Grim, 1953)

In this study, bentonite is approached as a blocking agent in the high permeable channels which same application as in-situ gel system and preformed cross-linked polymer gels. Bentonite is proposed as the blocking agent because it is much cheaper compared to existing chemical blocking agent as well as its reliability. Bentonite is widely known as swelling clays has the possibility to block the pore in the reservoir

and proved to have the ability in increasing viscosity, inhibit corrosion, bacteria-killing, emulsion breaking and many more (Lu, 1988). Bentonite clay was first found in Wyoming and describe as highly colloidal and plastic clay. The unique characteristic found in the bentonite clay which it can increase it volume for several times when small amount of clay is mixed with water and form viscous water suspension or called as thixotropic gels.

Swelling characteristic in forming thixotropic gels is occurred due to the existence of montmorillonite mineral in the bentonite. In addition, the non-clay mineral content of bentonite is rarely less than 10% (Grim, 1953). The other clay minerals that often content in the bentonite are illite and kaolite. Moreover, it large surface characteristic resulting it in good stabilizing emulsions and allow it as a medium to carry other chemicals. The particle size and grades of the clay varies in different disciplines. In geology term, the size of the particle is about 4 micron ( $4 \times 10^{-6}$ m) while in soil the upper size of the particle is 2 micron.

As refer to bentonite as a permeability modifier, bentonite with water alone could not achieve the objective of this study because it may change it properties when it reach into the deep wellbore area and tend to coagulate with each other along the wall of the wellbore. Bentonite may increase it volume in the wellbore or in the formation and block the wellbore or near wellbore area before it could solidify in the required high permeability zones. This is happen due to the additional of hydrogen ion exist in the wellbore or formation which will attracted by the free negative charge of clay minerals and increase the volume of bentonite and thus block the area. Figure 2.3 shows a simple chemical structure of swelling process of bentonite.

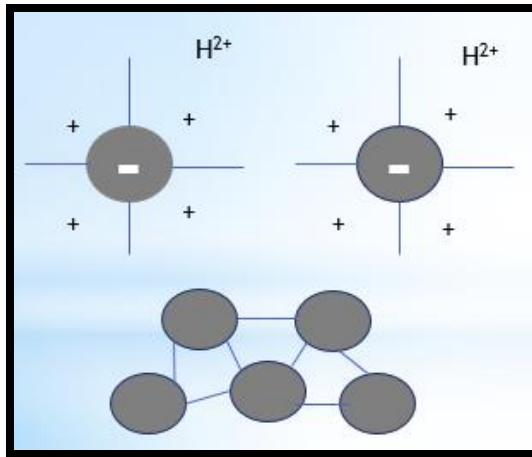


Figure 2.3. Swelling process of Bentonite

## 2.4 Cationic Surfactant

According to Lu (1988), cationic surfactant gives better results in clay swelling inhibiting compared to other additives. Previously, potassium chloride is used for clay stabilizer however it was not an ideal agent because it could decrease the viscosity of fracturing fluid with low salt resistant property. Other than that, due to its high acidic content it can corrode the equipment and have a short effective life. Thus, cationic surfactant is proposed to replace this additives as it could counter the drawbacks of potassium chloride application.

Cationic surfactant is a form of soaps or detergent with a positively-charged ions. At room temperature, cationic surfactant is an orange coloured fluid with pH of 5, specific gravity of 0.97 and its viscosity is 10 mpa/s at 13°C (Lu, 1988). Cations in the molecules will attracted to the negative charges of clay results in reducing the repulsion between the sheet structures. Adsorption process between cationic surfactant and clay will form a hydrophobic film on clay surface and preventing further water penetration and decrease the clay hydratibility (Lu, 1988).

In addition, it is found that cationic surfactant has a result of increasing of viscosity in water-base fracturing fluid (Lu,1988). Only small amount of cationic surfactant is required in order to increase the viscosity of the fluid thus improve the shear resistant performance.

Cationic surfactant application provide many advantages such as bacteria-killing, reducing of formation fluid interfacial tension, breaking of emulsion, good

compatibility with formation fluid, no formation damage and many more (Lu, 1988). It is also required only small amount of cationic surfactant for each application which it is conveniently used for the industry as the cost will be reducing compared to potassium chloride. As refer to Lu (1988), a written report of cationic surfactant for clay swell inhibitor was issued at the end of 1982 and since then it is used in petroleum industry providing with a good results. Since then the application of cationic surfactant is widened to pad fluid and washing fluid application as well as acidizing of gas wells.

In this study, cationic surfactant is proposed to prevent bentonite from coagulate with each other and allow the bentonite to flow through the high permeability channel and plug the pores. Specific characteristic and properties of bentonite and cationic surfactant will be further identified in order to obtain the best results in permeability modifier activity. The basic understanding of permeability modifier application in heterogeneous reservoir is shown in the Figure 2.4 below. Figure 2.4(a) shows reservoir with vertical conformance problem when there is barrier between the regions. Then, bentonite is injected into the high permeable channels and plugs the pores along the region. Figure 2.4(b) shows fluid flow in less favourable channels and sweeps the remaining hydrocarbons to the production well.

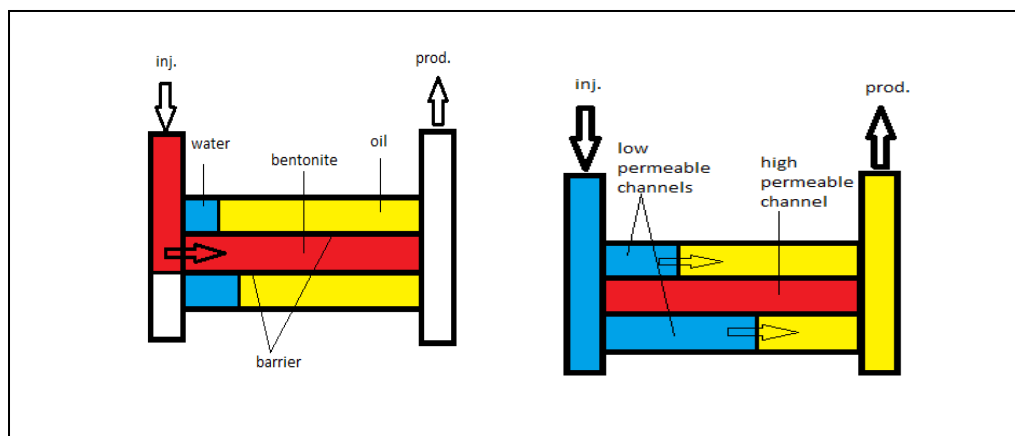


Figure 2.4. (a) Bentonite is flow into the high permeable channel and  
 (b) Fluid is flow into low permeable channel (Sydansk *et al.*, 2011)

## 2.5 Steric Stabilization

Colloidal system or the determination of the stability of the solution are dispersed phases finely subdivided in a dispersion medium. Mostly, the colloidal system occurs in a continuous liquid phase. The size of the particles may results in

different colloidal characteristics. For instance, the particles diameter between 1 to 1000nm may exhibits colloidal character due to the factor of the surface area of the particles that available for the interaction to occur. Lourenco (1996) stated that in order to reach stability in colloidal system it is traditionally influence either from electrostatic or steric effects. Stability is usually impaired when there are more than one mechanisms in the solution.

The stability of the solution is determined by the behaviour of the particles when it interacts with each other. The basic phenomena occur during particles interaction is either attraction or repulsion. The attraction force is dominates when the particles will adhere with each other and coagulation occur due to the bridging of the particles. Meanwhile, opposite phenomena occurred when repulsion dominates the system where it is more stable and remain in dispersed state. This phenomena is called Van Der Waals force where the interaction is due to the covalent bonds of the particles, or the electrostatic interaction between ions either in neutral or charged molecules.

The attractive force between molecules may form long bridging range of similar colloidal particles and in order to stabilize the system it is necessary to provide a long bridging of repulsive force as well. The repulsion gained should be at least as strong as the attractive force in order to break the attractive bridging molecules. There are three methods in obtaining stabilize colloidal particles:

- Electrical double layer which involve electrostatic or charge stabilization.
- Steric stabilization by adsorption or chemically attached polymeric molecules.
- Depletion stabilization by using free polymer in the dispersion medium.

In this study, the colloidal system involving bentonite particles and cationic surfactant particles can be related with steric stabilization method. Even though the particles interaction in this method involved polymeric molecules, it is also can be relate with this study as the mechanism of particles interaction is similar with steric stabilization method.

In order to achieve steric stabilization, the colloidal particles should attached with each other either by grafting or chemisorption of macromolecules to the particles surfaces. In addition, steric stabilization is a mechanism which enables certain additives attract to the particles and inhibit the coagulation. The additives should be

water-loving where it is naturally adsorb to the suspension particles. Cationic surfactant is one of the example in water-loving particles which the hydrophilic contains a positively-charged ions. This positive-charged ions is adsorbed to the clay surface and long-chain alkyl group forces a hydrophobic film on clay surface to prevent further water penetration. Thus, the clay particles will reduce it hydratibility which results in inhibiting the clay from swelling (Lu, 1988). Figure 2.5 shows the schematic figure of steric stabilization.

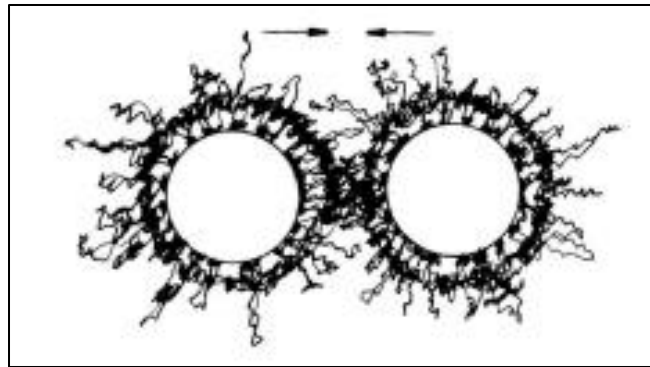


Figure 2.5. Schematic of Steric Stabilization (Jingyu, 2002)

The swelling ability, low cost and reliable are the main reasons bentonite is proposed as a blocking agent for permeability modifier. It could encounter the disadvantages of existing chemicals used for blocking agent. However, bentonite suspension could coagulate and solidify in the well before it penetrates into the reservoir. Therefore, cationic surfactant is proposed as an additive for the bentonite suspension where it could stabilize bentonite suspension and prevent coagulation from occur. Cationic surfactant is proposed in this study because it is proved by Lu (1988) that cationic surfactant gives better results in clay swelling inhibiting compared to other additives such as potassium chloride and anioinic surfactant. In this study, the stability of the modified-bentonite suspension is determined by identifying the optimum amount of bentonite as well as cationic surfactant need to be used in order to achieve stability. Steric stabilization concept should be achieve in modified-bentonite suspension where bentonite particles enables certain additives attract to the particles and inhibit coagulation.

## **CHAPTER 3**

### **METHODOLOGY**

There are four main steps of experiments to obtain the required results of this project. The steps are explained below.

#### **3.1 Materials**

Nanoclay from Sigma-Aldrich is compared with drilling mud obtained from Australian Mud Company is used in this study as a main material for blocking agent. N-Dodecyltrimethylammonium bromide (DTAB) supplied by Merck KGaA, a Germany company is added into the bentonite solution to determine its effectiveness of preventing bentonite from coagulating before it penetrates into the core. Chemicals such as sodium chloride (NaCl), hydrochloric acid (HCl) as well as sodium hydroxide (NaOH) obtained from R&M chemicals is solubilized in a low molarity or concentration which is used for the preparation for the samples.

#### **3.2 Sample Preparation**

##### **3.2.1 Particle Size Distribution and Preparation of Sample**

There are two types of bentonite prepared in this study which are drilling mud and nanoclay. Every samples are sieve accordingly in order to obtain different sizes of bentonite. In drilling mud, size of 32 $\mu$ m and 50 $\mu$ m are able to sieve whereas size for nanoclay are 20 $\mu$ m and 32 $\mu$ m. This activity is held at block 4 (chemical engineering block) with ambient temperature (approximately 25°C). After that, four brine solutions is prepared by adding 1g of sodium chloride (NaCl) into a 100mL of distilled water. Then, 0.1g of every sample is added into the brine solution.





Figure 3.1. Bentonite solution

### 3.2.2 Samples with Adjusted pH

In order to identify the best performance of bentonite as a blocking agent, solution samples are prepared with various pH values ranging from acidic to alkaline. Every samples will be prepared with different pH of 3, 5, 7, 9 and 11. 0.1M of sodium hydroxide (NaOH) and 0.1M of hydrochloric acid (HCl) is prepared to adjust the pH of every samples. PH meter is used in this section in order to determine the pH value of the samples. Then, the mixed solution is stirred overnight at 300rpm with room temperature (approximately 21°C) before it is tested with turbidimeter.



Figure 3.2. Bentonite solution is stirred overnight at 300rpm

### 3.2.3 Addition of n-dodecyltrimethylammonium bromide (DTAB)

In this process, different concentration of cationic surfactant is prepared in order to identify the optimum concentration of cationic surfactant when react with bentonite. The critical micelle concentration (CMC) of n-dodecyltrimethylammonium bromide (DTAB) at room temperature was determined to be  $15.7 \times 10^{-3}$  mmol/kg (Chauhan et. al, 2014). Thus, 5 different concentration of DTAB is prepared which are  $11.7 \times 10^{-3}$  mmol/kg,  $13.7 \times 10^{-3}$  mmol/kg,  $15.7 \times 10^{-3}$  mmol/kg,  $17.7 \times 10^{-3}$  mmol/kg and  $19.7 \times 10^{-3}$  mmol/kg and mixed with  $20\mu\text{m}$  nanoclay and  $32\mu\text{m}$  drilling mud solutions, respectively. All samples were stirred overnight before turbidity test is conducted. Below shows the mass added into the solutions with different concentration of DTAB.

Table 3.1: concentration of DTAB

Concentration of DTAB (mmol/kg)	Mass added per 100mL (g/mol)
$11.7 \times 10^{-3}$	0.361
$13.7 \times 10^{-3}$	0.422
$15.7 \times 10^{-3}$	0.484
$17.7 \times 10^{-3}$	0.546
$19.7 \times 10^{-3}$	0.607

### 3.3 Stability Determination

In stability determination, the turbidity of the bentonite is taking into account to compare the settling rate of bentonite with different pH as well as when it is mixed with different concentration of cationic surfactant. Turbidimeter will be used to obtain an accurate value of the turbidity of all samples. In this study, the turbidity results of every samples is collected after it has been stirred overnight. Turbidimeter is run for every 10 minutes for 1 hour and results are collected at every test. In this step, the optimum condition of modified-bentonite samples will be determine and selected samples with good stability results will proceed to the next steps for identifying the properties and characterization.

### 3.3.1 Preparation of bentonite and DTAB mixture with specific pH

After getting the results of bentonite and different concentration of DTAB mixture, the best test results were determined for 20 $\mu$ m nanoclay and 32 $\mu$ m drilling mud at specific DTAB concentration. From the results, the same sample is prepared with selected pH value which are pH5 and pH9. Then, the samples is tested with turbidimeter after it was stirred overnight.



Figure 3.3. Turbidimeter

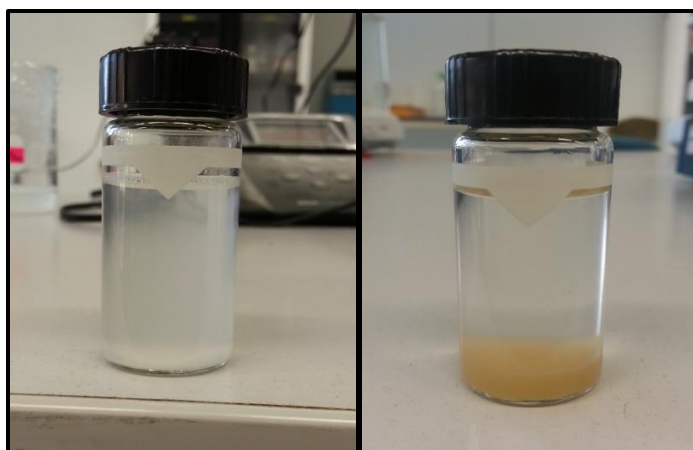


Figure 3.4. Turbidimeter test preparation

### 3.4 Investigation of Rheological Properties

In this study, the rheological properties such as plastic viscosity and its yield point of an optimum modified-bentonite sample is determined by using Model 35 Viscometer. This instrument shows direct-dial reading with six different speed which are 3rpm, 6rpm, 100rpm, 200rpm, 300rpm and 600rpm. This viscometer is able to

determining the rheological properties of fluids, Newtonian and non-Newtonian. The instrument is originally designed so that the viscosity results is in centipoise (cP) or millipascal second (mPa.s).

The general procedure when conducting the viscometer is firstly the fluid is filled in the annular space between an outer and inner cylinder. While running the viscometer, the outer cylinder is rotated at known velocity to produce a viscous drag exerted by the fluid. Then, the drag created on the inner cylinder is transmitted to a precision spring which cause deflection of the dial reading.

In this study, the optimum stabilize modified bentonite solution is used to determine the rheological properties of the solution. The solution is conducted by using 300rpm and 600rpm. The dial reading ( $\theta$ ) of each speed is recorded when the reading shows stable results while conducting the test. Lastly, manual calculation is performed in order to calculate the plastic viscosity and yield point. The reference in calculating viscosity is based on API Recommended Practice for Field Testing Water Based Drilling Fluid.



Figure 3.5. Model 35 Viscometer

### 3.5 Core Flooding Analysis

In this section, Benchtop Permeability System (BPS) is used for core flooding test by using the selected sample with the best stability results. Sandstone core is used in this activity and its original porosity and permeability is recorded by using Poroperm. The diameter and length of the core sample is measured before it is tested with

Poroperm. Then, the core is saturated in a brine solution for one day before it is tested with BPS instrument. After that, core flooding test is conducted by using a rate of 1 ml/s in a constant ambient temperature. Lastly, the sandstone core is again run in the Poroperm to identify its new porosity and permeability and obtain its difference value.

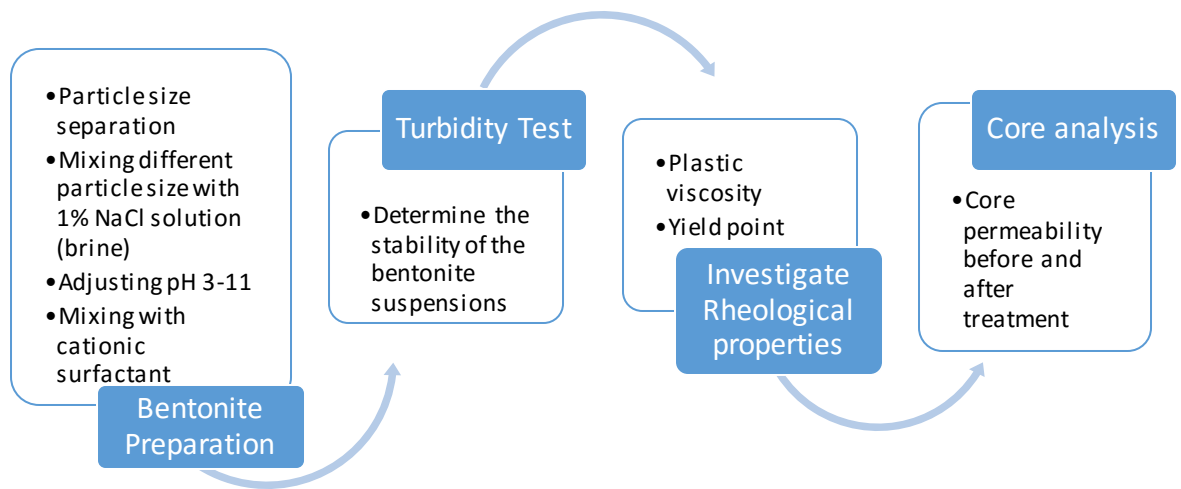


Figure 3.6. Poroperm



Figure 3.7. Benchtop Permeability System

### 3.6 Flow Chart of Experiments



## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 Bentonite Solution Preparation**

In this study, there are two type of bentonite materials were used in order to identify its particles stability when reacting with different pH as well as cationic surfactant. The materials of bentonite are processed bentonite called nanoclay as well as bentonite used as a drilling mud. The purpose of using different type of bentonite is because nanoclay or so called as ‘virgin’ bentonite may produces a good results in adsorption process as it only contain very fine bentonite size without contaminant existence which may increase the effectiveness of the adsorption process. However, the cost of using nanoclay is high especially when applying to industry as it requires huge quantity of bentonite to be injected into the reservoir.

In drilling process, bentonite is the main material in a drilling fluid as it is used mainly for transporting cuttings to the surface, controlling wellbore stability, cooling and lubricating the drillstring and also providing information on wellbore condition. Due to its swelling ability, bentonite has been proposed to use as a blocking agent in the reservoir and it is economically feasible if the drilling mud could be recycled and can be used as a blocking agent. Furthermore, it is also requires less cost as drilling mud is already available at the field as compared using nanoclay which need to be order and obtain from the supplier.

Both types of bentonite are sieved accordingly in order to obtain the smallest size of particles. This is because, as size of particles become smaller it will increase the external surface area of bentonite particles thus more active sites are exposed to the free ions which results in increasing the adsorption rate. Bentonite with particle sizes of 20 $\mu$ m nanoclay, 32 $\mu$ m nanoclay, 32 $\mu$ m drilling mud and 50 $\mu$ m drilling mud are successfully sieved and it is used for further preparation.

In addition, instead of using freshwater 1% of NaCl solution (brine) is prepared before it mixed with bentonite. This is because NaCl solution could prevent the swelling of clay which already existed in the core then plug the core before the solution enable to propagate into the reservoir. However, as brine is used in the solution it will decrease the stability of the solution. Thus, in this study several samples with different properties are prepared in order to determine the optimum stability of the solution.

## **4.2 Turbidity Results**

### **4.2.1 Bentonite solution with varies pH value.**

In this section, bentonite solution is prepared with pH ranging from acid to alkaline which are 3, 5, 7, 9, and 11. 1.0M of Sodium Hydroxide (NaOH) and Hydrochloric Acid (HCl) solutions are used for changing the pH of the sample solutions. The purpose of having a variety of pH value is because every bentonite solution with different pH will produce different turbidity value which indicates the stability of the solution. The interaction between bentonite minerals during collision will determine the dispersion stability of the solution. The particles will tend to attracted with each other during particles collision if the attractive Van Der Waals dominates which cause it to flocculates (Yalcin *et al.*, 2002). Thus, in order to achieve stable dispersions the repulsive force must exceed the attractive particles.

Turbidimeter is used in order to identify the turbidity value for each solution. It is to measure the cloudiness or haziness of a fluid caused by a formation of large numbers of particles which generally cannot be seen by naked eye. The test is conducted for about 1 hour where every reading were taken at every 10 minutes interval. This is to measure the settling rate of the solutions which also determine the stability of the particles. Below shows the turbidity results of every samples. The unit for the turbidity result is Nephelometric Turbidity Units (NTU).



Table 4.1. Results of 20  $\mu\text{m}$  nanoclay (NTU)

Reading	pH 3	pH 5	pH 7	pH 9	pH 11
1	290	286	297	275	263
2	380	251	268	248	206
3	86.4	423	329	228	53.5
4	18	162	409	85.9	23.2
5	12.4	34.4	82.8	22.5	17.9
6	8.92	14.7	24.6	18.6	14.3

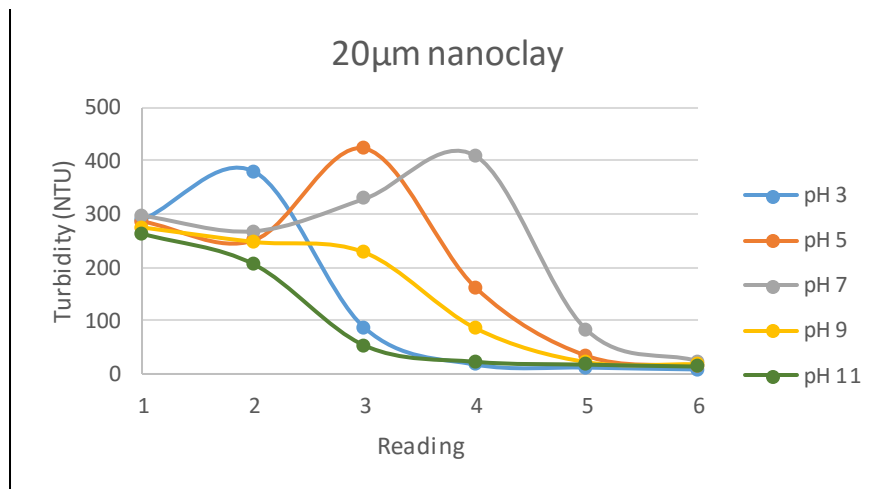


Figure 4.1. Results of 20  $\mu\text{m}$  nanoclay (NTU)

Table 4.2. Results of 32  $\mu\text{m}$  nanoclay

Reading	pH 3	pH 5	pH 7	pH 9	pH 11
1	299	286	250	270	274
2	499	268	235	262	332
3	62.2	363	272	212	45.3
4	14.3	522	455	105	22.5
5	10	168	179	86.7	16.9
6	9.13	50.4	56.4	45.6	13.5

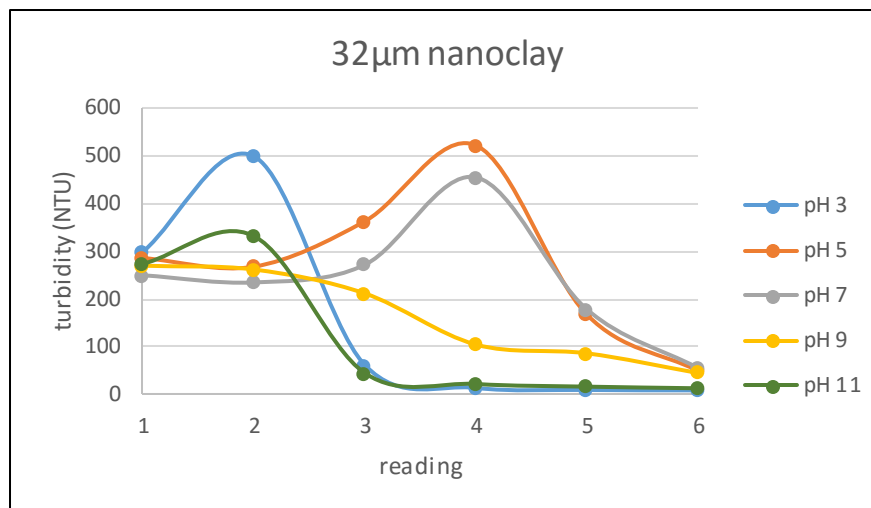


Figure 4.2. Results of 32 µm nanoclay (NTU)

The results of 20µm of nanoclays show that at first reading most of the samples give a result of average 280NTU. Then, at pH 3 the reading suddenly increase to 380NTU before it start to decrease rapidly at the next reading. At pH5, at first 2 readings the solution is stable but increased at reading 3 and then decreasing. Meanwhile, pH7 shows more stable solution as it shows gradually increasing of turbidity values until reading 4 which indicates that there is a repulsive force dominates between particles which prevent it aggregates and settled down. However, both alkaline samples, pH9 and 11 shows poor reading of turbidity as it decreasing rapidly from early reading due to its unstable particles which cause it to attract with each other and flocculates.

For 32µm nanoclay samples, the initial reading of turbidity of all samples are ranged between 276NTU. As shown in the figure 4.2, the pattern of the turbidity results is quite similar with the 20µm nanoclay samples where low pH (pH3) will have a high turbidity at reading 2 and then decreased rapidly at reading 3 to 6. Then, at pH 5 and 7 the solutions are stable from reading 1 to 3 and increased slidely at reading 4 before it start to decreased. Whereas at high pH (pH 7 and pH 11), the results shows unstable solutions where the turbidity results continue to decreased from reading 2 to 6 which indicates the particles flocculates and settled down at early times.

As shown in both results, bentonite particles are stable between pH5 and 7 where it takes a longer time for the particles to be settled down due to the domination of repulsive force of particles during the collision events. It is strongly stated by Choo et. al (2015), the value of the zeta potential decreased as more HCl is added into the

solution. Therefore, aggregation of particles will occur due to a decrease in the stability of the suspension.

Table 4.3. Results of 32  $\mu\text{m}$  drilling mud

Reading	pH 3	pH 5	pH 7	pH 9	pH 11
1	399	494	431	488	485
2	516	900	890	900	900
3	47.5	900	72.1	396	134
4	30.4	472	34.9	75.6	28.5
5	25.7	99.8	28.9	32.8	13.8
6	22	40.8	23.5	24.8	12.4

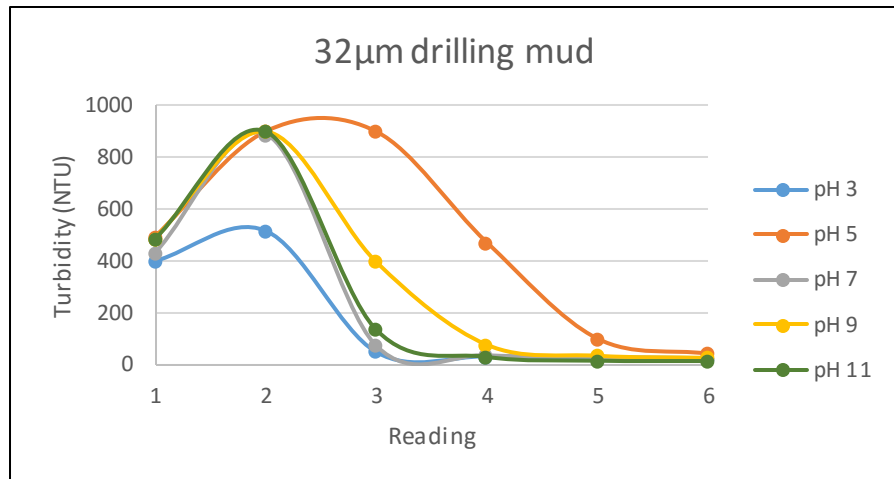


Figure 4.3. Results of 32  $\mu\text{m}$  drilling mud (NTU)

Table 4.4. Results of 50  $\mu\text{m}$  drilling mud

Reading	pH 3	pH 5	pH 7	pH 9	pH 11
1	498	406	416	411	551
2	85.7	489	152	158	34.7
3	50	191	36.9	32.8	20.6
4	38.8	39.9	25.9	23.8	8.93
5	33.1	23.7	22.5	18.4	5.21
6	27.7	16.5	18.6	15.7	4.22

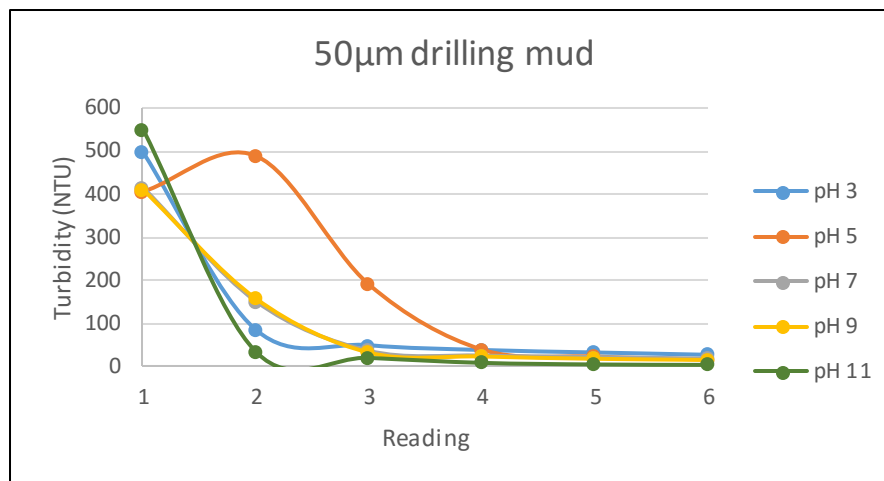


Figure 4.4. Results of 50 µm drilling mud (NTU)

As shown in figure 4.3, 32µm of drilling mud produce a high turbidity value at average of 460NTU at first reading. Then, all samples except sample with pH3 were increased rapidly at reading 2 and start to decrease from reading 3 to 6. This shows that the particles expand at early 10 minutes and start to flocculates with each other when attractive Van Der Waals dominates in the solution thus decrease the turbidity value as the solution become clearer due to the sedimentation of the particles. However, pH5 sample shows slower settling rate as compared with other samples when the turbidity value is still high at reading 3 and decreased slowly until reading 6.

Different with 50µm of drilling bentonite, all samples except sample with pH5 shows rapid in decreasing value of turbidity at early time which indicates unstable particles due to the attractive force is higher than the repulsive force between particles. Moreover, from the results it shows that at this particular particle size, the adsorption process between bentonite and the solution is less effective as less surface area of every particle of bentonite is exposed to the free ions. This is results in coagulating of same bentonite particles which cause the bentonite to settle down rapidly.

#### 4.2.2 Bentonite solution with varies DTAB concentration

In this section, bentonite solution is prepared with different DTAB concentration in order to determine the optimum concentration of DTAB which achieve the most stable bentonite solution. Bentonite solution with particle size of 20µm nanoclay and 32µm drilling bentonite is used in this section which every sample is prepared with five different concentration of DTAB. The critical micelle concentration (CMC) of n-dodecyltrimethylammonium bromide (DTAB) at room

temperature was determined to be  $15.7 \times 10^{-3}$  mmol/kg (Chauhan et. al, 2014). Thus, 5 different concentration of DTAB is prepared which are  $11.7 \times 10^{-3}$  mmol/kg,  $13.7 \times 10^{-3}$  mmol/kg,  $15.7 \times 10^{-3}$  mmol/kg,  $17.7 \times 10^{-3}$  mmol/kg and  $19.7 \times 10^{-3}$  mmol/kg. Below shows the results of the turbidity test of every sample.

Table 4.5. Results for 20 $\mu$ m nanoclay with DTAB

	Turbidity reading (NTU) for 20 $\mu$ m nanoclay with DTAB				
Reading	$11.7 \times 10^{-3}$ mmol/kg	$13.7 \times 10^{-3}$ mmol/kg	$15.7 \times 10^{-3}$ mmol/kg	$17.7 \times 10^{-3}$ mmol/kg	$19.7 \times 10^{-3}$ mmol/kg
1	878	939	649	736	837
2	594	609	172	246	215
3	417	478	79.3	124	95
4	303	387	47.6	84	77.9
5	213	294	19.5	58.7	30.9
6	183	241	15.6	35.3	21.1

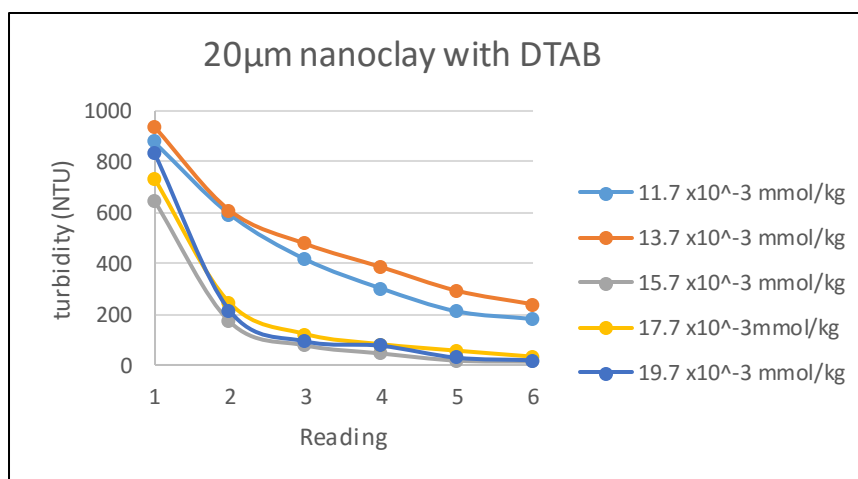


Figure 4.5. Results for 20 $\mu$ m nanoclay with DTAB (NTU)

Table 4.6. Results for 32 $\mu$ m nanoclay with DTAB

	Turbidity reading (NTU) 32 $\mu$ m nanoclay with DTAB				
Reading	$11.7 \times 10^{-3}$ mmol/kg	$13.7 \times 10^{-3}$ mmol/kg	$15.7 \times 10^{-3}$ mmol/kg	$17.7 \times 10^{-3}$ mmol/kg	$19.7 \times 10^{-3}$ mmol/kg
1	744	774	955	953	959
2	246	391	520	331	331
3	115	235	318	147	163
4	58.1	175	228	74.1	95.1
5	21.4	132	170	47.9	52.6
6	18.3	75	114	24.7	45.4

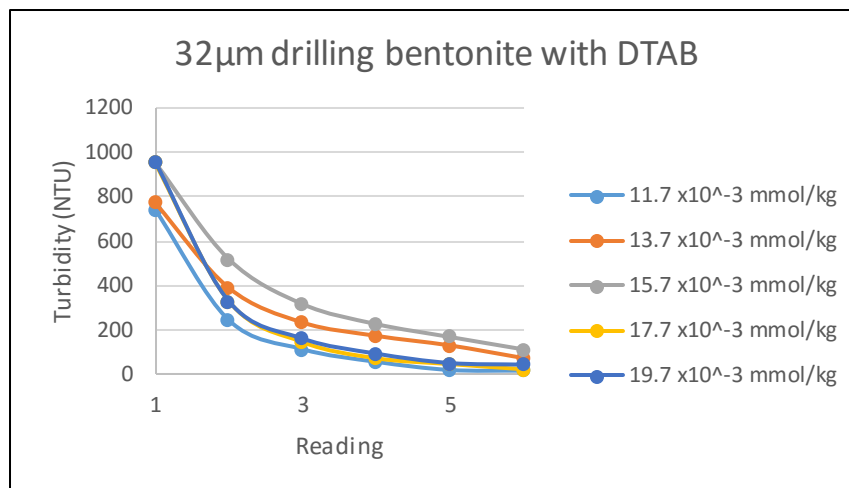


Figure 4.6. Results for 32µm nanoclay with DTAB (NTU)

As refer to Figure 4.5, the highest turbidity value is obtained when  $13.7 \times 10^{-3} \text{ mmol/kg}$  is added into the solution. It is also shows that it gives the most optimum solution stability because the turbidity value from reading 1 to 2 is decreasing lesser as compared to other solutions. In higher concentration, it shows rapid in decreasing turbidity value from reading 1 to 2 and having a low value for the rest testing. However, in lower concentration which is  $11.7 \times 10^{-3} \text{ mmol/kg}$  the stability of the solution is likely same with  $13.7 \times 10^{-3} \text{ mmol/kg}$  but has less value in turbidity.

In addition, as refer to Figure 4.6 the trend of the turbidity results of the solutions are most likely same which they decreased rapidly at early times. However, too much and too less of DTAB concentration gives unstable solution as  $11.7 \times 10^{-3} \text{ mmol/kg}$  concentration has lowest turbidity value while  $19.7 \times 10^{-3} \text{ mmol/kg}$  concentration shows rapid decreased in turbidity value at early times. Thus, from the results it shows that additional of  $15.7 \times 10^{-3} \text{ mmol/kg}$  of DTAB concentration gives the highest turbidity results and the most stable solution as compared to other solutions.

The bentonite suspension is said to be unstable due to electrostatic attraction between negatively charged faces and positively charged edges (E-F) of the particles (Ebru *et al.*, 2006). This is may be due to the excessive present of sodium chloride and potassium chloride in the surfactant. Thus, the remaining floc diameter of solution will deposited below the solution. The optimum result of modified-bentonite sample is the solution which do not have or less amount of the deposit below the solution.

Moreover, as compared with 20 $\mu$ m nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration and 32 $\mu$ m drilling mud with 15.7x10<sup>-3</sup>mmol/kg of DTAB concentration both show decreasing in turbidity value from reading 1 to 6 but 32 $\mu$ m drilling mud with 15.7x10<sup>-3</sup>mmol/kg of DTAB concentration shows more rapid decreasing turbidity value at early times and has less value for the rest of the results. Even though at first reading of 20 $\mu$ m nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration has lower value which is 939NTU as compared with 955NTU but it decreased slowly and more stable than 32 $\mu$ m drilling mud with 15.7x10<sup>-3</sup>mmol/kg of DTAB concentration. According to Ebru (2006), at high surfactant concentrations it will increase the positively charged surfactant which cause decreasing of the zeta potential of the bentonite thus coagulation is occurred.

Different results in both comparison solution is may be due to the several factors which are due to the size of the particles and the type of the bentonite itself. The smaller the size of bentonite particles will results in efficient adsorption of bentonite with DTAB particles. This is because as smaller the particle size, more space area is exposed for the adsorption process thus optimum number of particles could attracted with each other and stabilized the solution. In addition, the existence of contaminant in drilling mud may affect the adsorption process as other particles may ionized in the solution and attracted to bentonite particles instead of DTAB particles thus reduce the stability of the solution.

#### **4.2.3 Sample of 20 $\mu$ m nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration with adjusted pH value.**

The most stabilized solution from all tested solutions is 20 $\mu$ m nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration. This solution is in neutral solution condition which is pH7. In this section, the solution is adjusted with acidic and alkaline solution which are pH5 and pH9, respectively. This is to determine which solution is more stable when having a different solution condition. Below shows the turbidity results of the samples.

Table 4.7. Results of sample with adjusted pH

Reading	turbidity reading (NTU)		
	pH 7	pH9	pH5
1	939	909	771
2	609	304	123
3	478	155	60.4
4	387	90.6	26
5	294	75.1	22.8
6	241	61.3	19.4

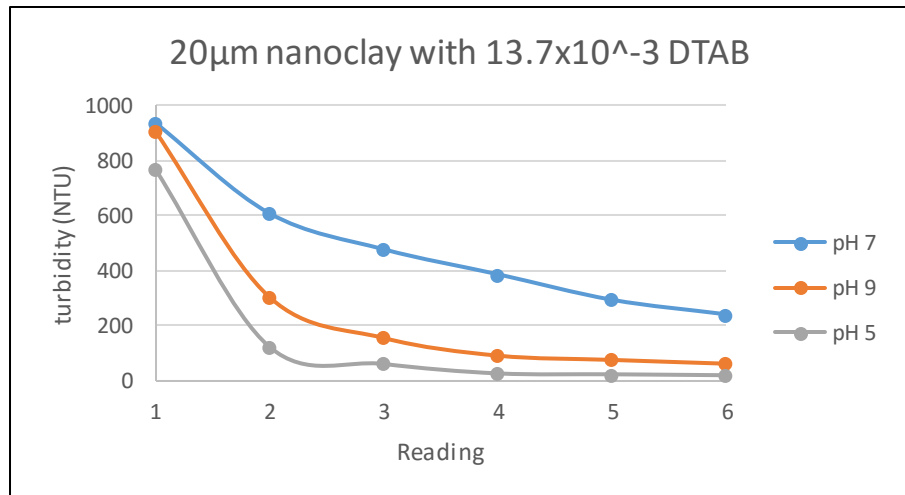


Figure 4.7. Results of sample with adjusted pH

As shown in figure 4.7, the solution with adjusted pH to acidic and alkaline condition shows unstable solution condition where it shows rapid in decreasing turbidity value at early times and start to stable at low values. Meanwhile, solution with pH7 shows slow decreasing turbidity value and has a highest turbidity value compared to other solutions. Thus, in this case solution with pH7 has more stabilized solution compared to other solutions with pH5 and pH9.

#### 4.2.4 Turbidity results for Different Agents

In this section, the optimum stabilized solution obtained in the test which is 20µm nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration is compared its stability with nanoclay solution with different agents. The agents that has been tested are grafted polymer and anionic surfactant (Sodium dodecyl sulfate, SDS). The unmodified bentonite solution has also been tested in order to compare and proved the



importance of adding agent into the bentonite solution for its stability. The results are obtained from the previous Final Year Project which is for the purpose of finding the best permeability modifier in Enhanced Oil Recovery (EOR) method. Below shows the normalized turbidity results for bentonite solution with various agents.

Table 4.8. Results of normalized turbidity results for different agents

Reading	Normalized Turbidity Results, %			Original Bentonite
	grafted polymer	SDS	MTAB	
1	100	100	100	100
2	95.96153846	64.36781609	64.85623003	83.72093023
3	94.80769231	31.26436782	50.90521832	80.23255814
4	93.46153846	29.88505747	41.21405751	44.18604651
5	92.11538462	28.73563218	31.30990415	8.139534884
6	91.53846154	28.27586207	25.6656017	6.511627907

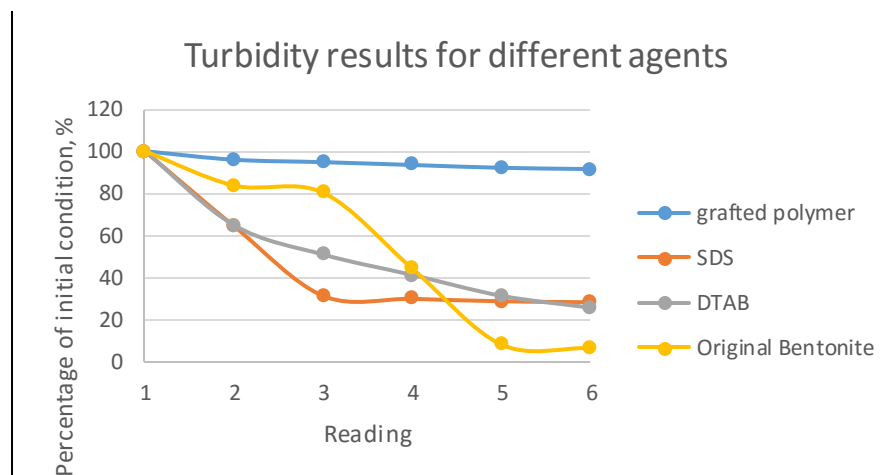


Figure 4.8. Results of normalized turbidity results for different agents

As shown in figure 4.8, bentonite solution with grafted polymer has the most stable solution where it just has a minor decreasing turbidity value from reading 1 to 6. While for bentonite with SDS, it shows rapid decreasing value from reading 1 to 3 and start to stable from reading 3 to 6 at low value. Bentonite with DTAB shows constant decreasing value of turbidity thus it is more stable than SDS. Lastly, bentonite solution without agent shows stable at early times which is from reading 1 to 3 and start to decrease rapidly from reading 3 to 5 and stable to reading 6 at lowest value. This shows that bentonite without agent added into the solution is the most unstable solution as compared with others because bentonite particles will naturally swell when

in present of water and started to coagulate due the attractive force between bentonite particles are stronger than the repulsive force.

Different type of agents has been tested in order to compare and determine the most stable solution when react with bentonite by testing the turbidity of the solutions. A stabilized solution means that the solution has a stable turbidity values throughout the test. Bentonite with agent added into the solution has most stabilized solution when the agent could prevent the domination of Van Der Waals attracting force between bentonite particles. This is by filling every surface area of the bentonite particles with the agent particles thus dominating the repulsive force. This results in preventing the coagulation and settling down of bentonite particles.

### 4.3 Rheological Properties Results

In this section, the viscosity of 20 $\mu$ m nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration is determined by using Model 35 Viscometer. According to Lagaly (2000) the stability state and the rheological properties of bentonite dispersions are strongly influenced by organic cations. Thus it is important to determine the rheological properties of this solution. The viscometer is designed with six different speed which are 3rpm, 6rpm, 100rpm, 200rpm, 300rpm and 600rpm. The purpose of having different speed is because different type of fluid required different speed in measuring viscosity. For instance, it is required to use speed of 600rpm when measuring strength of gel.

The viscosity of modified bentonite solution may be read directly from the dial reading when 300rpm and 600rpm is used for this testing. This is because according to the manual the rotor-bob-torsion spring combination when using 300rpm and 600 rpm is R1-B1-F1. This parameter is used when 300rpm and 600rpm is used during the test. PV which represent the slope of the straight line between two radial reading meanwhile YP represents the theoretical point at which the intercept point of straight line and the vertical axis. Thus, PV and YP is calculated by using the equation 4.1 and 4.2, respectively:

$$PV (cP) = \theta(600rpm) - \theta(300rpm) \quad (4.1)$$

$$YP \left( \frac{lb}{100ft^2} \right) = \theta(300rpm) - PV \quad (4.2)$$

According to the test conducted, the dial reading of 600rpm and 300rpm are 4 and 2.5, respectively. Therefore, it is indicate that the plastic viscosity of the modified bentonite solution is 1.5cP. Meanwhile, the yield point of this solution is 1 lb/100ft<sup>2</sup>.

#### 4.4 Core Flooding Test Results

In this section, the selected solution which is 20µm nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration is tested by using Benchtop Permeability System (BPS) in order to determine the effectiveness of the solution when applying to the core sample. It is mean that, the objective of conducting this experiment is to identify the reduce amount of porosity and permeability of the core sample after it is treated with the modified bentonite solution.

Poroperm is used in this study before the core is tested with BPS in order to determine the porosity and permeability data of the original sample. The data is listed in the Table 10. In addition, helium gas is used to measure the porosity and permeability of the core and it is different with BPS where liquid permeability is measured instead of gas permeability. Therefore, Klinkenberg Effect is used to convert the air permeability to liquid permeability by using equation 4.3:

$$k_l = \frac{k_g}{[1 + \frac{b}{P}]} \quad (4.3)$$

Where:

$k_l$  = liquid permeability, mD

$k_g$  = gas permeability, mD

$b$  = constant for a particular gas in a given rock type, psi

$P$  = mean flowing pressure, psi

Table 4.9. Poroperm Data

<b>Sandstone Core Data Properties Before BPS Test</b>					
<b>Weight,g</b>	<b>Diamete r, mm</b>	<b>Length , mm</b>	<b>Porosity, %</b>	<b>Air Permeab ility, mD</b>	<b>Liquid Permeab ility, mD</b>
186.59	37.95	75.04	16.8	37.03	36.52

The core is saturated with brine solution in order to fill the empty spaces of the sample with brine solution and it is stored for 24 hours in a desiccator. Desiccator with pump is used to extract the gas from the pore and allows in replacing it with brine solution. Then, the core sample is tested with BPS at constant temperature by using ambient temperature as well as constant flowrate which is 1ml/s. Then, results of liquid permeability and pressure difference is determine and constructed in a graph. The results are shown in figure 4.9.

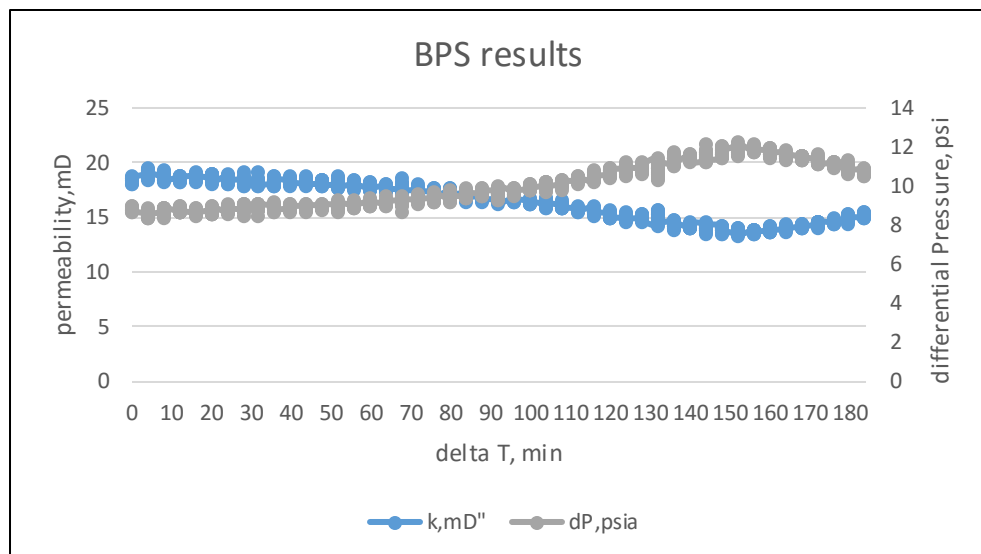


Figure 4.9. BPS Results

As shown in the results, at early times it shows constant differential pressure which is around 9psia and liquid permeability of 19mD because during that time the modified bentonite solution is tried to flow and push the saturated brine solution out from the pore spaces before it enables to plug the pore. After that, as time is increasing the liquid permeability is reduced to 13mD at approximately  $\Delta t= 164$ min and start to increase to 15mD a few minutes after that until the end of the testing. The differential

pressure shows an increasing values as liquid permeability is reduced because during that time it requires more pressure for modified bentonite solution to flow in the core as more pore has been blocked by it. As shown in Table 4.9, the original liquid permeability of core sample is 36.52mD. After that, the permeability is determine to decrease until 13mD during the core flooding test. Therefore, in this study it shows that modified bentonite solution is able to reduce the permeability of the core sample in about 64% from the original permeability.

## **CHAPTER 5**

### **CONCLUSION**

As shown by various results in the present investigation, bentonite solution needs to be modified by adding an effective agent with specified concentration to stabilize its suspension. The unmodified bentonite solution tends to swell and plug the pore in the pay zones before it could penetrate into the reservoir. This could improve the displacement by plugging the pore spaces in that region. This can be proved by the results of the bentonite solution with adjusted pH. From the results, all bentonite solution with adjusted pH shows less stabilization of solution as compared with the modified bentonite solution. Thus, it is found that the bentonite solution having it most stability with addition of specific DTAB concentration. From the results, it is found that the most stabilized bentonite solution is 20 $\mu$ m nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration.

The performance of the chosen permeability modifier which is 20 $\mu$ m nanoclay with 13.7x10<sup>-3</sup>mmol/kg of DTAB concentration is determined by using Benchtop Permeability System (BPS). The modified bentonite solution is able to reduce the liquid permeability of the core sample in about 64% from the original permeability. Therefore, it shows that bentonite is proved to have a tendency to be used as a blocking agent in a permeability modifier method in order to improve the successful of EOR project. Moreover, laboratory testing helps in the improvement of permeability modifier by identifying the problems occurred during the testing and could be improved in further actions. In addition, the rheological properties of the modified bentonite solution is determine and the result of plastic viscosity is 1.5 cP and yield point is 1 lb/100ft<sup>2</sup>. The value of plastic viscosity is low as low amount of bentonite is used in this study and it requires low power to pump the solution to the well when it is apply in the industry. Whereas, the yield point is low which means it is very sensitive to any force or stress occurred in the solution as it may change it characteristics when more stress exerted to the solution.

## **CHAPTER 6**

### **RECOMMENDATIONS**

There are several recommendations that can be improved for this study; firstly, the size of bentonite particles gives huge impact on the stability of the solution. The adsorption process is more effective when the size of bentonite particles are smaller. The larger the size of particles will results in faster settling rate in the solution. In this study, the smallest size of particles that could be sieved is only 20 $\mu$ m of nano-clay and it is better to have a smaller size of particles in the future for the purpose of improving the adsorption process thus results in more stabilized bentonite solution.

Secondly, a salinity test could be conducted for future studies to identify the best salinity condition of the bentonite solution. There are clay which already exist in the reservoir itself and tend to swell and plug the pores when bentonite with freshwater is injected into the reservoir. A saline bentonite solution could prevent clay swelling activity and helps bentonite solution to propagate through the required high permeability region. In this study, only 1% of NaCl solution is prepared for all bentonite solution which could be test with 2% or 4% of NaCl solution in future studies. Refractometer is an instrument which is used to measure the index of refraction by producing a specific critical angle. The test will determine the degree of the salinity of the solution thus results in the rate of bentonite swelling ability.

Thirdly, it is important to have a specific composition of each type of bentonite used in this study because bentonite composition gives an important role in producing a good stability results of the solutions. For instance, drilling bentontie may have various contamination because it is already been used for drilling activity. With this available data, the composition of both bentonite can be compared and specific procedure can be conducted to eliminate the unnecessary composition to improve the stability results of both type of solution.

Lastly, the chosen bentonite solution which is in this case is 20 $\mu$ m nanoclay with  $13.7 \times 10^{-3}$  mmol/kg of DTAB concentration could be tested at different temperature condition. This is to determine the stability of the solution when varies temperature is used during the core analysis testing. Even though bentonite is stable at high temperature, the modified bentonite solution with present of DTAB may have different stability condition. Core analysis testing with varies temperature can be conducted by using Relative Permeability System (RPS) however, due to several factors Benchtop Permeability System (BPS) has been used in this studies by using ambient temperature.



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