Phase Behavior & Core Flooding of Low Acid Number Crude Oil for Application in Alkaline EOR

by

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Dissertation submitted in partial fulfillment of
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CERTIFICATION OF APPROVAL

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Approved by,

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CERTIFICATION OF ORIGINALITY

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

(THIVYASHINI A/P THAMILYANAN)

Contents

Abstract

Alkaline flooding is one of the most promising methods to be applied in recovering residual oil in the reservoir. The process behind this method is, when the alkali being injected in the reservoir, it reacts with the naturally occurring acids in the crude oil and produce soaps or in-situ surfactants. The produced surfactants will help in removing oil from the rock surface and escapes from the pore easily. A proper study in the characteristic of the crude oil and reservoir before injecting the alkali can also change the wettability of the rock to the favorable condition. The oil components that react with alkali are not specifically known, although acid number is cited as one. However, the current application of alkaline flooding in the industry is limited to the reservoirs with high acid number crude oil although many there are no proven correlation which shows that acid number is related to the surfactant produced. In this study, two different types of crude oils from Dulang field, high acid number crude oil and low acid number crude are tested with two different alkali, namely Sodium Hydroxide (NaOH) and Sodium Carbonate (Na_2CO_3). Both crude oils and alkalis are tested for compatibility with various salinity and alkaline concentration. Then, the interfacial tension was measured between the alkaline solution and crude oil to observe the IFT reduction. Finally, core flooding is done with optimum concentration of alkaline with both high acid number crude oil and low acid number crude oil with the best selected alkaline, Na_2CO_3. The results shows the possibility of alkaline flooding in low acid number crude oil and increase in the application of alkaline flooding in potential reservoirs.

Acknowledgement

First and foremost I would like to express my thanks to Almighty God on successful completion of this project and report. This dissertation is a milestone in my academic career. I have been fortunate to learn theories, concepts and apply the engineering knowledge that I have learnt through this project.

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CHAPTER 1

1.0 Introduction

1.1 Background of study

As a reservoir continues to produce, along time the reservoir pressure will decrease and much oil will remain in the reserve as residual oil(Cooke Jr., Williams, & Kolodzie, 1974). Numerous Enhanced Oil Recovery (EOR) techniques are being applied in the industry to recover residual oil in the reservoirs. One of the effective recovery methods is alkaline flooding. This method appeal to be more attractive due to its very low cost compared to other methods. Cheap alkalis such as Sodium Hydroxide (NaOH) and Sodium Carbonate ($NaCO_3$) will be injected into the reservoir to react with naturally present acid in the crude oil to produce in-situ surfactants which lowers the Interfacial Tension between the crude oil and rock surface and oil escapes easily.

This project focuses on the cause for recovery in low acid number crude oil using strong and weak alkali. Therefore, all the parameters and factors contributing to the alkaline flooding such as acid number in crude oil, alkaline concentration and type, brine salinity and reservoir condition are tested and determined earlier by compatibility test(Mayer, Berg, Carmichael, & Weinbrandt, 1983). Furthermore, at the end of this study, a comparison study of mechanisms like wettability alteration and emulsification involved in achieving a high oil recovery in low acid number crude is also determined. Lastly, the scope of work and objective of this project has been narrow down for it to be feasible to be conducted within the given time frame.

Although many works have been done earlier in this topic, improvements and a more detailed study has to be conducted in order to get accurate results and correlations. Firstly, the previous work did not focus on the type of emulsion formed when alkaline solution is injected into the reservoir. Therefore, a test will be conducted in this project to identify the nature of the emulsion formed, whether it is oil-in-water emulsion or water-in-oil emulsion. Secondly, the wettability alteration done in the work earlier only used the indirect method which is core flooding but this project will test using the direct method which is using slide glass to test the effect when it is oil-wet or water-wet. Finally, study on displacement efficiency and mobility ratio in alkaline flooding are also will be covered in this project.

1.2 Problem Statement

A minimum acid number in the range of 0.5 to 1.5 mg KOH per gram of oil has been suggested as a pre-requisite for a successful oil recovery of oil through alkaline waterflooding[11]. Application of alkaline flooding has been limited in many cases due to a low content of naturally occurring acid in the crude oil. Crude with low number of acid is said to interact less with the alkali injected, leading to less in-situ surfactant formation. Thus, the recovery of residual oil is less compared to high acid number crude oil as Interfacial Tension (IFT) cannot be reduced(Labrid, 1979). However, there is no proven correlation which states that acid number in the crude is directly related to in-situ surfactant production. Magnitude of enhanced oil recovery and increased production did not correlate neither with acid number nor Interfacial Tension beyond the threshold values(Ehrlich & Wygal Jr., 1977). This project is to investigate the possibilities of alkaline flooding in low acid number crude oil by testing various mechanisms involved in recovering residual oil and determining the adequate condition to apply alkaline flooding in recovering the residual oil.

1.3 Objectives

1. To determine the optimum concentration of aqueous solution for alkaline flooding.
2. To characterize the nature of the emulsion formed, whether it Type I, Type II or Type III emulsion.
3. To examine the emulsion retention using high and low acid number crude.
4. To observe the reduction in interfacial tension.
5. To investigate the recovery of residual oil through alkaline flooding in low acid number crude oil reservoir (core flooding).

1.4 Scope of Study

1. Optimum concentration of aqueous solution
2. Characterization of emulsion
3. Emulsion retention of microemulsion

4. Interfacial tension
5. Core Flooding

1.5 Significance of Project

Low acid number in crude oil limits the application of alkaline flooding in recovering residual oil in the reservoir(Chlwetelu, Neale, Hornof, & George, 1992). But no correlation till date has shown that acid number in crude oil is directly related to reduction in Interfacial Tension. Since alkaline flooding is very cost efficient compared to other methods in enhanced oil recovery (EOR), it is very important to prove that alkaline flooding can also be successful in the reservoir with low acid number crude oil. The research and experiments conducted in this project is to contribute to the study of alkaline flooding in Dulang crude oil.

1.6 Relevance of Study and Time Frame

Enhanced Oil Recovery is an important aspect in the process of recovering residual oil where various tertiary recovery methods are applied in the reservoir. One of the favorable EOR method, alkaline flooding is being studied in this project(Cooke Jr., et al., 1974). This project requires and applies engineering knowledge and oil field familiarization which is vital to a successful petroleum engineer. This project is relevant to the scope of my study which is Petroleum Engineering. The time frame to complete this project is very feasible as I have two semesters to complete it.

CHAPTER 2

2.0 Literature review

2.1 Theory

Enhanced Oil Recovery (EOR) is an artificial method used to recover additional oil after the primary and secondary production. EOR is usually applied to reservoirs which have enough potential for the economical production after cost limitations have been carefully assessed (Bortolotti, Macini, & Srisuriyachai, 2009). There are three main types of EOR, namely, chemical flooding (alkaline flooding, or micellar-polymer flooding), miscible displacement carbon dioxide or hydrocarbon injection) and thermal recovery (steam flood or *in-situ* combustion). The application of each type depends on the temperature, depth, net pay, permeability, residual oil and water saturations, porosity and fluid properties such as oil API ad viscosity (Bortolotti, Macini, et al., 2009).

Alkaline flooding also known as caustic flooding is simple in process and low in cost but its mechanism is very complicated. Alkaline solutions are injected into the reservoir to recover residual oil trapped in the pores. Injected alkali will react with organic acids present in the crude oil and form in-situ surfactants, known as soaps, which help in releasing oil from rock. Alkaline flooding is more attractive compared to various non-thermal processes because alkaline reagents are abundant and quite cheap compared with conventional surfactants (Chlwetelu, et al., 1992). The history of alkaline flooding dated back to the early 1920’s, as a combination of reservoir engineering and chemistry. Interaction of the injected alkali with crude oil, water, brine and rock in the reservoir is a chemistry scope while using interactions to recover oil is reservoir engineering scope (Mayer, et al., 1983).

Till date, no less than eight mechanisms have been suggested (Tong, Liu, Zhang, & Zhu, 1986). All the mechanisms recognize that surface active substances or soaps, formed by certain compounds found in crude oil reacts with alkali, are adsorbed at the oil/water or solid/liquid interfaces as to change the properties of the interface. The adsorption of the soap substance at the interface lowers the interfacial tension (IFT) significantly (Tong, et al., 1986).

Even though the oil components that react with alkali area not specifically known, acidic components have been cited as candidates (Smith, 1993) . While the presence of some naturally occurring acids is a must, no direct correlation has been made to relate acid number and enhanced oil recovery through alkaline flooding (Mayer, et al., 1983). When good alkaline formulations with good mobility control are used, more than 90% oil recovery can be achieved in outcrop and reservoir cores(Yang et al., 2010). Before any alkaline flooding is done, one of the most important task to be done is phase behavior experiments to determine the optimal formulation(Tong, et al., 1986). Therefore, a great deal of screen work should be always carried out.

2.2 Principles of Oil Recovery

Hydrocarbon recovery can be defined as the overall efficiency with which oil is displaced by some other fluid(Okasha & Alshiwaish, 2009). The two important concepts in oil recovery are Mobility Ratio, M, and the Capillary Number, N (Thomas, Scoular, Verkoczy, & Ali, 1999).

2.2.1 Mobility Ratio

Mobility ratio is the ratio of the mobility of displacing fluid(oil in his theory) divided by mobility of displaced fluid. Mobility is defined as permeability divided by viscosity (k/μ). When $M>1$, the displacement is inefficient, because the displacing fluid will flow past much of the displaced fluid. When $M>1$, the displacing fluid will channel past oil ganglia, called “viscous fingering”. For a maximum displacement efficiency, mobility ratios should be less than one, $M<1$, denoted as “favorable mobility ratio”.

M can be made smaller by making the displacing fluid more viscous, usually done by adding polymer to water, or by heating the displaced fluid to make it less viscous (Thomas, et al., 1999).Mobility ratio is also directly proportional to areal or vertical sweep efficiency.

2.2.2 Capillary Number

Capillary Number, N_C can be defined as $\mu v/\sigma$, where μ is the displaced fluid viscosity, v is Darcy velocity, σ is IFT between the displaced and displacing fluids (Thomas, et al., 1999). Capillary number is inversely proportional to residual oil saturation. Capillary number can be increased and residual oil saturation will decrease when oil viscosity is reduced, or increasing pressure gradient, the most significant one will be reducing IFT. A good solvent will lower oil viscosity but the cost will be prohibitive (Thomas, et al., 1999).

2.3 Mechanisms in Alkaline Flooding

2.3.1 Oil-Alkali Interaction

When the injected alkali interacts with and acids present in crude oil precipitation occurs. The interaction between alkali and components in the crude produce surface active agents, known as surfactants. The surfactants can help to release the oil from rock surface. This process is also known as emulsification (Mayer, et al., 1983). Studies (Bortolotti, Macini, et al., 2009) involving the effect of sodium ion on the activity of in situ formed surfactant have shown that activity decreases as sodium concentration increases. Emulsion can be defined as colloidal system in which one phase is dispersed as fine droplets in the other phase where both are equally miscible. There are four types of emulsions, oil-in-water(emulsification and entrainment), water-in-oil(emulsification and entrapment), bi-continuous and isotropic micellar solution. Oil-in-water and water-in-oil are two phase systems, bi-continuous is a three phase system and isotropic micellar solution is a single phase system. The first three types are the most common emulsions.

2.3.1.1 Oil-in-water (O/W)

O/W emulsion involves the mechanism of emulsification and entrainment where oil droplets are dispersed into the water phase. Water exists as the flowing medium. According to Lorrondo, et. al., higher alkali and/or sodium chloride concentration tend to the favor the formation of o/w type.

2.3.1.2 Water-in-Oil (W/O)

W/O emulsion involves the mechanism of emulsification and entrapment. It is also known as oil emulsion because oil exists as the dispersion medium and water as the dispersed phase. W/O emulsion is temporarily mobile and improves the rate of recovery(alters the WOR) but does not decrease the residual oil saturation (Mayer, et al., 1983). Water-in-oil emulsion can improve oil recovery but the disadvantage will be the need for large pressure gradient for normal flow rates because it is usually more viscous than oil, thus it is better to avoid W/O emulsions (Kumar, Dao, & Mohanty, 2012).

2.3.1.3 Winsor Type III

Winsor type III emulsion is a condition where the oil and water are separated into 3 layers. Oil will be on top, microemulsion will be in middle and water at bottom. This type of emulsion is most favorable reservoir as it can carry the oil easily in the form of microemulsion to the surface and the stability is higher.

Emulsions can be broken into two different phases by many ways such as heating, centrifugation, freezing and de-emulsifier application. A stable emulsion is favorable in the reservoir during alkaline flooding. Emulsion stability, as defined by the coalescence rate of oil droplets in water-external emulsions, is determined as a function of salinity and alkaline type (Chang & Wasan, 1980). The emulsion is expected not to break until it reaches the surface or transportation system. Emulsion type and formation depend on the salinity, hydrophilicity of surfactants and WOR, while the transport of emulsion depends on the formations' wettability and pore structure (Kumar, et al., 2012). The application of dilute emulsions and polymers in alkaline flooding is continuously being studied (Mayer, et al., 1983).

2.3.2 Alkali-Rock Interaction

Alkali-rock interaction during alkaline flooding causes wettability alteration (Mayer, et al., 1983). Wettability is the tendency of one fluid to adhere onto solid surfaces in presence of other immiscible fluids (Bortolotti, Macini, et al., 2009). The wettability of a rock varies from strongly water-wet to strongly oil-wet, depending on the interactions between oil, water and reservoir rock(Bortolotti, Gottardi, Macini, & Srisuriyachai, 2009). Some porous mediums that do not show preferential wettability to any one of the two fluids are referred as “neutral wettability” or “intermediate wettability”. Most reservoirs are water-wet because they originated in aqueous environments. Oil wettability is an unfavorable condition because oil tends to be captured onto the rock and matrix surfaces rather than being produced along with the flowing streamline resulting in very poor oil recovery. Wettability is the most important yet least quantified aspect of alkaline flooding.

Many properties of injected water has been proven to affect the amount of oil recovered through wettability impacts (McGuire, Chatham, Paskvan, Sommer, & Carini, 2005). E.F. deZabala, et. al., found that alkali adsorption on the rock surface decreases the amount of alkali available to the oil/water interface, which results in higher tension levels that may be ineffective for the mobilization of residual oil. Therefore a significant alkali adsorption on the rock surface can abrogate in alkaline flooding process.

Reservoir rock interaction with the alkali by far has been the largest contributor for alkaline consumption. High consumption occurs when the clay content is high but slow when quarts, dolomite and calcite exist (Mayer, et al., 1983). This reaction is both reversible and irreversible. Under ideal condition of pH value, salinity and temperature, it is possible to change the wettability of some porous media from oil-wet to water-wet.

2.3.3 Alkali-Water Interaction

Mixing of in the injected alkaline solution with the hardness ions in the reservoir water causes chemical precipitation (Mayer, et al., 1983). According to Mayer, et al., once the alkali contacts the ions, precipitates of calcium and magnesium hydroxide, carbonate or silicate may form depending on the pH, ion concentration and temperature. A careful concentration of alkali should be calculated before injecting as it can cause scale precipitation which is an unfavorable condition.

Any or all the mechanisms stated above can operate in one application.

2.4 Potential Reservoirs for Alkaline Flooding

Identifying reservoirs with potential to recover residual oil economically by alkaline flooding is very important before alkaline flooding is done. In a research done by J.E.Smith (1993) using 239 crude oils for a period of 10 years, chemical properties of crude oil are found to be much stronger indicators of alkaline reactivity than physical properties. J.E.Smith has added : physical properties that generally indicate a heavy, nonvolatile oil correlate weakly with alkaline reactivity; oil form a given formation may have different reactivity tendencies than average due to the characteristics typical to the formation. High molecular weight ring components such as aromatics, naphthenes and asphalts tend to favor reactivity while paraffinic compounds tend to discourage reactivity. Oils from carbonate rocks are found to be more reactive compared to sandstone. A reservoir should contain little or no gypsum, the divalent ion exchange capacity should be less than 5 meq/kg, and the in situ pH should be greater than 6.5 for it to be a candidate for alkaline flooding (French & Burchfield, 1990). All these properties should be studied carefully in determining the feasibility of alkaline flooding in a reservoir.

2.5 Phase behavior

Phase behavior experiments are done to determine the behavior of crude oil, chemicals and aqueous solutions in the reservoir condition. Phase behavior tests requires a careful observation of the compatibility of aqueous solutions and chemical mixtures with crude oil and brine over a period of time for it to reach equilibrium or to be rejected if it fails (Yang, et al., 2010). Yang *et. al.* also mentioned that ideally low viscosity micro-emulsions will form within a few days and ultra-low IFT will exhibit between the crude oil and water phases.

2.5.1 Total Acid Number (TAN)

Total Acid Number (TAN) is widely used as an indicator of the activity of crude oil by determining the quantity of naphthenic acid in crude oil. TAN reflects the free carboxylic groups in the crude oil (Yang, et al., 2010). The measurement is done by determining the amount of milligram of potassium chloride needed to neutralize 1gram of crude oil. Accurate measurements of TAN are reproducible and correlate with IFT properties of crude oils (Fan & Buckley, 2007).

2.5.2 Viscous Oil

A high viscosity oil is more difficult to displace in a reservoir compared to a low viscosity oil but a more viscous oil has many advantages as targets for chemical EOR and the upper limit of oil viscosity to what is considered feasible is much greater than it was 30 years ago (Yang, et al., 2010).

Much larger EOR target is found in high viscous oil reservoir as the water flood residual oil saturation is typically higher in these reservoirs, high porosity and high permeability, so the characteristics take as more attractive compared to light oil reservoirs where they have been water flooded to very low residual oil saturations over many decades (Yang, et al., 2010).

2.5.3 Brine Sanity

In a study done by Okasha & Alshiwaish (2009) to investigate the effect of brine dilution, temperature and pressure on IFT of dead and recombined oil in Arab-D carbonate reservoir, it was found that the dilution of brine reduces the FT between oil and brine which may help in improving oil recovery in case of injecting low salinity brine. The reduction of IFT with brine dilution reflects the potential implication of low salinity flooding in improving oil recovery (Okasha & Alshiwaish, 2009).

2.5.4 Interfacial Tension (IFT)

Interfacial measurements between a crude oil and an alkaline solution have generally been accepted as a screening factor to evaluate the EOR potential of the crude by the alkali (Burk, 1987). IFT behavior exists at any alkali concentrations. During alkaline flooding, the IFT may be reduced to a level that permits the mobilization of residual oil (deZabala & Radke, 1986).

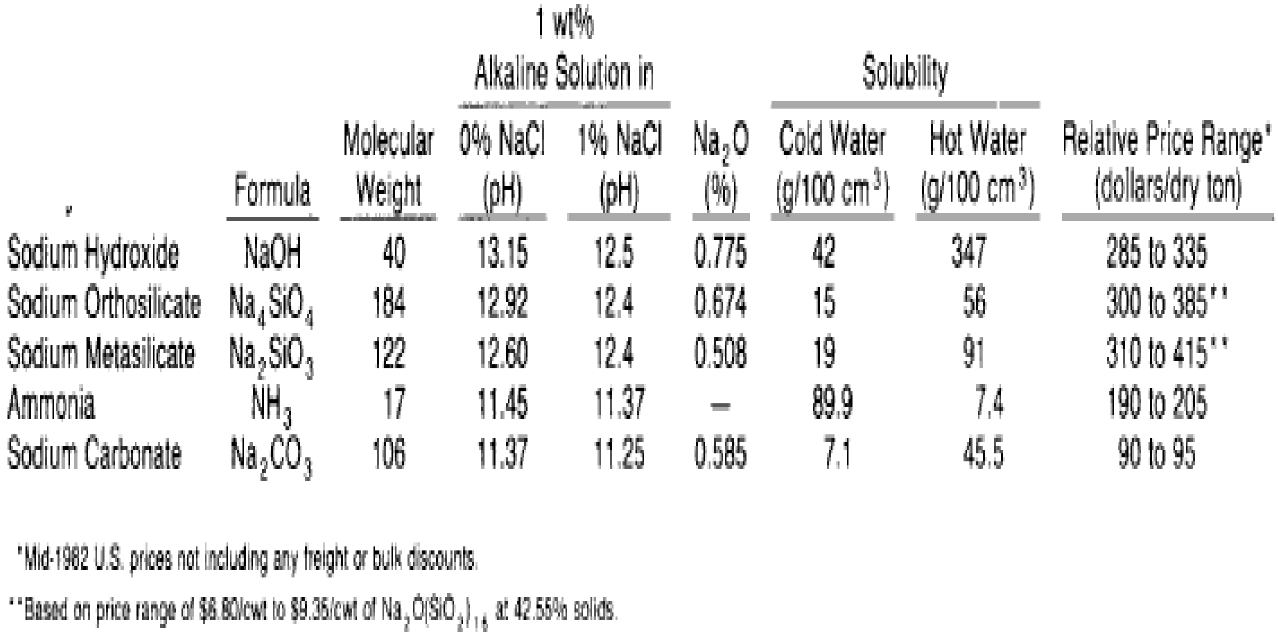
The reduction in IFT between acidic oil and alkaline water is influenced by the pH of the water, the concentration and type of salts in the solution, and the concentration and type of organic acids in the oil (Cooke Jr., et al., 1974). Relatively low IFTs (less than 1dyne/cm) are usually measured by spinning drop method. This method is also applied in this project. The spinning drop apparatus basically consists of a cylindrical glass tube that rotates about its horizontal axis. Inside the tube, oil is dispersed in the denser aqueous phase (alkaline solution). The oil drop stays at the horizontal axis when the interfacial force is counterbalanced by the rotational force. Therefore, the IFT is a function of drop radius, rotational speed and density difference between two phases (Li, Wang, & Gu, 2003; Mayer, et al., 1983).

2.5.5 Core Flooding

Once the suitable surfactant formulations are selected based on their phase behavior, they should be evaluated by testing the formulations through core flooding (Yang, et al., 2010). If the final oil saturation is almost zero, the micro-emulsion screening test is considered a success and the oil recovery performance is good.

2.6 Chemicals

There are five possible alkalis to be used for alkaline flooding(sodium hydroxide, sodium orthosilocate, sodium metasilicate, ammonia and sodium carbonate) but the most widely used alkali will be Sodium Hydroxide(NaOH) (Mayer, et al., 1983). A comparison of alkali was made by Mayer, et.al. and the result is tabulated in Table 1.



	Formula	Molecular Weight	1 wt% Alkaline Solution in 0% NaCl (pH)	1 wt% Alkaline Solution in 1% NaCl (pH)	Na_2O (%)	Solubility Cold Water (g/100 cm^3)	Solubility Hot Water (g/100 cm^3)	Relative Price Range* (dollars/dry ton)
Sodium Hydroxide	$NaOH$	40	13.15	12.5	0.775	42	347	285 to 335
Sodium Orthosilicate	Na_4SiO_4	184	12.92	12.4	0.674	15	56	300 to 385**
Sodium Metasilicate	Na_2SiO_3	122	12.60	12.4	0.508	19	91	310 to 415**
Ammonia	NH_3	17	11.45	11.37	–	89.9	7.4	190 to 205
Sodium Carbonate	Na_2CO_3	106	11.37	11.25	0.585	7.1	45.5	90 to 95

*Mid-1982 U.S. prices not including any freight or bulk discounts.

**Based on price range of $6.80/cwt to $9.35/cwt of $Na_2O(SiO_2)_{1.6}$ at 42.55% solids.

Table 1 Comparison of alkali. Retrieved from (Mayer, et al., 1983)

Referring to Table 1, sodium hydroxide has poor solubility in cold water while Ammonium Hydroxide has low solubility in hot water. Sodium orthosilicate has been identified to a effective pre-flush for chemical/surfactant floods. Potassium-based alkalis usually will not be preferred economically because of their high cost, but in areas where sodium based alkalis are not suitable due to clay-swelling or injectivity problems, ammonia or potassium-based alkalis become the option. J.H Burk's research showed that sodium carbonate solutions are less corrosive to sandstone than sodium hydroxide or sodium orthosilicate and sodium carbonate buffering action can reduce alkali retention in the rock formation.

Alkali selection for a particular reservoir will depend on the reservoir conditions, comparative laboratory testing and availability and overall economics.

There are two alkalis to be tested in this project, Sodium Hydroxide (NaOH) and Sodium Carbonate (Na_2CO_3). These alkalis dissociate and cause an increment in pH value, by the following reaction :

1. Sodium Hydroxide : $NaOH \xrightarrow{\hspace{2cm}} Na^+ + OH^-$
2. Sodium Carbonate : $NaCO_3 \xrightarrow{\hspace{2cm}} 2Na^+ + CO_3^{2-}$

Crude oil samples to be used in laboratory screening should be free from chemical additives, such as emulsion breakers and corrosion inhibitors (JENNING JR., 1975). The crude oil sample to be used in this project will be from Dulang field.

2.7 Challenges in Alkaline Flooding

Conditions such as extremely high temperature, high salinity and hard brine pose a great challenge in applying chemical EOR process (Yang, et al., 2010).

2.7.1 Hard Brine

Each petroleum reservoir has some hardness (divalent cations) in the water and poses a great threat during the injection of chemicals in the reservoir. Divalent cations can cause precipitations when they meet and react with alkali, polymer and surfactant and plugs the reservoir (Yang, et al., 2010). There, careful measurements should be taken to test the compatibility of these chemicals with brine.

2.7.2 Micellae formation

When the acids in oil phase combines with caustic soda, it produces water-water soluble sodium salts, known as micellae. Critical micellae concentration depends mainly on the concentrations of caustic soda and salt in equilibrated aqueous phase, thus the formation of micellae in aqueous phase should not be ignored (Tong, et al., 1986).

2.7.3 Heavy Oil Reservoir

Density of heavy oil usually ranges from 11 ° to 20° API (Chlwetelu, et al., 1992). Heavy oil has low mobility because of its high viscosity, therefore the mobility ratio would be unfavorable in any fluid displacement process (Thomas, et al., 1999). Use of an alkali would promote the formation of emulsion which in turn will make the effective mobility ratio less unfavorable. The emulsion, usually micro-emulsion, will lower the mobility of the displacing fluid through entrainment and entrapment. Flow rates and sweep efficiency are also key problems in heavy oil recovery. Micro-emulsions are a mixture of water, a naturally occurring solvent, an alcohol co-solvent and a surfactant blend of linear and branched ethoxylates (Paktinat, Pinkhouse, Williams, & Penny, 2005).

2.7.4 Loss of Alkali

Loss of alkali happens as a result of adsorption and reaction with minerals in the reservoir. The loss will be more if the clay content in the reservoir is high (Thomas, et al., 1999). When designing the alkaline formulation for alkaline flooding in reservoir, alkaline consumption by the rock and the fluids saturating the rock should be taken into account (Bortolotti, Macini, et al., 2009). Presence of clay in a reservoir contributes to alkali loss and is affecting by the amount and type of clay minerals present, connate water composition, caustic concentration, temperature and time of contact (Ehrlich & Wygal Jr., 1977). In the research done by Bortolotti, et al., addition of sodium chloride to the alkaline solution appeared to help decrease the amount of alkali consumed, although did not contribute to additional oil recovery. Alkaline consumption by reservoir rock is predictable from X-Ray diffraction analysis of mineral composition.

Insufficient attention to any of the above mentioned factors could seriously impair the efficacy of the alkaline system (Chlwetelu, et al., 1992).

CHAPTER 3

3.0 Methodology / Project Activities

3.1 Acid Number Determination

Experiment Title	**Acid Number Determination**	
Objective	To determine the acid number in crude oil and add carboxylic acid to crude oil to make the crude oil high acid content.	
Theory	Crude oil naturally has acids in them. But these acids varies in each crude. Some has high and some low. This experiment will measure the acid number in the crude. The method described is a color titration method ASTM D974 in order to determine at which titration volume should be stopped, a pH meter was used to indicate that the solution has been neutralized due to the fact that crude oil is black to brown in color but it is visible in the solvent.	
Methodology	**Equipment/Apparatus**	burette, graduated cylinder, weighing scale, retort stand, beaker 1 liter
	Material/Chemical	Ethanol or Isopropanol, Potassium Hydroxide, p-naphtholbenzene Solution, Carboxylic acid like acetic acid, crude oil and pH meter.
Hazard Identification	All the chemicals involved are volatile. Inhalation can cause irritation to the lungs and will cause irritation to the skin.	

Experiment procedure	a. **Preparation** i. Alkaline solution titration was prepared by weighing out 0.1 mole of KOH (= 2.805 g), dissolved it in ethanol to make the total volume equal to exactly 500 ml. This gave 0.1M KOH solution in ethanol. ii. Then 500 ml solvent for crude oil is prepared consist of 250ml tolune,225ml ethanol, and 25ml deionized distilled water.3 to 4 drops of phenolphthalein solution were added and titrated with 0.1M KOH alcoholic solution. The color of the indicator changes from colorless to pink. iii. Accurately 10g of sample is weighed and was added into 100 ml of the solvent in the beaker. The sample is swirled completely until dissolved by the solvent.
	b. **Sample testing** i. Then it is titrated immediately with 0.1M KOH alcoholic solution at room temperature, using a 25 ml burette. The solution is swirled vigorously until the color of the indicator changes from colorless to pink as was with solvent and crude oil neutralization.

3.2 Compatibility Test

Experiment Title	**Compatibility Test of Alkali Solution**	
Objective	To find the optimum concentration and salinity aqueous solution using NaOH, NaCO3 with crude oil and brine water(NaCl) or distilled water to avoid precipitation of micro white particle	
Theory	Crude oil naturally has acids in them. But these acids varies in each crude. Some has high and some low. This experiment will test the compatibility of alkali solution with the acidic and de-acidified crude. solutions are made with mass percentage according to major papers rather than molarity of the solution.	
Methodology	**Equipment/Apparatus**	Test tube, graduated cylinder, weighing scale
	Material/Chemical	Sodium Hydroxide, Sodium Carbonate, Sodium Chloride, Distilled water, crude oil
Hazard Identification	All the chemicals involved are volatile. Inhalation can cause irritation to the lungs and will cause irritation to the skin.	
Experiment procedure	**a. Preparation** i. The mass of sodium hydroxide needed is calculated using the following formula: Mass = (volume x mass percentage) / (100 - mass percentage) *For example, to make a 1 percent solution using 60 mL of distilled water, this equation used to determine the amount of sodium hydroxide to be used: Mass = 60 x 1 / (100 - 1) = 0.6 g ii. The calculated amount of sodium hydroxide is weighed on the scale. Distilled water of 60 mL is poured into the test tube, and add sodium hydroxide. The solution is mixed with the spoon or gently swirl the test tube until the salt dissolves completely. iii. Then, the mass of sodium chloride is calculated using	

	above formula for example 1% solution in 60 mL then add into test tube. iv. About 40mL crude oil (de-acidified and acidic) is measure and added into test tube to make the solution 100 mL.
	b. Sample testing i. The test tube is shacked and waits for several minute to see whether precipitation occurs or not. If the precipitation occurs, above step is repeated until there is no precipitation. ii. This procedure also applied to sodium carbonate and is kept in oven at 70°C.

3.3 Emulsion Retention Test

<table>
<tr><td>Experiment Title</td><td colspan="2">Emulsion retention test of oil-in-water emulsion with alkaline solution by measuring separation of oil layer as function of time.</td></tr>
<tr><td>Objective</td><td colspan="2">To examine emulsion retention of oil-in-water emulsion for low and high acid number crude oil and relation with dynamic interfacial tension.</td></tr>
<tr><td>Theory</td><td colspan="2">Once alkali is injected into the real reservoir, they will be left for months and even years to react and form emulsions. Therefore, in laboratory scale testing it should be left at least 3 days minimum to react and stabilize.</td></tr>
<tr><td rowspan="2">Methodology</td><td>Equipment/Apparatus</td><td>Test tube, graduated cylinder</td></tr>
<tr><td>Material/Chemical</td><td>Brine water(Sodium Chloride), crude oil, alkaline solution (Sodium Hydroxide and Sodium Carbonate),</td></tr>
<tr><td>Hazard Identification</td><td colspan="2">Alkali should be tightly closed upon usage as they can melt in room temperature and any contact with skin should be rinsed immediately.</td></tr>
<tr><td>Experiment procedure</td><td colspan="2">a. Preparation
i. The mass of sodium hydroxide needed is calculated using the following formula:
Mass = (volume x mass percentage) / (100 - mass percentage).
*For example, to make a 1 percent solution using 60 mL of distilled water, this equation used to determine the amount of sodium hydroxide to be used:
Mass = 60 x 1 / (100 - 1) = 0.6 g
ii. The calculated amount of sodium hydroxide is weighed on the scale. Distilled water of 60 mL is poured into the test tube, and adds sodium hydroxide. The solution is mixed with the spoon or gently swirl the test tube until</td></tr>
</table>

	the salt dissolves completely. iii. Then, the mass of sodium chloride is calculated using above formula for example 1% solution in 60 mL then add into test tube. iv. About 40mL crude oil (de-acidified and acidic) is measure and added into test tube to make the solution 100 mL. v. The test tube is shacked 50 times at room temperature then, it is put in oven at reservoir temperature which is 70oC and waits for several minute. vi. The emulsion is determined visually by measuring the oil separated from the emulsion at 70°C immediately and after 5 days.

3.4 IFT Measurement Test

Title	**IFT measurement test using spinning drop method for given duration period to make correlation between static IFT and retention of emulsion.**
Material	Crude oil, alkaline containing brine solution (Sodium Hydroxide and Sodium Carbonate)
Apparatus	Test tube, syringes
Machine	Spinning Drop Machine
Procedure	Static IFT between aqueous solution contain brine and alkaline with oil sample is measured at 70°C with Reactivity index for aqueous solution containing brine and alkaline solution should be determined earlier. The method used is spinning drop and run to get the static IFT.

3.5 Core Flooding

Title	**Core Flooding**
Objective	To run the alkaline flooding and see the recovery
Material	Crude oil, alkaline containing brine solution (Sodium Hydroxide and Sodium Carbonate), brine, distilled water
Apparatus	Core
Machine	Core Flooding Machine, suction pump, oven
Preparation	Core cleaning is done before the experiments are conducted. Brine is prepared by dissolving reagent grade salts in distilled water in pre-determined salinity. Connate brine of 10000pp consisting of NaCl is prepared.
Porosity Measurement using Saturating Method	Porosity is the percentage of pore volume over bulk volume of the core sample. 1. The core sample is dried in the oven. 2. Length and diameter of the core sample is measured using Vernier calipers. Bulk Volume = $\pi r^2 L$ 3. Core is weighed to get the dry weight. 4. Core is placed in a beaker with 500ml distilled water and the air is sucked using suction pump for 20 minutes. 5. The core is left to be saturated for a minimum of 6 hours. 6. After saturation, the wet weight of the core is measured instantly. Pore volume = (Wet weight – Dry weight) / Bulk volume

	7. Pore volume is calculated by : Porosity = Pore volume / Bulk volume X 100%
Oil flood to determine residual water saturation	1. The core sample is saturated with 10000ppm brine. Then the sample is again injected with more brine to make sure 100% brine saturation is achieved, also known as steady state condition. 2. Next, Dulang crude oil is injected into the brine saturated core at the rate of 0.3cc/min until no more water is recovered. (Confining pressure : 2500psi, Injection pressure : 1900psi, Temperature : 70°C). 3. Injection is continued until no more water is produced. This indicates irreducible water saturation. 4. The volume of oil produced, volume of brine produced and pressure drop is recorded as a function of time. Material balance will be used to calculated connate water saturation (S_{wc}).
Secondary brine flood to determine residual oil saturation	Currently the core is at connate water saturation and initial oil saturation. 1. The core will be flooded with 10000ppm brine after the oil flood at an injection rate of 0.3-0.5cc. (Confining pressure : 2500psi, Injection pressure : 1900psi, Temperature : 70°C) 2. The volumes of oil and brine produced and pressure drop is measured as a function of time. 3. Flooding is done until no more oil is produced. 4. Residual oil saturation is determined through material balance.

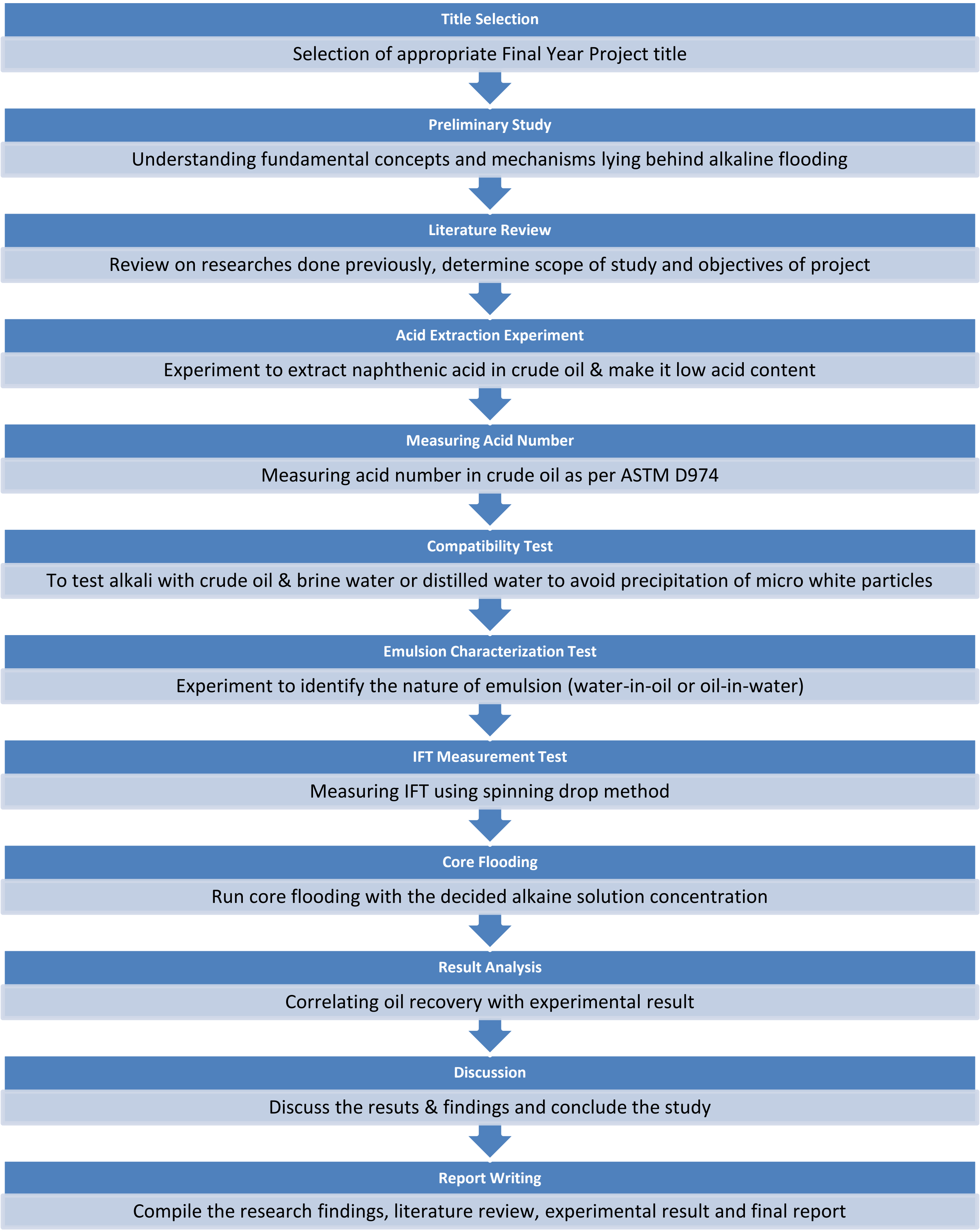
Tertiary recovery using alkaline flooding	1. Currently the core is left with residual oil saturation. 2. Injection rate is set to be 0.3-0.5cc and injection pressure at 1900psi. 3. Alkaline flooding is done by injection of alkaline solution of determined concentration. 4. Inlet and outlet pressure is recorded along with produced water and oil. Collected oil and water will be used to measure the recovery factor.

Cores will be saturated previously with 10000ppm brine before the core flooding process begins.

Two runs of core flooding will be done with the following combinations:

a. Low acid number crude oil with best selected alkaline solution
b. High acid number crude oil with best selected alkaline solution

3.7 Project Activities



3.8 Gantt chart

Table 2 Gantt chart for FYP 1

No.	Activities / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic	■	■												
2	Preliminary Research Work			■											
3	Literature Review				■	■	■								
4	Proposal Submission							★							
5	Experimental Procedure Preparation							■	■						
6	Proposal Defense								★						
7	Provision of Chemical and Crude for Experiment								■	■					
8	Acid Number Measurement								■	■	■	■	■		
9	Compatibility Test											■	■	■	
10	Emulsion Retention Test											■	■	■	■
11	Initial Results											★	■	■	■
12	Draft of Interim Report											■	■		
13	Interim report Submission												■	★	

* ★ represents key milestones

Table 3 Gantt chart for FYP 2

No.	Activities / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	IFT Measurement Test	▨	▨	▨											
2	Core Flooding			▨	★										
3	Results & Discussion				▨	▨	▨								
4	Progress Report							★							
5	Draft Report								▨						
6	Dissertation (Soft Copy)									▨					
7	Technical Paper							▨	▨	▨	▨	★			
8	Oral Presentation											▨	★		
9	Project Dissertation (Hard bound)													▨	★

3.9 Key Milestones

3.9.1 FYP 1

1. Submission of extended proposal
2. Proposal defense
3. Complete literature review
4. Complete methodology
5. Completion of phase behavior experiments

3.9.2 FYP 2

1. Core flooding experiment
2. Progress report submission
3. Technical paper submission
4. Oral presentation
5. Dissertation submission

CHAPTER 4

4.0 Results and Discussion

4.1 Acid Number Determination

The result of this experiment is divided into two which is low acid number crude oil(Dulang) and high acid number crude oil(Dulang). The results of titration and pH value is shown in table below.

Dulang crude oil according to previous studies has been found to be naturally low acid number crude oil (around 1.0mg KOH/mg oil). Therefore, the original Dulang crude oil in this experiment is taken as low acid number crude oil. To make acidic crude oil, the Dulang crude oil is artificially added with acetic acid to give it a high acid number. 0.04ml of acetic acid is added per 10grams of crude oil. Figure 1 shows the titration for acid number determination in low acid number crude oil while Figure 2 shows the titration for acid number determination in high acid number crude oil. Table 4 shows the acid number measurement results of this experiment.

The formula to calculate acid number is based on ASTM D974 and ASTM D3339 book 2005 shown below :

Acid number (mgKOH/g) = 56.10 M (A-B) / W

A : Titration Volume (ml) KOH solution required for titration of sample

B : Blank level (0.1ml) KOH solution required for titration of the blank

M : Molarity (0.1)

W : Sample used (g)

Table 4 Results from acid number measurement using ASTM D974

Crude Oil	**Crude oil weight (g)**	**Volume of alcoholic titration KOH (ml)**	**pH value before titration**	**pH value after titration**	**Acid number mg KOH/mg oil**
Low Acid	10	2.0	5.8	7.3	1.122
High Acid	10	8.7	2.9	7.2	4.881

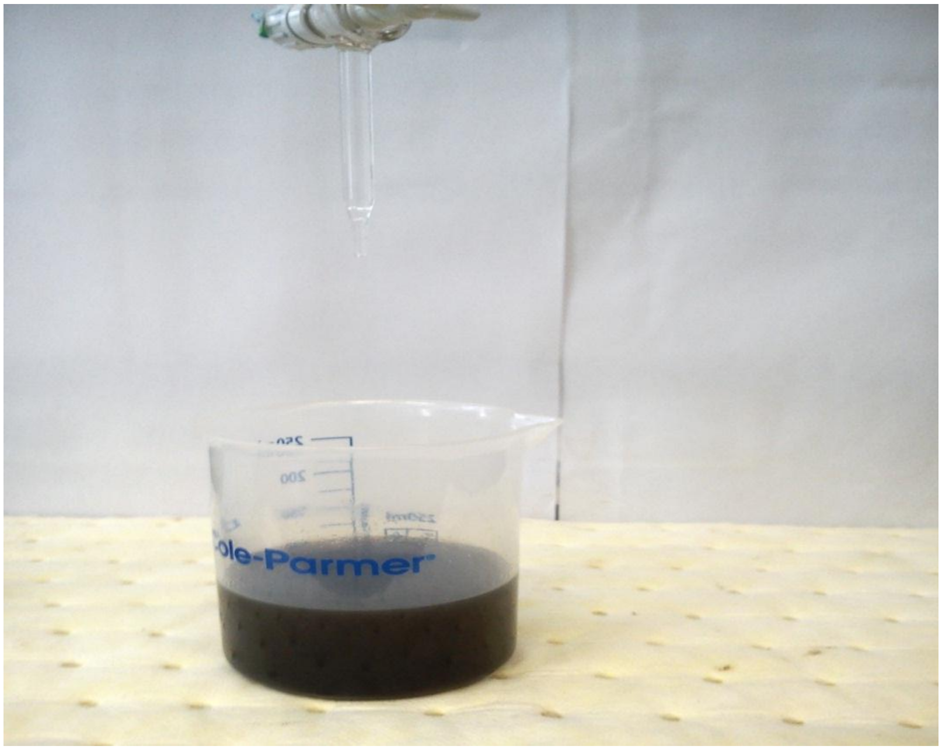


Figure 1 Titration for acid number determination in low acid number crude oil

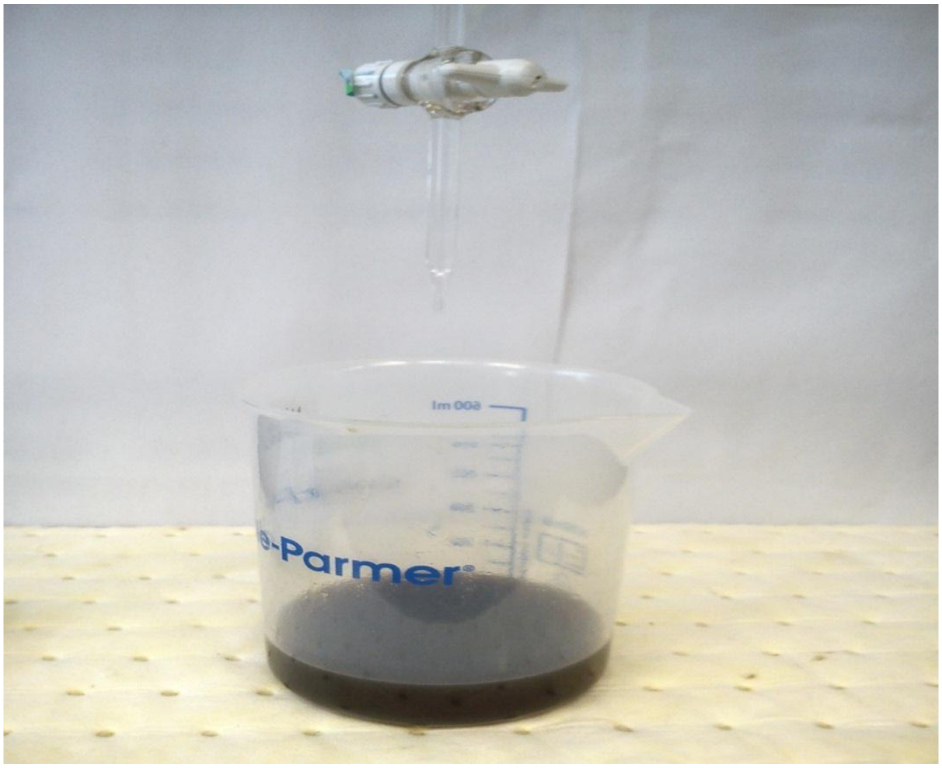


Figure 2 Titration for acid number determination in high acid number crude oil

4.2 Compatibility Test

Compatibility test was done for all 18 samples with high acid number crude oil and low acid number crude oil. Alkaline solution and brine was mixed with crude oil in the ratio of 3:2 in a 10ml measuring cylinder, mixed well and kept in oven at the temperature of 70°C for 5 days. Results are categorized according to alkali type and provided in Table 11, 12, 13 and 14.

The salinity and concentration range was selected based on studies done on Dulang field previously. 3 salinities, 1wt%, 1.5wt% and 2wt% of NaCl were chosen and 3 concentrations, 0.5wt%, 1wt% and 1.5wt% of alkali were chosen. For each salinity and concentration, the volume of water (Vw) and oil (Vo) the microemulsion contained was first measured and normalized to total alkaline volume (Vs) to obtain oil and water solubilization ratio (Vw/Vs and Vo/Vs, respectively). Then, these solubilization ratios were plotted for each salinity, and subsequently fitted with curves to form solubilization curves. The intersection of oil and water solubilization ratio curves is defined as the optimal solubilization ratio and optimal salinity. The plottings of solubilization ratio curves are shown in Figure 7 to Figure 18.

Table 11 NaOH and brine with low acid number crude oil

NaCl (wt%)	NaOH(wt%)	Oil(ml)	Emulsion(ml)	Water(ml)	Vo/Vs	Vw/Vs
1	0.5	3.8	4.2	1.8	0.633333	0.3
1	1	4	5	1	0.666667	0.166667
1	1.5	3.8	4.4	1.8	0.633333	0.3
1.5	0.5	3.7	4.4	1.6	0.616667	0.266667
1.5	1	4	4.6	1.5	0.666667	0.25
1.5	1.5	3.6	4.6	1.4	0.6	0.233333
2	0.5	3.6	4.7	1.3	0.6	0.216667
2	1	3.6	5	1	0.6	0.166667
2	1.5	3.7	4.4	1.7	0.616667	0.283333

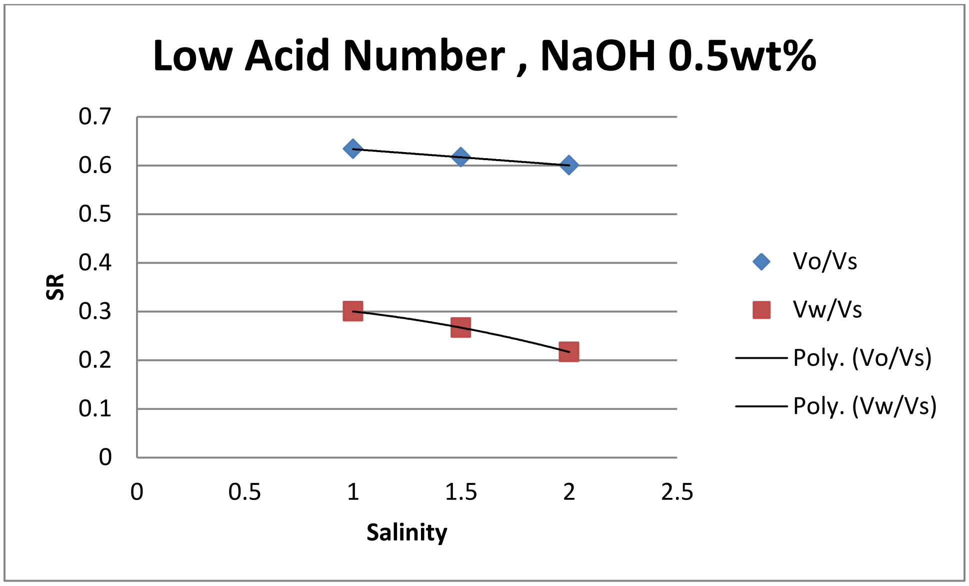


Figure 7 0.5wt%NaOH solubilization ratio with low acid number crude oil

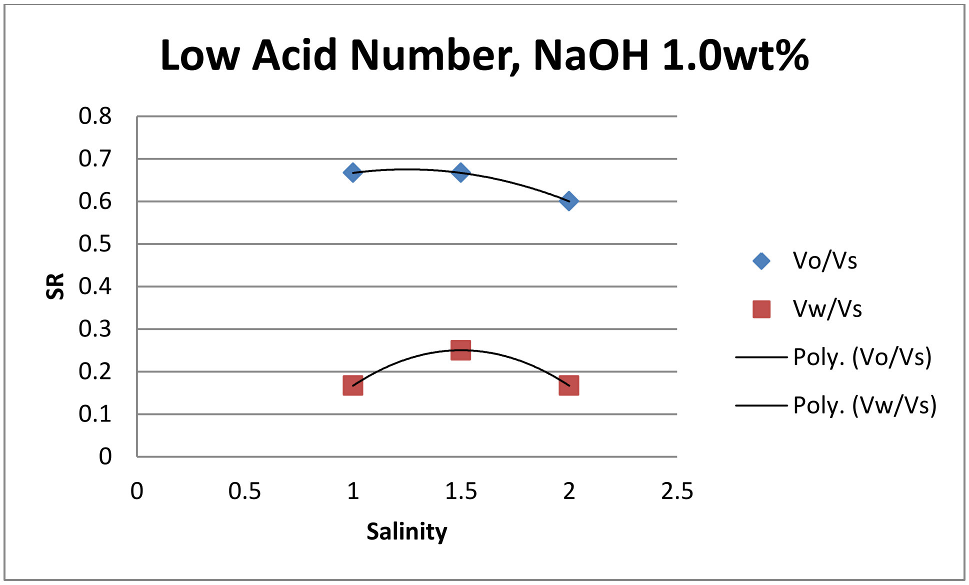


Figure 8 1wt%NaOH solubilization ratio with low acid number crude oil

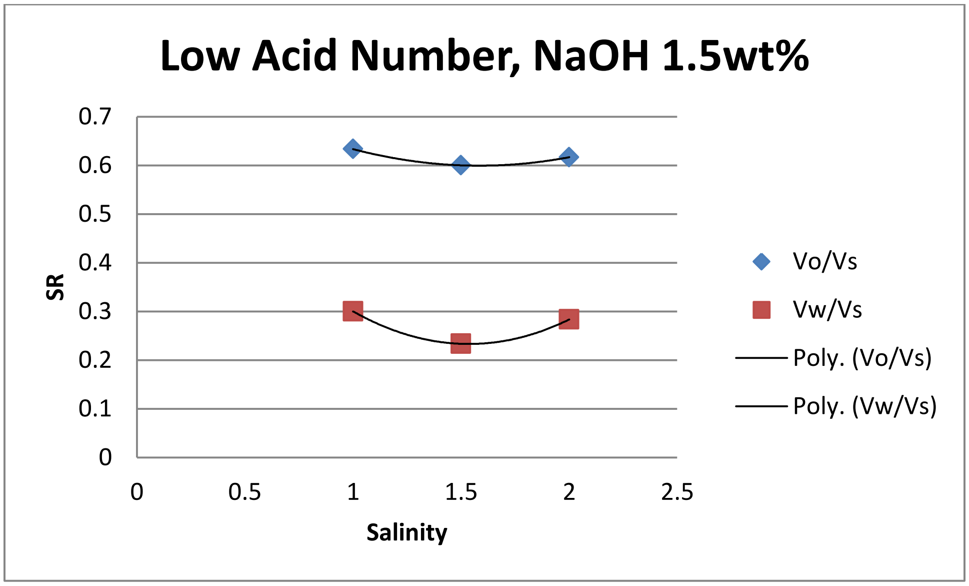


Figure 9 1.5wt%NaOH solubilization ratio with low acid number crude oil

Table 12 NaOH and brine with high acid number crude oil

NaCl (wt%)	NaOH	Oil	Emulsion	Water	Vo/Vs	Vw/Vs
1	0.5	3.2	5	1.3	0.533333	0.216667
1	1	3.5	5.2	1.3	0.583333	0.216667
1	1.5	3.2	5.2	1.3	0.533333	0.216667
1.5	0.5	3.5	4.8	1.4	0.583333	0.233333
1.5	1	4.2	4.9	1.3	0.7	0.216667
1.5	1.5	3.5	4.8	1.7	0.583333	0.283333
2	0.5	3.3	5.4	1.1	0.55	0.183333
2	1	3.6	5.7	0.6	0.6	0.1
2	1.5	3.6	5.4	1	0.6	0.166667

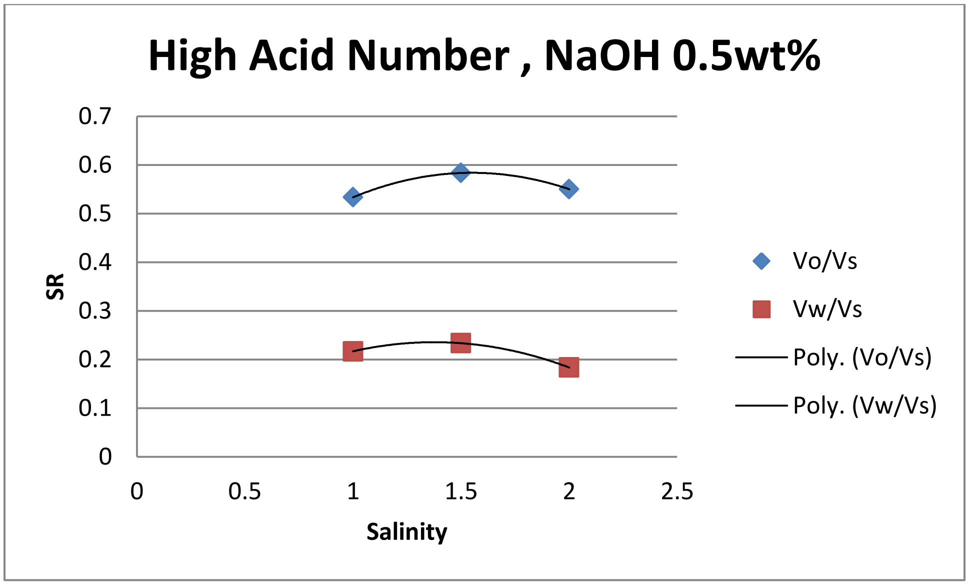


Figure 10 0.5wt% NaOH Solubilization Ratio with high acid number crude oil

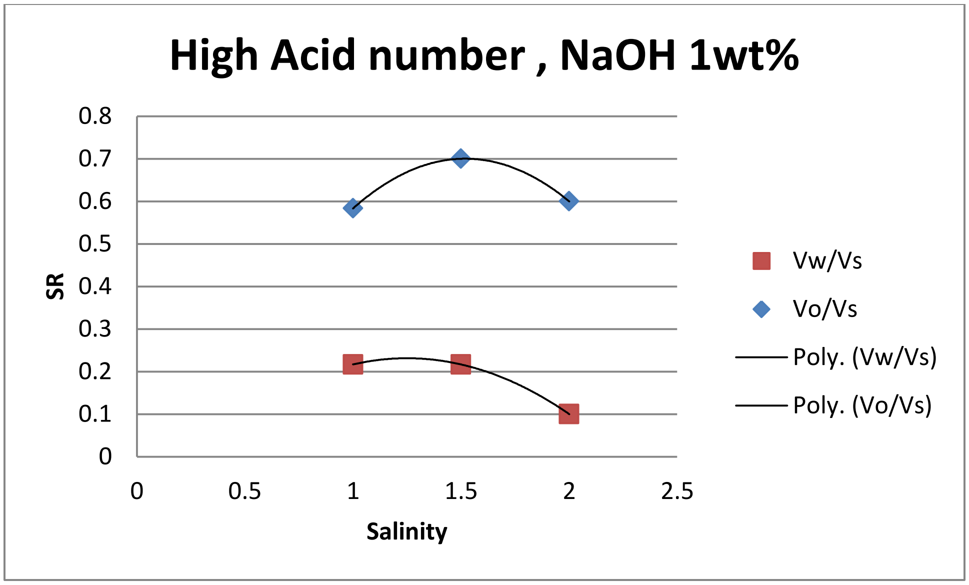


Figure 11 1wt% NaOH Solubilization Ratio with high acid number crude oil

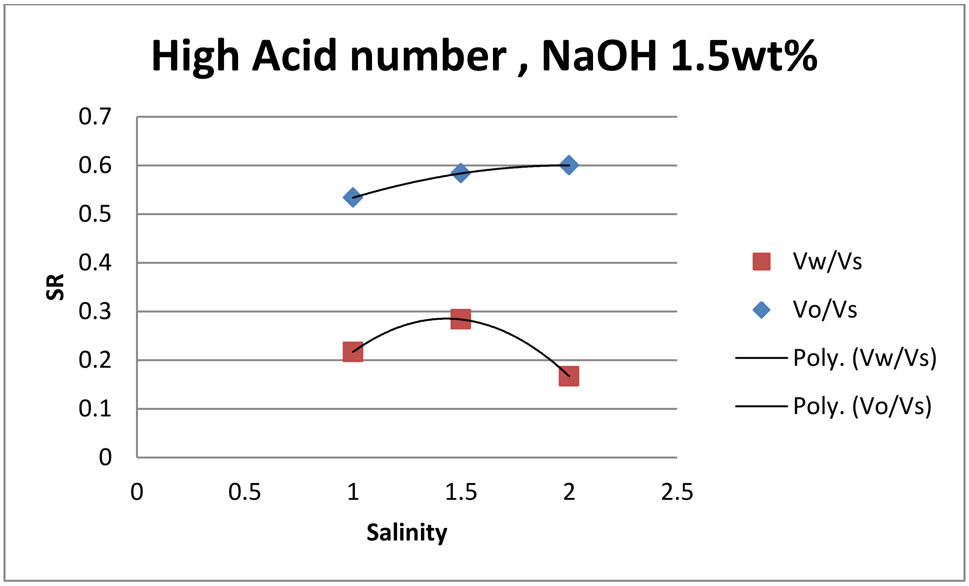


Figure 12 1.5wt% NaOH Solubilization Ratio with high acid number crude

Table 13 Na_2CO_3 and brine with low acid number crude oil

NaCl	Na_2CO_3	Oil	Emulsion	Water	Vo/Vs	Vw/Vs
1	0.5	4	1.4	4.6	0.666667	0.766667
1	1	3.7	4.1	1.8	0.616667	0.3
1	1.5	3.6	3.2	3	0.6	0.5
1.5	0.5	3.5	2.6	3.5	0.583333	0.583333
1.5	1	3.5	2	4.2	0.583333	0.7
1.5	1.5	3.8	1.9	4.2	0.633333	0.7
2	0.5	3.9	1.1	5.2	0.65	0.866667
2	1	3.8	1.5	4.7	0.633333	0.783333
2	1.5	3.7	3.7	2.5	0.616667	0.416667

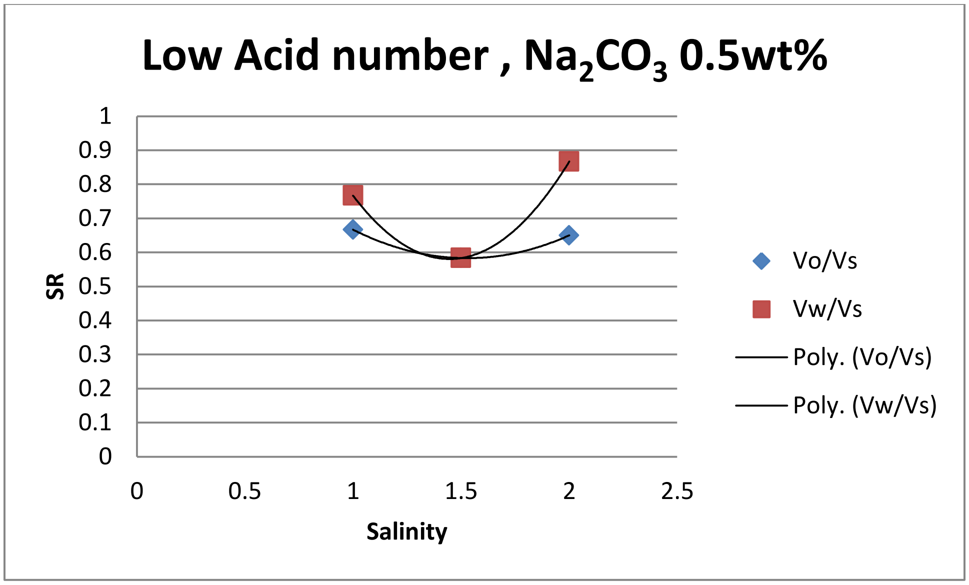


Figure 13 0.5wt% Na_2CO_3 Solubilization Ratio with low acid number crude oil

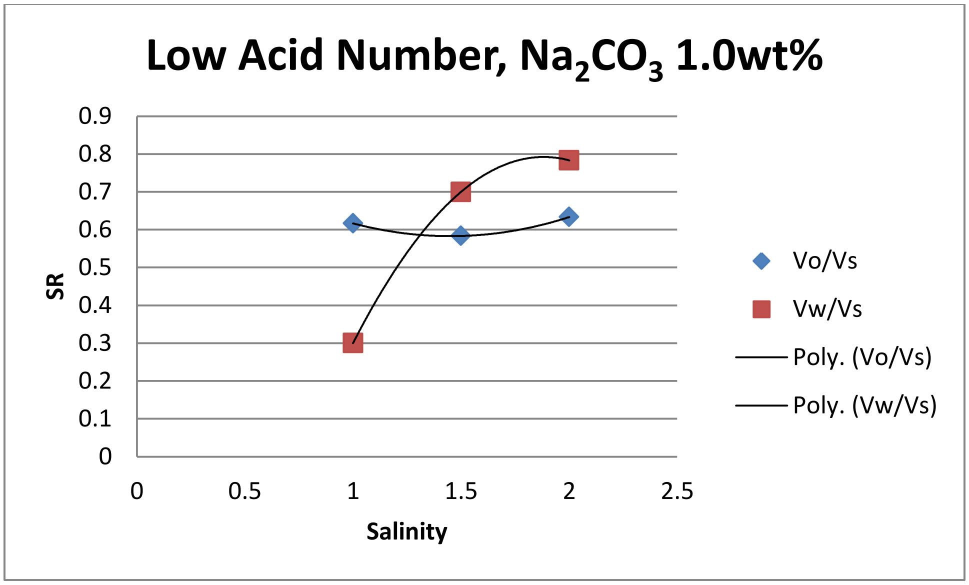


Figure 14 1wt% Na_2CO_3 Solubilization Ratio with low acid number crude oil

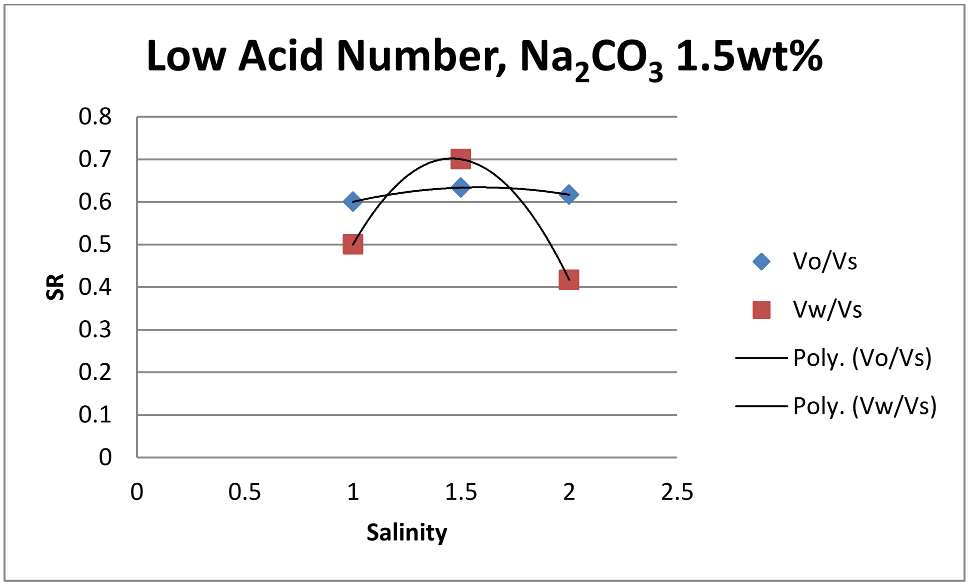


Figure 15 1.5wt% Na_2CO_3 Solubilization Ratio with low acid number crude oil

Table 14 Na_2CO_3 and brine with high acid number crude oil

NaCl	Na_2CO_3	Oil	Emulsion	Water	Vo/Vs	Vw/Vs
1	0.5	4.1	1	5	0.683333	0.833333
1	1	3.9	1.1	5.1	0.65	0.85
1	1.5	4	2	4	0.666667	0.666667
1.5	0.5	3.8	4.7	1.8	0.633333	0.3
1.5	1	3.8	1.3	5	0.633333	0.833333
1.5	1.5	3.9	2.4	4	0.65	0.666667
2	0.5	4	3.4	2.6	0.666667	0.433333
2	1	3.9	2.4	3.7	0.65	0.616667
2	1.5	3.7	4.3	2	0.616667	0.333333

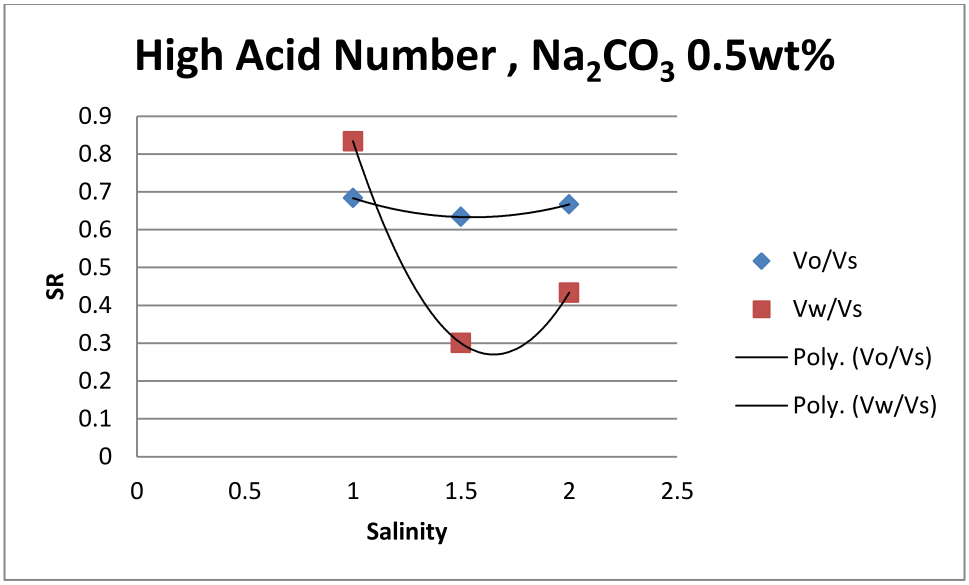


Figure 16 0.5wt% Na_2CO_3 Solubilization Ratio with high acid number crude oil

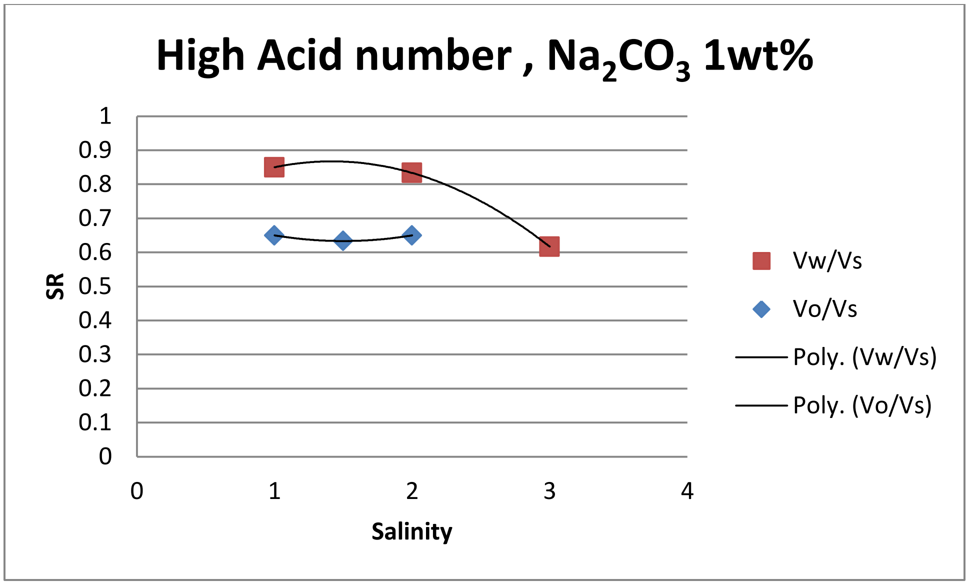


Figure 17 1wt% Na_2CO_3 Solubilization Ratio with high acid number crude oil

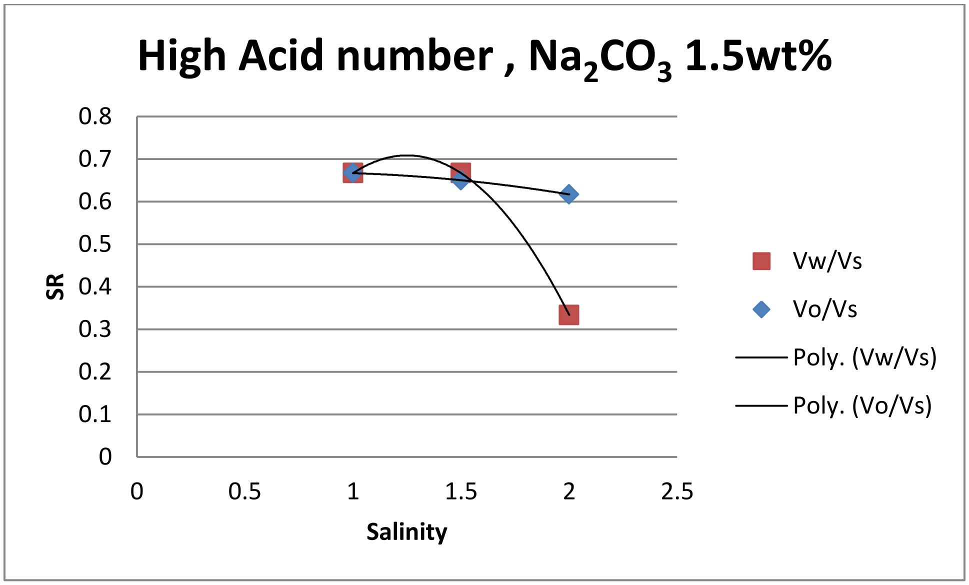


Figure 18 1.5wt% Na_2CO_3 Solubilization Ratio with high acid number crude oil

The compatibility test for altogether 36 samples were done in 2 weeks and results recorded. The length of emulsion formed shows the reaction between oil, alkaline and brine solution. High emulsion length is preferred because more emulsion can carry more oil to the surface. The emulsion is preferred to be stable because it should not break until it reaches the surface. No precipitation formed in this compatibility test because no co-surfactants are added.

In Table 11, the highest emulsion length formed between Sodium hydroxide, brine and low acid number crude oil was 5ml in 1wt% NaCl and 1wt% NaOH, and 2wt%NaCl and 1wt%NaOH. The compatibility test between Sodium Hydroxide, brine and high acid number crude oil gives emulsion as high as 5.7ml at 2wt%NaCl and 1wt%NaOH.Table 13 shows the microemulsion formed between low acid number crude oil, Sodium Carbonate and brine. 1wt%NaCl and 1wt%Na_2CO_3 gives the highest microemulsion length of 4.1ml. 4.7ml of microemulsion was formed in 1.5wt%NaCl and 0.5wt% Na_2CO_3 with high acid number crude oil.

Solubilization ratio curves show intersection in Na_2CO_3 but no intersection is found in NaOH which shows Na_2CO_3 is a better alkaline to be used for both high acid and low acid Dulang crude oil. Lack of salinity, extra salinity, inadequate or additional concentration might be the reasons why the curves do not intersect. Multiple intersections are also found in high acid and low acid number crude oil with 1.5wt% Na_2CO_3. Both the values gives optimum salinity twice but these values should be further tested with the salinity added with hard ions that are usually found in brine water which usually cause chemical precipitation in reservoir (Mayer, et al., 1983; Yang, et al., 2010).

4.3 Emulsion Retention Test

Figures 19, 20, 21 and 22 in Appendix show the results of percentage of oil separated from water immediately after shaking and after five days keeping in oven at 70°C. Immediately after shaking, the emulsions formed a mixture as shown in the pictures where the aqueous phase at the bottom and mixture on top. After 5 days kept in the oven to stabilize, the microemulsion separated into 3 distinct layers, oil, water and emulsion, forming emulsion Winsor Type III. None of the microemulsion breaks before the 5th day. All the microemulsion samples were stable throughout the observation and therefore they are suitable to be used for alkaline flooding in low acid number crude oil and high acid number crude oil reservoirs.

For a simple aqueous system, surfactant type and structure will determine the formation of microemulsion. In this test, the microemulsion formed spontaneously after shaking, proving the formation of in-situ surfactant formation when mixing alkaline and crude oil. The immediate formation of microemulsion as soon as alkaline and alkaline is mixed is a new finding compared to Yang et.al.'s study which states that microemulsions will take few days to form. This condition should be further tested with shorter intervals which will show the exact time it takes for the mixture to form emulsion.

4.4 Spinning Drop

The addition of alkaline reduces the interfacial tension between oil and water, and this reduction is a parameter of interest in alkaline flooding as it helps in mobilizing residual oil in the reservoir.

A total of 18 samples, with three different salinity and three different concentrations were prepared to be used for spinning drop test. Table 15 shows the prepared samples list for spinning drop test.

Table 15 Samples for spinning drop test

Brine (%)	**NaOH (%)**	**Na_2CO_3(%)**
1.0	**0.5**	**0.5**
	1.0	**1.0**
	1.5	**1.5**
1.5	**0.5**	**0.5**
	1.0	**1.0**
	1.5	**1.5**
2.0	**0.5**	**0.5**
	1.0	**1.0**
	1.5	**1.5**

The IFT measurement was done using Spinning Drop machine. Dulang crude oil was used and the densities are listed below:

a) Low acid number crude oil : 0.8002g/cm^3
b) High acid number crude oil : 0.8000g/cm^3

Results for the spinning drop test are provided in Table 16, 17, 18 and 19 in Appendix and represented in graph in Figure 23 to 26.

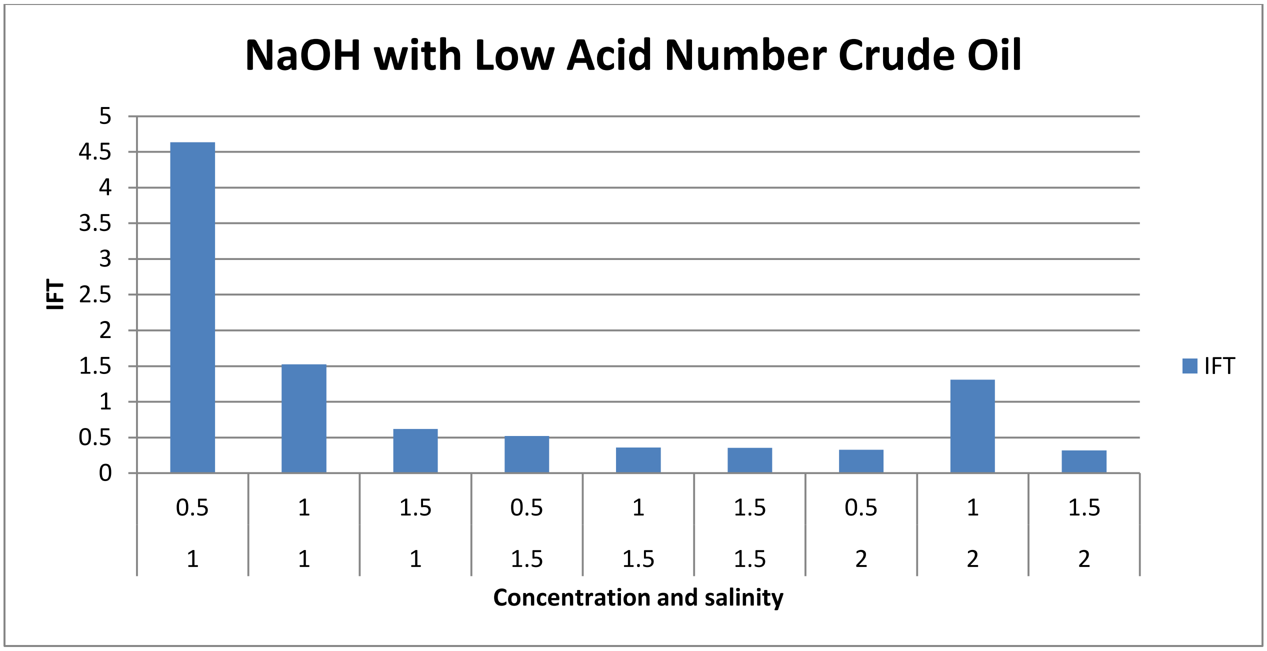


Figure 23 NaOH with Low Acid Number Crude Oil

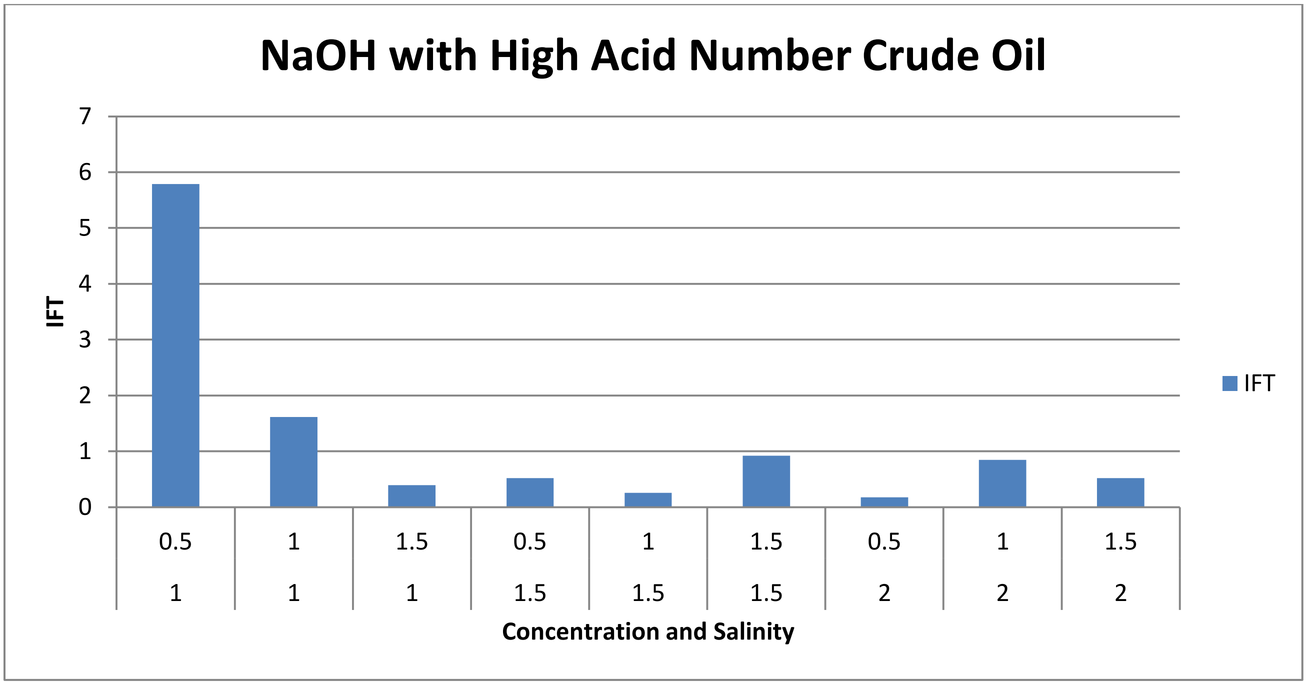


Figure 24 NaOH with High Acid Number Crude Oil

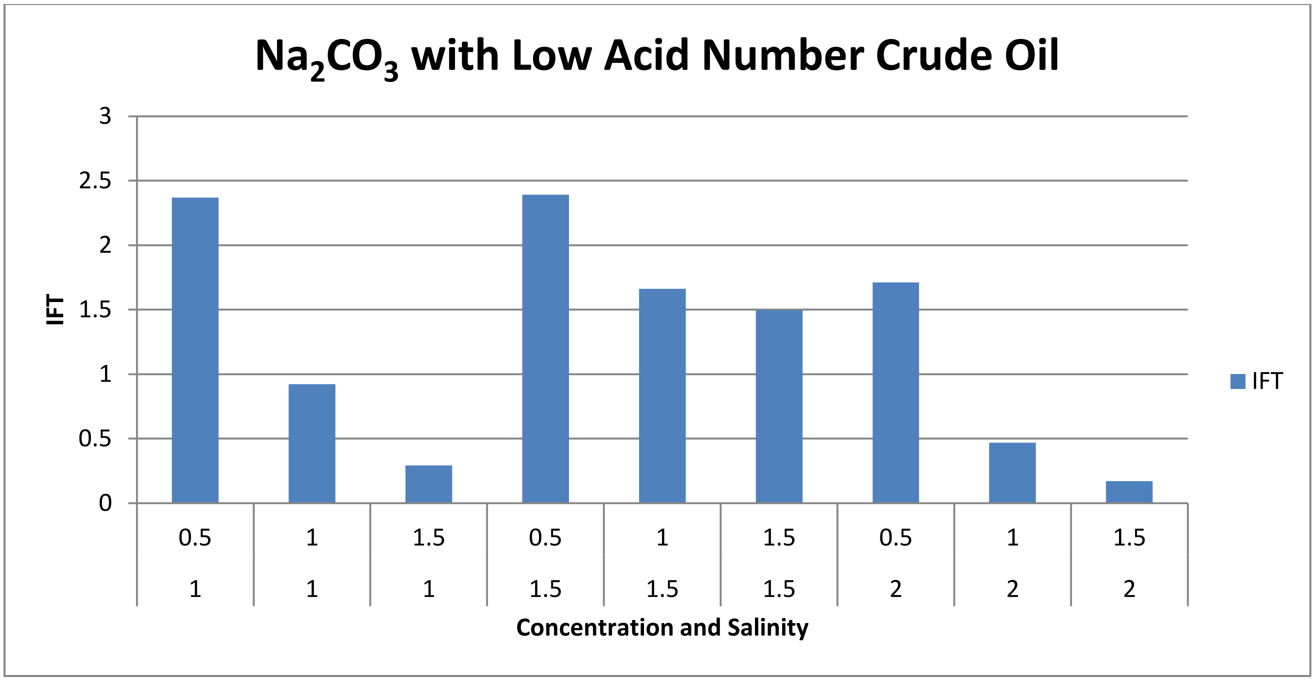


Figure 25 Na_2CO_3 with Low Acid Number Crude Oil

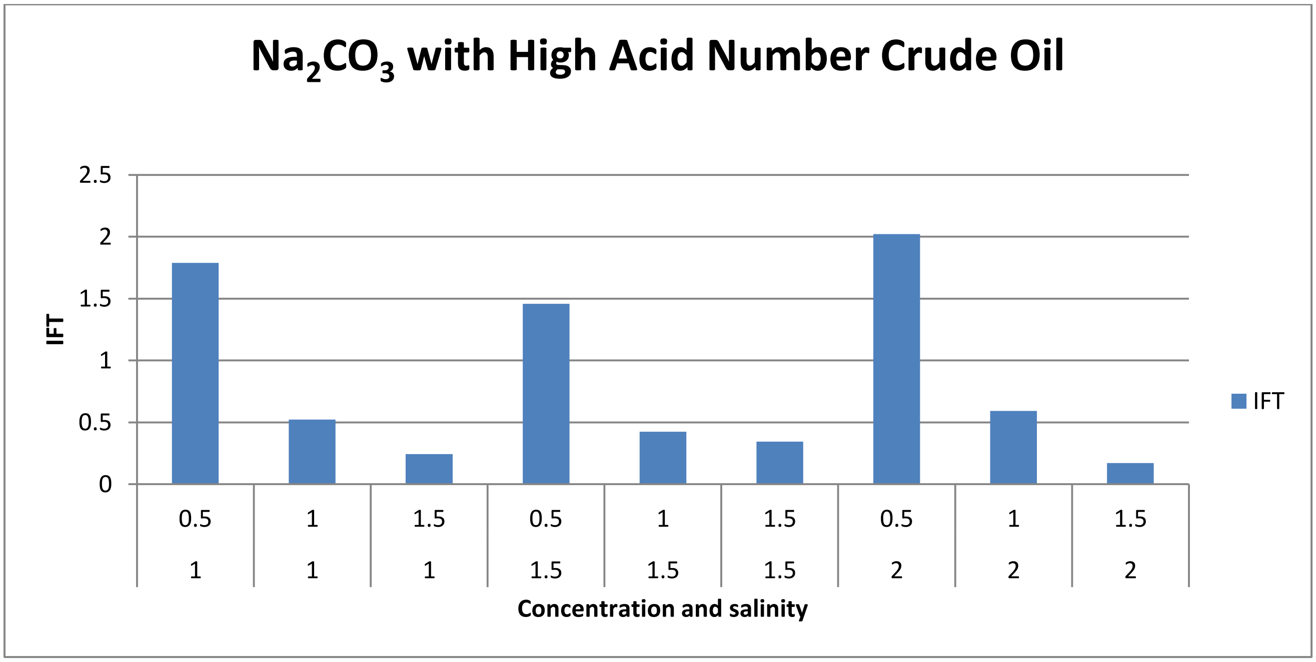


Figure 26 Na_2CO_3 with High Acid Number Crude Oil

Figure 23 shows the bar chart representation of interfacial tension (IFT) results between alkaline solutions with brine and different crude oils. The IFT between low acid number crude oil and Sodium Hydroxide solution with brine has the lowest reading at 2wt%NaCl and 1.5wt%NaOH. Solutions with concentrations of 1.5wt%NaCl and 1wt%NaOH, 1.5wt%NaCl and 1.5wt%NaOH, and 2wt%NaCl and 0.5wt% NaOH gives low IFT readings which falls between 0.2mN/m and 0.5mN/m.

The IFT between high acid number crude oil and Sodium Hydroxide with brine observed at Figure 24 the lowest reading at 2wt%NaCl and 0.5wt%NaOH. Low readings are observed at the concentrations of 1wt%NaCl and 1.5wt%NaOH, and 1.5wt%NaCl and 1wt%NaOH.

Figure 25 shows the IFT between Na_2CO_3 with brine and low acid number crude oil. Low IFT readings are observed at 1wt%NaCl and 1.5wt% Na_2CO_3, and 2wt%NaCl and 1wt% Na_2CO_3. 2.0wt% salinity and 1.5wt% concentration of Na_2CO_3 gives the lowest IFT reading which is 0.1722.

Finally, in Figure 26, the lowest IFT could be observed between Na_2CO_3 with brine and high acid number crude oil when salinity is 2wt%NaCl and 1.5wt% Na_2CO_3. At 1wt%NaCl and 1.5wt% Na_2CO_3, 1.5wt%NaCl and 1wt% Na_2CO_3, and 1.5wt%NaCl and 1.5wt% Na_2CO_3, IFT readings are low, 0.2432, 0.4239 and 0.3435 respectively.

From the graphs, the lowest IFT is reached with Na_2CO_3 for both crude oils at 2wt%NaCl and 1.5wt% Na_2CO_3.

IFT is calculated based on the following formula:

$$\boxed{\gamma = \frac{1}{4} r^3 \Delta\rho\omega^3}$$

γ= IFT

r= radius of drop (oil droplet)

ρ= density of fluid

ω= rotational velocities

Before running the spinning drop test, refractive index (RI) and density of the fluids at 70°C are measured earlier. Spinning drop test is done by injecting a denser fluid (alkaline solution in this case) into the capillary tube and set to rotate. Following that, a drop of crude oil will be injected into the spinning capillary tube. At low rotational velocity, the oil drop will take ellipsoidal shape, but when the velocity is large enough, it will become cylindrical. The radius of the oil droplet is a function of IFT, $\Delta\rho$ and rotational velocities. 70°C is used as reservoir temperature to control the velocity. Shape of the oil droplet is set as the constant variable for all the samples for accurate results.

The mechanism behind spinning drop test is emulsification. When a drop of crude oil in injected into the capillary tube, it undergoes emulsification with the alkali and the machine read the interfacial tension between these two fluids based on parameters input. The mechanism is visualized in Figure 27.

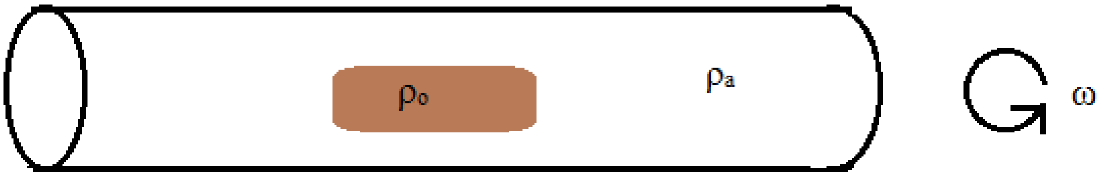


Figure 27 Schematic of spinning drop method

4.5 IFT-Chun Huh Equation

Apart from spinning drop method, IFT can also be calculated by Chun Huh equation. A simplified form of his IFT theory shows that IFT is inversely proportional to the square of the solubilization ratio.

$$\boxed{\gamma = \frac{C}{\sigma^2}}$$

γ = IFT
σ = solubilization ratio

C is 0.3dynes/cm for most of the crude oils and microemulsions, and it makes a good approximation too. Therefore, Chun Huh method is a quick way to calculate interfacial tension and can be used to compare with the measured IFT. Results of IFT using Chun Huh method and spinning drop method and the error calculation is shown in Table 21 to 24 in Appendix.

The results of IFT measured with Chun Huh equation are highly contradicting with the measured IFT results. The error measurement reaches as high as 88.8%. This may be caused by the human error because the solubilization ratio which is used in this calculation is using emulsion length which needs an accurate measurement. As the experiment is conducted which naked eyes without any special measuring devices, human error can be a major contributor of the faulty results. Therefore, IFT measurement calculated by spinning drop method is more reliable in this experiment.

Based on the results, 2.0wt% salinity and 1.5wt% concentration of Na_2CO_3 is selected as the optimum concentration for both low acid number crude oil and high acid number crude oil reservoir to run core flooding.

4.6 Porosity of Core- Core saturating

Two Berea sandstone cores were used in this core flooding and the property data used to characterize them are shown in Table 25. The property data include: core diameter, core length, dry weight, wet weight, bulk volume, pore volume, porosity and area. The crude oil used in this experiment is produced from Dulang field, which is a sandstone reservoir, therefore it is suitable to be represented by Berea sandstone cores throughout the experiment.

Porosity of the core is calculated by saturation method. Dry weight of core is measured before saturating it in 500ml distilled water. It is then kept in dessicator attached to a vacuum pump and left for saturation overnight. Once the core is fully saturated, the wet weight of core is measured and used to calculate the pore volume and porosity.

Table 25 Property data of cores

Core	A1	A2
Diameter(cm)	3.692	3.690
Length(cm)	6.878	7.266
Dry weight(g)	161.0356	168.9223
Wet weight(g)	182.172	173.728
Bulk Volume (cc)	73.63	77.70
Pore volume(cc)	21.14	4.8057
Porosity(%)	28.71	6.18
Area	10.71	10.69

4.7 Core Flooding

The results are presented in graphs, tables and pictures to show the comparison between the two runs :

a) Run 1 : low acid number crude oil with Na_2CO_3
b) Run 2 : high acid number crude oil with Na_2CO_3

Figure 28 and 29 show the tertiary recovery for both runs.

From the tertiary recovery, the results obtained are not consistent. For example, as we can see in Figure 28, post flush 1 and 3 have very less oil compared to post flush 2. This could possibly be because the oil is taking different path in the core plug where there is least oil. Therefore, there is no formation of in-situ surfactants and no residual oil recovery. In Figure 29, there is no oil in post flush 2 and 3, while post flush 1 has very little oil.

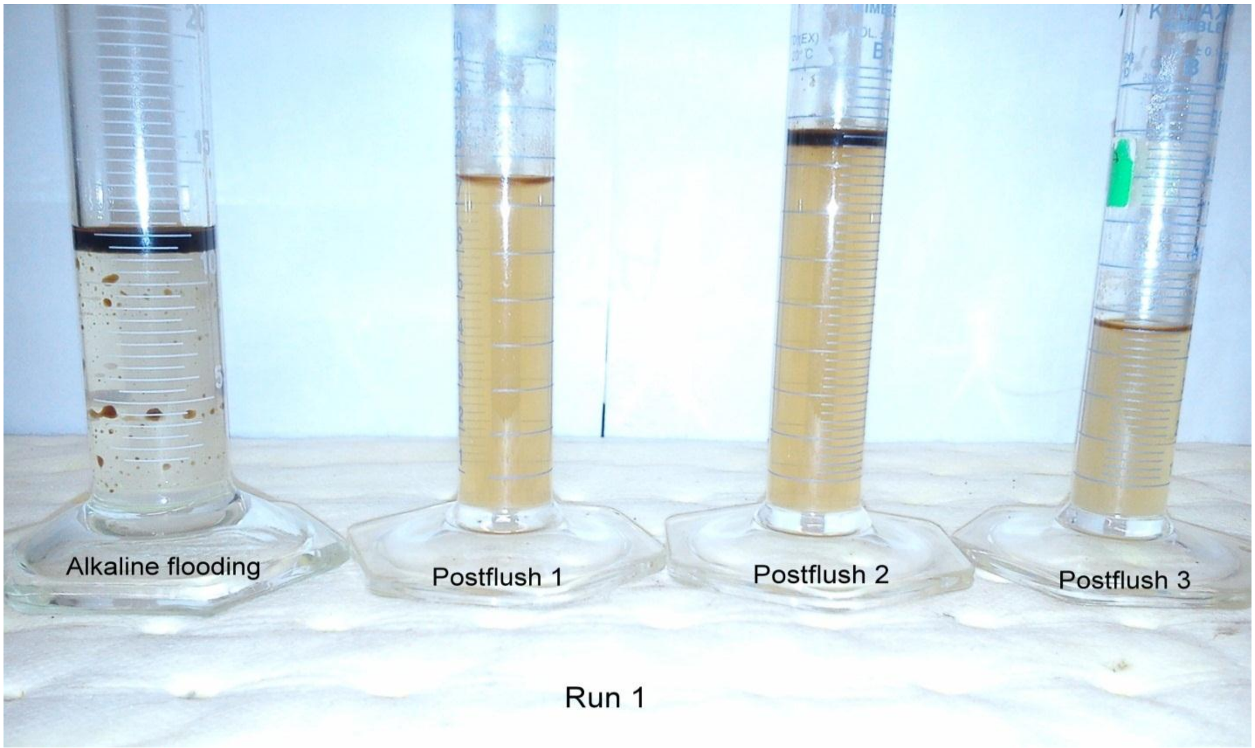


Figure 28 Tertiary recovery collected in Run 1

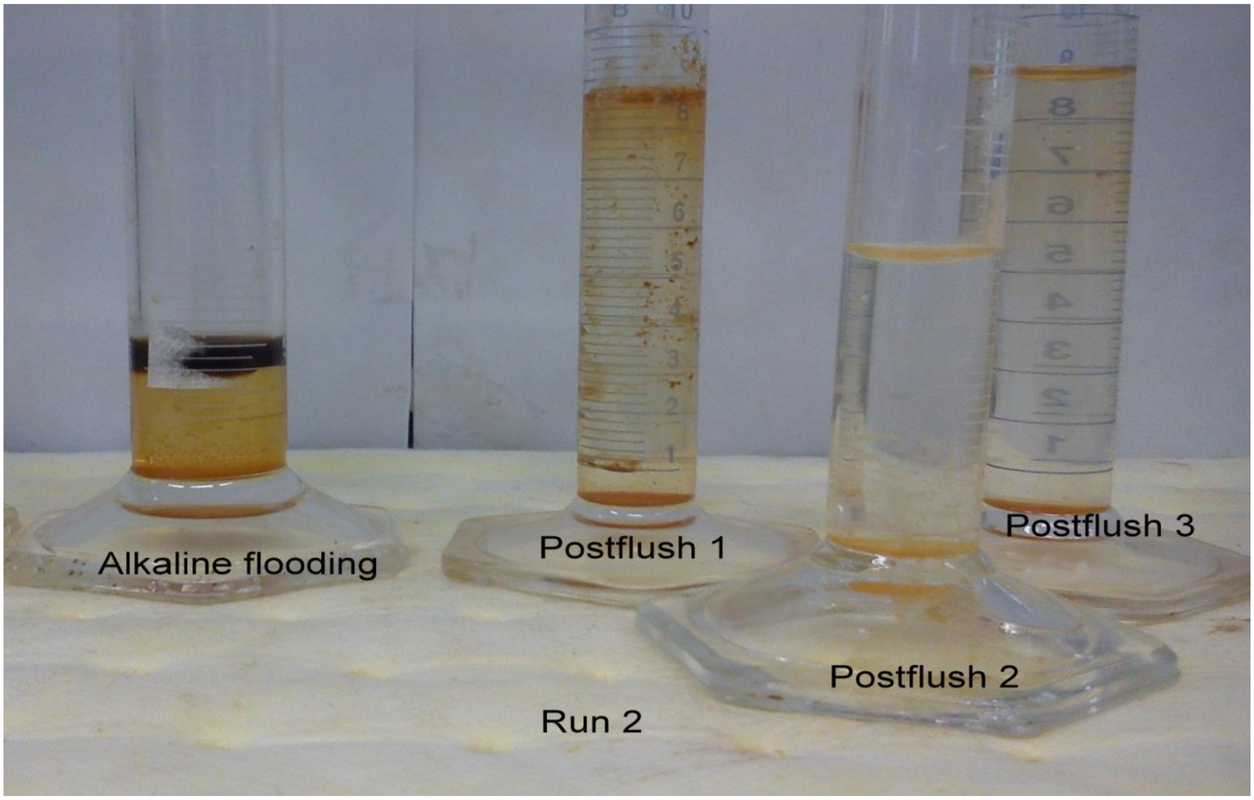


Figure 29 Tertiary recovery collected in Run 2

As shown in Table 26, the oil recovered after alkaline flooding in Run 1 is 2ml at pore volume of 0.5. Alkaline flooding in Run 2 recovered 1 ml at pore volume of 0.5. The results indicate that alkaline flooding is possible in any reservoirs regardless of acid number as there are recoveries in both the runs corresponding to Mayer. *et al*. which states that there is no proven theory of acid number affecting the recovery in oilfield.

Table 26 Volume of oil and water displaced and recovery factor for Run 1 and Run 2

Run	**1**	**2**
Original water in place (ml)	16	13
Connate water (ml)	5	5
Connate water saturation (Swc)	0.3125	0.3846
Original oil in place (ml)	11	8
Volume of oil displaced after water flooding (ml)	6	5
Volume of oil displaced after alkaline flooding (ml)	2	1
Recovery factor of water flooding (%)	54.54	62.50
Recovery factor of alkaline flooding (%)	72.72	75
Incremental recovery (%)	18.18	12.50

The tertiary recovery comparison for both runs is shown in Figure 30.

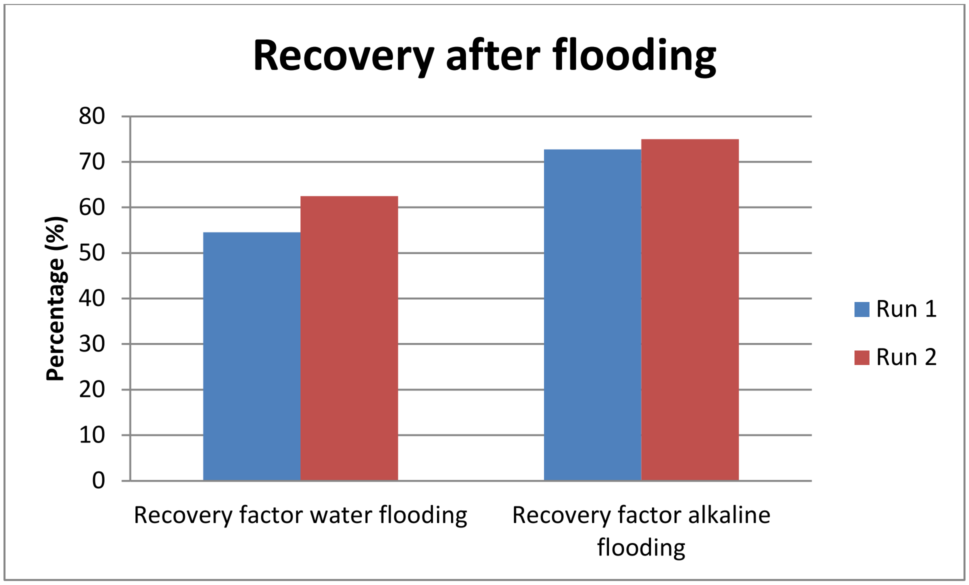


Figure 30 Recovery factor comparison for all runs

An incremental recovery of 18.18% is obtained in Run 1 which is low acid number crude oil while an increment of 12.50% is achieved in Run 2 which is high acid number crude oil. The difference is merely caused by different permeability value, porosity and size of the cores.

The result for end-point relative permeability and mobility ratio for Run 1 and 2 are shown as below :

Table 22 Result for core flooding for Run 1 and Run 2

Run	**Average end point k_{rw} (water flooding)**	**Average end point k_{rw} (alkaline flooding)**	**Average mobility ratio (water flooding)**	**Average mobility ratio (alkaline flooding)**	**Swc**	**Sor (water flooding)**	**Sor (alkaline flooding)**
1	0.3758	0.2339	1.4202	0.8839	0.3125	0.4545	0.2727
2	0.0257	0.0012	0.0971	0.0045	0.3846	0.375	0.2500

The reduction in end point relative permeability of water after alkaline flooding shows that the core plug becomes more water-wet throughout the process. This condition agrees with Mayer *et.al*.'s statement of oil-alkali interaction can help in wettability alteration in reservoir, from oil-wet to water-wet.

Reduction in mobility ratio shows that the displacement is stable, without any viscous-fingering condition. M<1 obtained in this result is a favorable mobility ratio in the reservoir. This result also shows that the formation of microemulsion inside the core might have aided in the mobilization and production of oil. Pressure difference is found to be stable for both the runs shows that the production was stable and good sweep efficiency throughout core flooding.

As a conclusion it can be said that the indirect mechanism calculated in this experiment is wettability alteration as stated by Bortolotti, Gottardi, et al. Reduction in end point relative permeability after alkaline flooding shows that the core plug became more water-wet. Further studies should be conducted on this mechanism to prove its validity by testing it on various crude oils and setting Dulang crude oil as controlled variable. They should also be further tested along IFT measurement and emulsification.

The calculation method for end point relative permeability and mobility ratio is shown in appendix together with related data and pictures.

CHAPTER 5

5.0 Conclusion & Recommendation

5.1 Conclusion

In conclusion, alkaline flooding is successful even in low acid number crude oil where the recovery of residual oil in Run 1 is 72.72% which also gives the increment of 18.18%. In comparison to low acid number crude oil, Run 2 which is high acid number crude oil also has the residual oil recovery of 75% and increment of 12.5%. Sodium Carbonate has been chosen and used as the preferable alkali for Dulang crude oil as it is found to be the most compatible and effective alkali through phase behavior experiments.

The objectives of this project are achieved because the optimum salinity and concentration of alkaline solution is identified as 2wt% NaCl, 1.5wt% Na_2CO_3 through compatibility test. The emulsion formed immediately after mixing alkaline solution and crude oil for both high and low acid number crudes and became Type 3 after 5 days of stabilisation. The emulsion formed between both crudes and alkaline are stable for 5 days without breaking and IFT between the aqueous solution and crude is obtained as low as 0.1722mN/m.

A new theory that can be driven through this project is high acid number crude oil is not a requirement for alkaline flooding, but instead the presence of a minimal amount of acid in the crude oil is sufficient for it to be successful. Reduction in relative permeability and mobility ratio indicates that wettability alteration has taken place in the core plug to be more water-wet and stable sweeping throughout the recovery process.

Lastly, the most important mechanisms that aids in alkaline flooding are identified as wettability alteration and emulsification because these two mechanisms are very obvious through core flooding. However, IFT, which was used as one of the important characterization in selecting the alkaline type and concentration, is also a key mechanism in alkaline flooding.

5.2 Recommendation

After conducting the experiments, there are some results which are found to be different from the studies and journals previously. First is the emulsification. Journal stated that it will take a few days to form microemulsion between crude oil and alkaline solution, but during this project, the mixture formed microemulsion immediately after shaking. This finding need to be further tested in terms of time to find out the exact time it takes to form the microemulsion and should be observed in shorter intervals for a long observation period. Through this, a more accurate and detailed result on microemulsion formation can be identified.

Secondly, the core flooding should be done with both Sodium Hydroxide and Sodium Carbonate solution for low and high acid number crude oils. More runs will show a good comparison of the alkaline solution on its effectiveness for both types of crude oils. Thirdly, the core flooding should be conducted on cores cut from the same type of core with same size and properties. A better result could be obtained if the original wettability of the core is restored while doing the experiment at its initial conditions. Fourthly, alkaline consumption throughout the core flooding should also be measured in order to find out the adsorption rate of alkali on the rock surfaces. This finding can prevent excessive alkaline injection into the core.

Finally, this project should be conducted with a wide range of crude oils with different properties and concentration of alkali apart from Dulang in measuring the IFT, wettability alteration, compatibility and recovery through core flooding so that the result could also be applicable to various types of crude oils.

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Appendix

Emulsion Retention Test

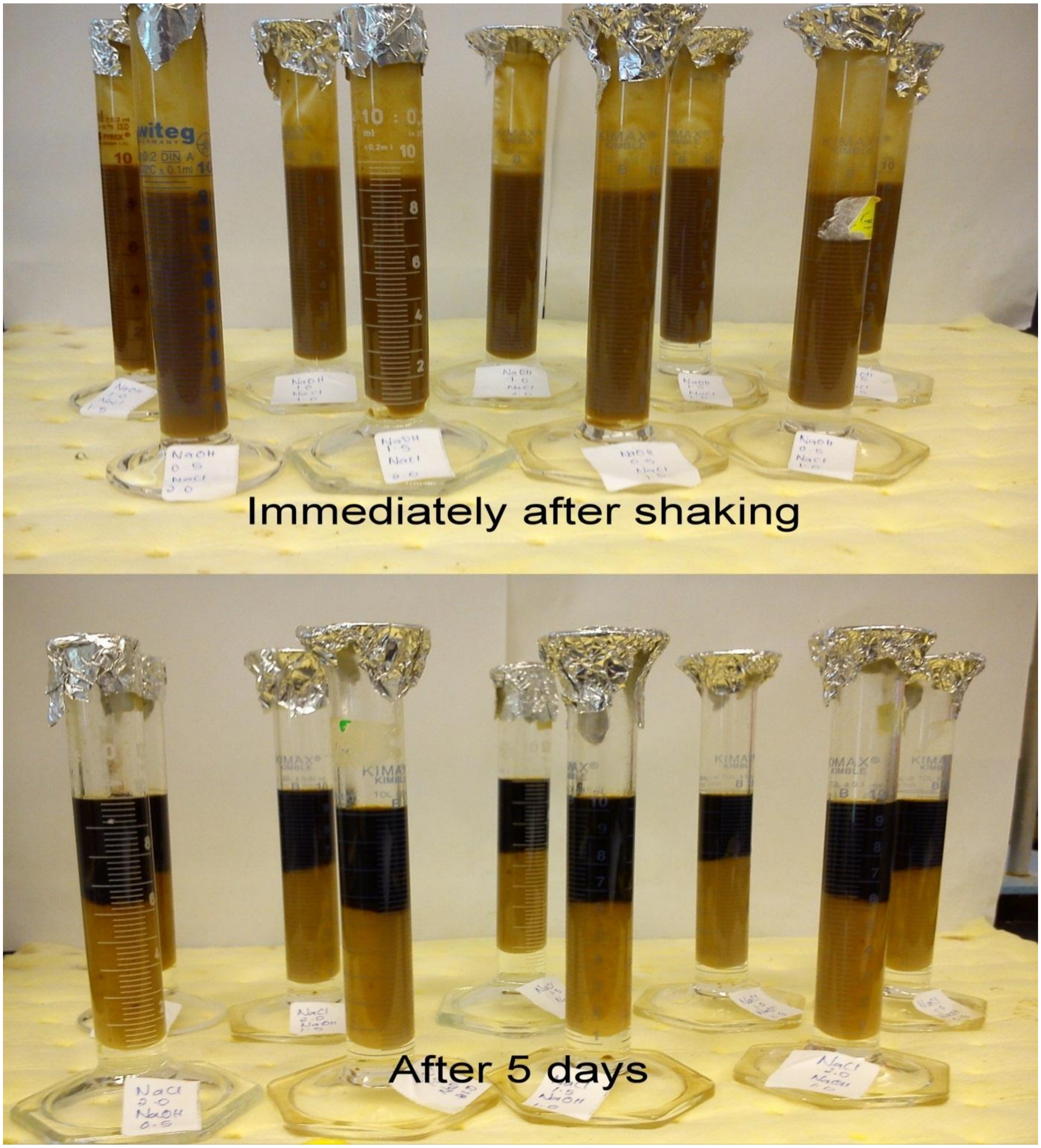

Figure 19 Observation after 5days showing the microemulsion formed between NaOH, brine and low acid number crude oil

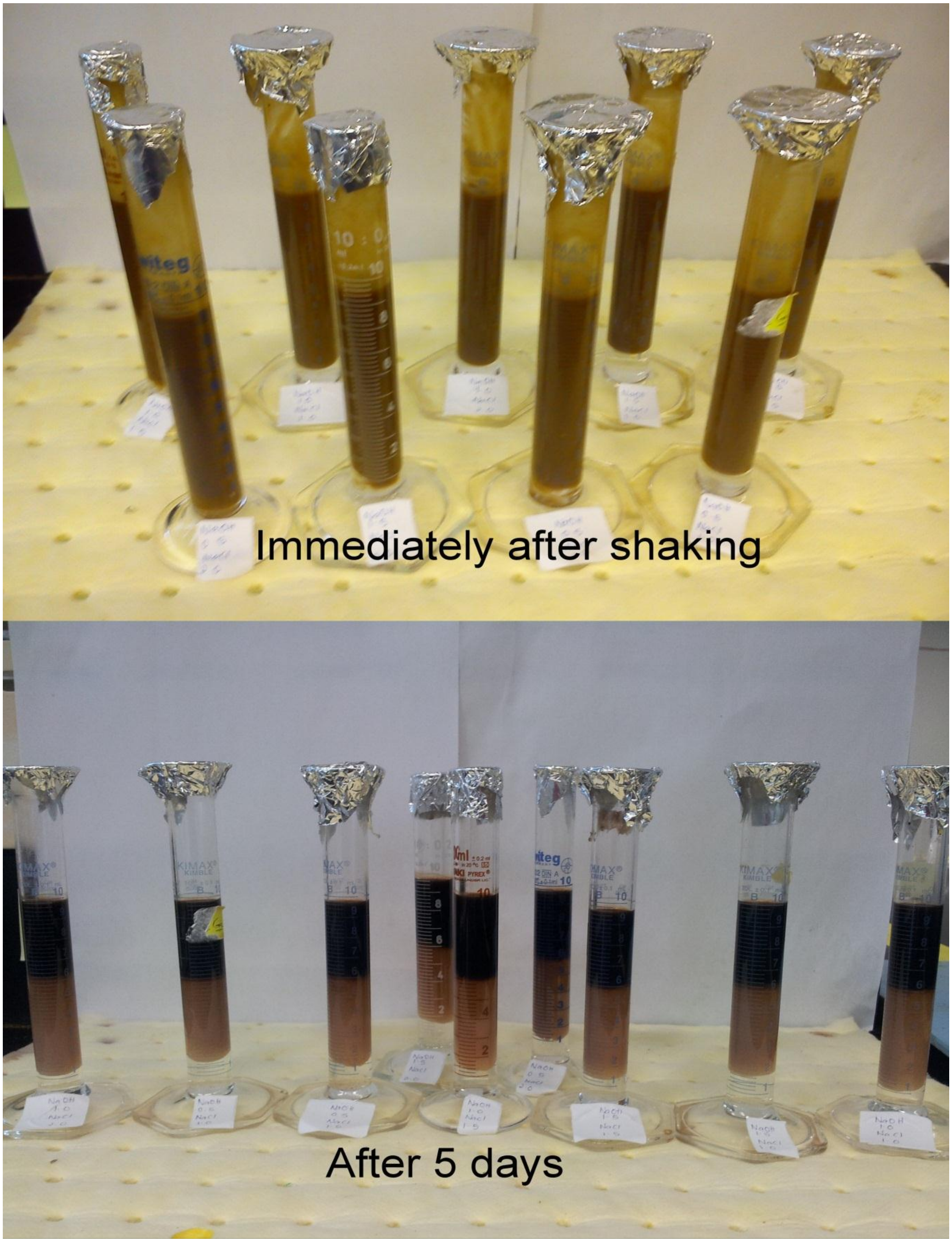

Figure 20 Observation after 5 days showing microemulsion formed between NaOH, brine and high acid number crude oil

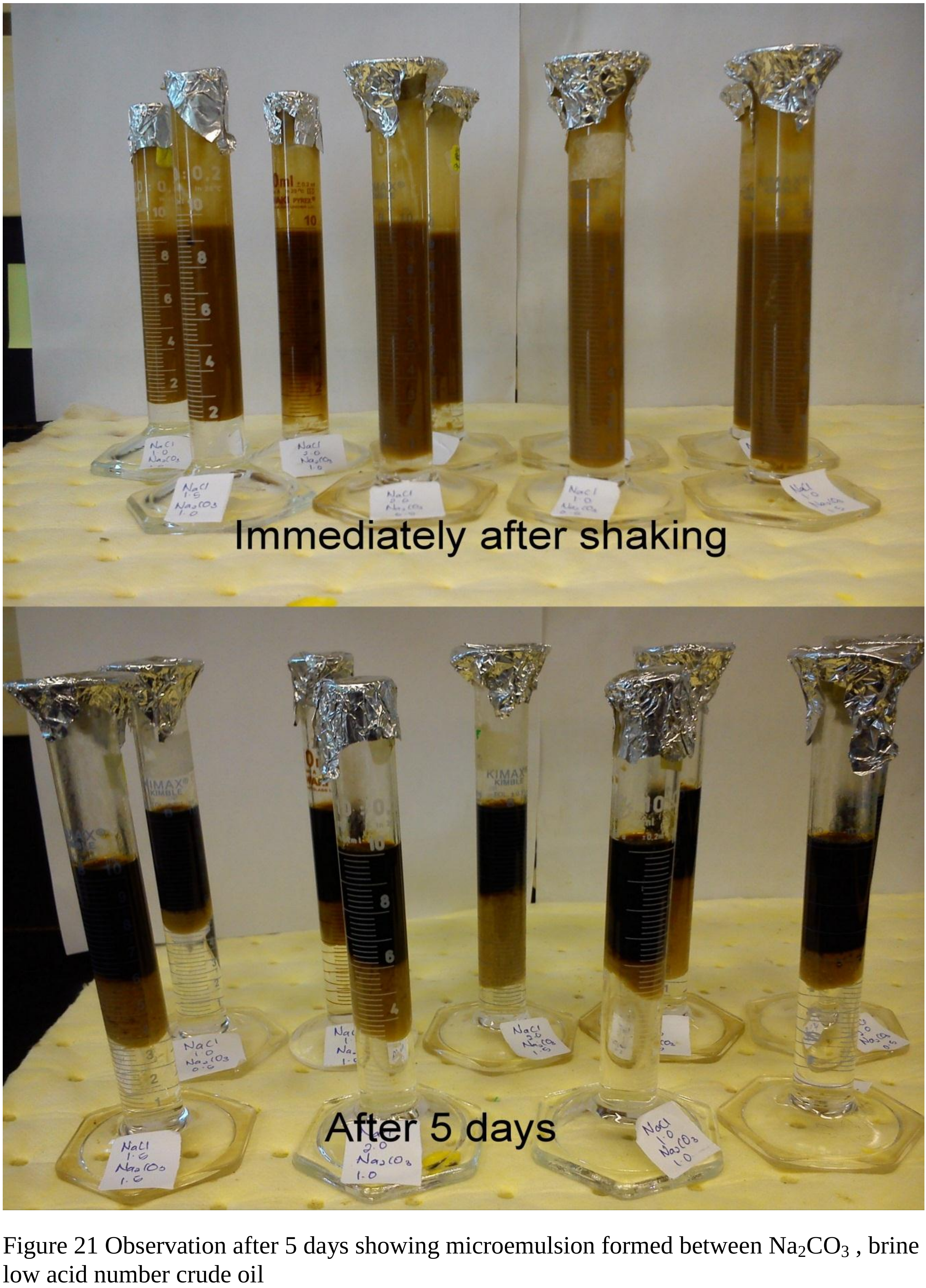

Figure 21 Observation after 5 days showing microemulsion formed between Na_2CO_3 , brine and low acid number crude oil

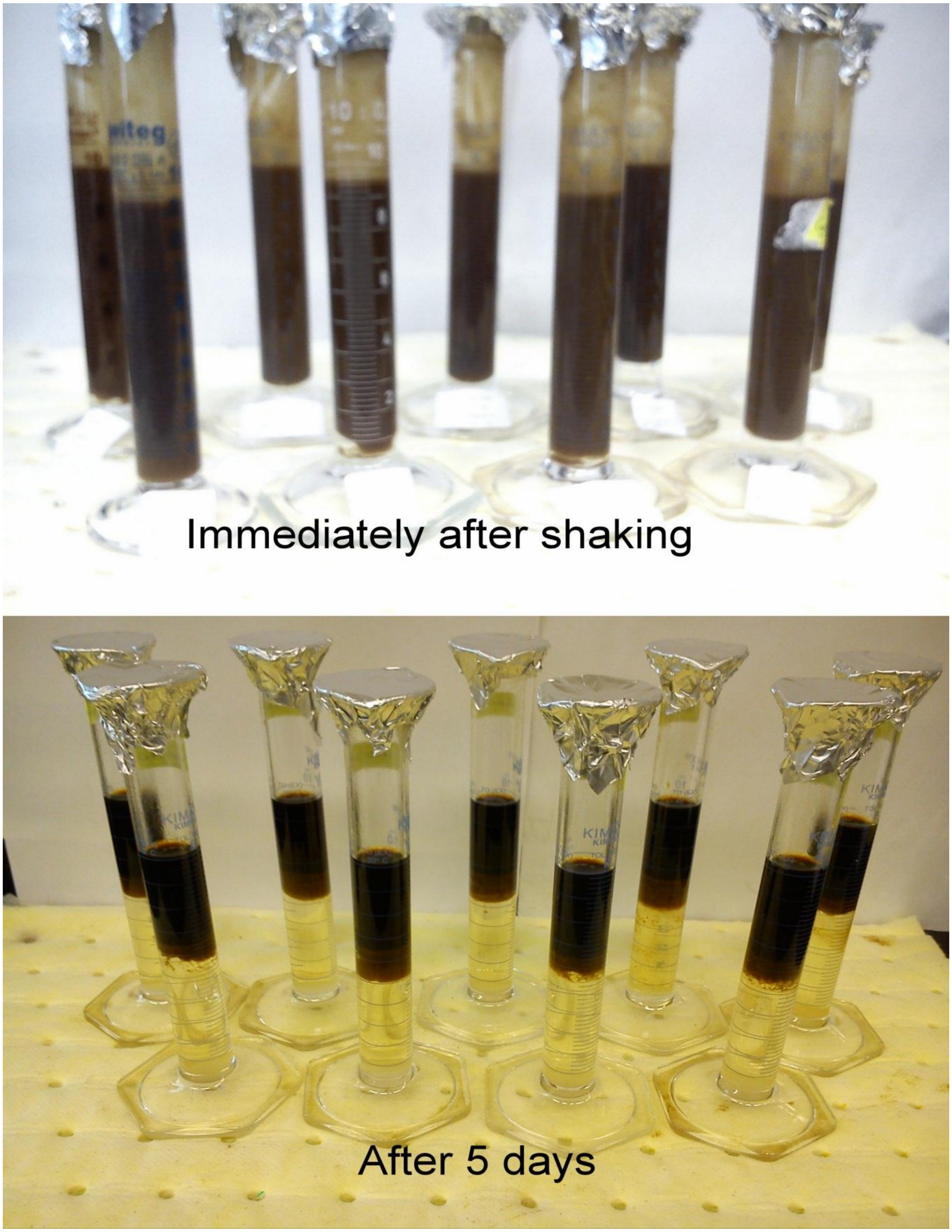

Figure 22 Observation after 5 days showing microemulsion formed between Na_2CO_3, brine and high acid number crude oil

Spinning Drop

Table 16 Spinning drop results for NaOH with brine with low acid number crude oil

NaCl(wt%)	NaOH(wt%)	Density(g/cm^3)	RI	RPM	IFT (mN/m)
1	0.5	0.9916	1.3281	4764	4.6351
1	1	0.997	1.3292	4206	1.525
1	1.5	1.0019	1.3304	2675	0.6189
1.5	0.5	0.9954	1.329	3004	0.5195
1.5	1	1.0001	1.3301	2074	0.3576
1.5	1.5	1.0055	1.3318	2417	0.356
2	0.5	0.9995	1.3303	2103	0.3278
2	1	1.0036	1.3311	2417	1.3117
2	1.5	1.0093	1.3322	2846	0.32

*Color codes are defined in Table 10.

Table 17 Spinning drop results for NaOH and brine with high acid number crude oil

NaCl (wt%)	NaOH (wt%)	Density (g/cm^3)	RI	RPM	IFT(mN/m)
1	0.5	0.9916	1.3281	4206	5.789
1	1	0.997	1.3292	4378	1.6137
1	1.5	1.0019	1.3304	2918	0.3932
1.5	0.5	0.9954	1.329	2532	0.5195
1.5	1	1.0001	1.3301	1545	0.2581
1.5	1.5	1.0055	1.3318	2089	0.9232
2	0.5	0.9995	1.3303	1803	0.1755
2	1	1.0036	1.3311	3776	0.8449
2	1.5	1.0093	1.3322	2802	0.5186

Table 18 Spinning drop results for Na_2CO_3 and brine with low acid number crude oil

NaCl(wt%)	Na_2CO_3 (wt%)	Density(g/cm^3)	RI	RPM	IFT(mN/m)
1	0.5	0.9911	1.3278	4278	2.3691
1	1	0.996	1.3289	2573	0.9232
1	1.5	0.9963	1.3299	1675	0.2935
1.5	0.5	0.9966	1.3289	2702	2.3903
1.5	1	0.9997	1.3295	2847	1.6619
1.5	1.5	1.0044	1.3306	2803	1.4953
2	0.5	0.9979	1.3294	2661	1.7106
2	1	1.003	1.3304	2661	0.4681
2	1.5	1.0092	1.3319	1659	0.1722

Table 19 Spinning drop results for Na_2CO_3 and brine with high acid number crude oil

NaCl(wt%)	Na_2CO_3 (wt%)	Density(g/cm^3)	RI	RPM	IFT(mN/m)
1	0.5	0.9911	1.3278	4890	1.789
1	1	0.996	1.3289	2045	0.5221
1	1.5	0.9963	1.3299	1415	0.2432
1.5	0.5	0.9966	1.3289	2517	1.4579
1.5	1	0.9997	1.3295	2318	0.4239
1.5	1.5	1.0044	1.3306	1674	0.3435
2	0.5	0.9979	1.3294	3219	2.0202
2	1	1.003	1.3304	1760	0.5925
2	1.5	1.0092	1.3319	2961	0.1722

The color codes in the tables refer to the classification as provided in Table 20.

Table 20 Classification of IFT readings

🟪	Low IFT (0.2-0.5)
🟧	Lowest IFT <0.2

IFT-Chun Huh Equation

Table 21 IFT results for NaOH with low acid number crude oil

NaCl	NaOH	Spinning Drop IFT	Chun Huh IFT	Error
1	0.5	4.6351	0.7479	83.8639
1	1	1.525	0.6750	55.7377
1	1.5	0.6189	0.7479	20.8471
1.5	0.5	0.5195	0.7889	51.8570
1.5	1	0.3576	0.6750	88.7584
1.5	1.5	0.356	0.8333	57.2800
2	0.5	0.3278	0.8333	60.6640
2	1	1.3117	0.8333	36.4692
2	1.5	0.32	0.7889	59.4370

Table 22 IFT results for NaOH with high acid number crude oil

NaCl	NaOH	Spinning Drop IFT	Chun Huh IFT	Error
1	0.5	5.789	1.054688	81.7812
1	1	1.6137	0.881633	45.3658
1	1.5	0.3932	1.054688	62.7188
1.5	0.5	0.5195	0.881633	69.7079
1.5	1	0.2581	0.612245	57.8437
1.5	1.5	0.9232	0.881633	4.5025
2	0.5	0.1755	0.991736	82.3038
2	1	0.8449	0.833333	1.3690
2	1.5	0.5186	0.833333	60.6890

Table 23 IFT results for Na_2CO_3 with low acid number crude oil

NaCl	**Na_2CO_3**	**Spinning Drop IFT**	**Chun Huh IFT**	**Error**
1	0.5	2.3691	0.675	71.5082
1	1	0.9232	0.788897	14.5476
1	1.5	0.2935	0.833333	64.7800
1.5	0.5	2.3903	0.881633	63.1162
1.5	1	1.6619	0.881633	46.9503
1.5	1.5	1.4953	0.747922	49.9818
2	0.5	1.7106	0.710059	58.4906
2	1	0.4681	0.747922	59.7783
2	1.5	0.1722	0.788897	78.1721

Table 24 IFT results for Na_2CO_3 with high acid number crude oil

NaCl	**Na_2CO_3**	**Spinning Drop IFT**	**Chun Huh IFT**	**Error**
1	0.5	1.789	0.642475	64.0875
1	1	0.5221	0.710059	36.0006
1	1.5	0.2432	0.675	63.9704
1.5	0.5	1.4579	0.747922	48.6986
1.5	1	0.4239	0.747922	76.4384
1.5	1.5	0.3435	0.710059	51.6238
2	0.5	2.0202	0.675	66.5875
2	1	0.5925	0.710059	19.8412
2	1.5	0.1722	0.788897	78.1721

Core Flooding

Properties of fluid at 70°C

Density of crude oil (acidic), g/cc	0.8000
Density of crude oil (low acid), g/cc	0.8002
Density of 1wt% NaCl, g/cc	1.01
Density of alkaline, 1.5wt%Na_2CO_3, 2wt% NaCl , g/cc	1.0092
Viscosity of crude oil (acidic), cp	3.817
Viscosity of crude oil (low acid), cp	3.817
Refractive index of alkaline	1.3319

Core flooding input data for all the runs

Flow rate for brine, cc/min	0.5
Flow rate for oil, cc/min	0.3
Flow rate for alkaline, cc/min	0.3
Inlet pressure(initial conditioning), psig	1900
Overburden pressure(initial conditioning), psig	3500

End Point Relative Permeability Calculation

General Darcy Law formula

$$\boxed{Q = \frac{kA\Delta P}{\mu \Delta x}}$$

Q = Flow rate (cm^3/s)

k = Absolute permeability (Darcy)

A = Core area (cm^2)

μ = Viscosity (cp)

ΔP = Pressure difference, Pinlet-Poutlet (atm)

Δx = Core length from inlet to outlet (cm)

Incorporating water displacement in oil using relative permeability system and rearranging the formula yields,

$$\boxed{krw = \frac{1000\ X\ TEF\ X\ \frac{q}{60}\ X\ \mu\ X\ L}{\pi\ X\ \frac{d2}{4}\ X\ \frac{\Delta P}{14.7}\ X\ k}}$$

TEF = temperature effect factor, usually 1

Q = flow rate(ml/min)

μ = viscosity

l = core length (cm)

d = core diameter (cm)

P = pressure (psig)

k = permeability (mD)

In the core flooding, once residual oil is produced either by alkaline flooding or water flooding, inlet and outlet pressure is measured to ensure a near steady state condition is achieved, where the ΔP/Δt is stable.

This can be obtained when,

Qinjectedfluid@inlet = Qproducedfluid@outlet

This is important in ensuring the relative permeability derived from Darcy law is valid because Darcy law requires a steady state, isothermal, incompressible and laminar flow.

Mobility ratio calculation using end points

$$\boxed{M = \frac{krw/\mu w}{kro/\mu o}}$$

Krw = relative permeability of water

Kro = relative permeability of oil

µw = viscosity of water

µo = viscosity of oil

This method is applied in all the mobility ratio and end point relative permeability calculation for all the runs.