Phase Behavior Study of Branched Alcohols as Additives in Surfactant Flooding

by

Nik Mohd Qusyairi Bin Mohd Zulkifli

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Supervisor: Mr Iskandar Dzulkarnain

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

APRIL 2012

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Petroleum Engineering Programme
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Bachelor of Engineering (Hons)
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Approved by,

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(Iskandar Dzulkarnain)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
April 2012

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.

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(Nik Mohd Qusyairi Bin Mohd Zulkifli)

Bachelor of Engineering (Hons) Petroleum Engineering
Tel: +609-6173154 (D/L); +019-4304595 (H/P)
Email: nik.qusyairi@gmail.com

ABSTRACT

The application of surfactant flooding for enhanced oil recovery is often precluded in reservoir where there is high brine salinity, high temperature and presence of hard water or divalent ions. This is because all these factors will degrade the surfactant to the extent that it will no longer be useful to be used in reducing the interfacial tension of oil-water phase. Therefore additives are usually used as part of the slug mixture to counter the negative effects inflicted by the above factors. As such we investigate the use of branched alcohols as possible additives to enhance surfactant flooding. Branched alcohol is chosen because it has lower miscibility in water and its potential for withstanding high temperature and high salinity. Previous research shows that the use of lower concentration of branched alcohol result in **similar amount of interfacial tension reduction** by using alkali. In this work branched alcohol samples were tested with anionic surfactants such as Dodecyl Trimethyl Ammonium Bromide (DTAB) and Sodium Dodecyl Sulfate (SDS) to evaluate their compatibility. Furthermore the formulations were optimized in order to withstand high temperature, hard water (> 500 ppm Mg^{2+}) and high brine salinity (>50,000 PPM). Phase behaviour study were also conducted to obtain low interfacial tension (<1.0 mN/m) and Winsor type III microemulsion suitable for surfactant flooding. In this work it was found that the formulation of 0.3 wt% of 2-methyl 1-butanol and 0.2 wt% of 3-(n, n –dimethylocatadecylamminia) propane sulfonate would form a Winsor Type III microemulsion. This will give an optimum salinity of 58,000 PPM with low interfacial tension of 0.12 mN/m, thus fulfill the objectives of this study.

As the demand of oil worldwide increased, the oil price is also increased gradually and with this enhanced oil recovery is becoming more important to oil and gas industry. These projects confer three solid **objectives.** First objective is to produce chemical formulation that can withstand high temperature, hard water (>500 PPM) and high brine salinity (>50,000 PPM). Second objective is to produce low interfacial tension (<1.0 mN/m) that form Winsor type III for the study of phase behavior-microemulsion characteristic in surfactant flooding. . Third objective is to measure the absorption of surfactant formulation above for fluid-fluid study. The

problem statement identified is that; surfactant flooding for enhanced oil recovery does not tolerant to (1) high salinity (2) high temperature (3) high hardness.

For the **methodology**, author focus on phase behavior screening, and then the formation was tested to demonstrate their performance in porous media. For the acceptable result, the next step is to run the core floods to test the potential use of chemical flooding for a field application with Dulang crude oil. The methodology will be discussed further in the phase behavior section. The **scopes of studies** include branched alcohol studies, phase behaviour, Winsor type system, and high salinity of brine, interfacial tension, and fluid properties such as density, refractive index etc and absorption test. Previous research showed that primary alcohols are able to reduce the interfacial tension (IFT) between surfactant and oil when added even in small amounts. **The finding for this research is** the formulation of 0.3 wt% of 2-methyl 1-butanol and 0.2 wt% of 3-(n, n –dimethylocatadecylamminia) propane sulfonate would form a Winsor Type III microemulsion. This will give an optimum salinity of 58,000 PPM with low interfacial tension of 0.12 mN/m, thus fulfill the objectives of this study.

ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious, the Most Merciful. Praise to Him the Almighty that in His will and given strength, author managed to complete the Final Year Project I and II in partial fulfilment of the requirement for the Bachelor of Engineering (Hons) in Petroleum Engineering at Universiti Teknologi PETRONAS.

Special and heartfelt thanks to author's beloved Supervisor, Mr Iskandar Dzulkarnain for the valuable guidance and advice. No word could possibly describe how indebted the author was to his supervisor. Enlightening and countless hours spent in sharing his insightful understanding, profound knowledge and valuable experiences, he inspired the author greatly to partake in Oil and Gas Industry in the future especially in PETRONAS. Without Mr Iskandar's help, the author would face a great difficulty in completing this project.

Deepest gratitude also goes to the names below, who's continuous support, and proactive leadership have truly been a great inspiration to author.

- Prof. Dr. Mariyamni Binti Awang from Petroleum Engineering Department
- Mr Sandeep from EOR Research Centre
- Mr Arsalan from EOR Research Centre
- Dr Khaled Abdalla Elraes from Petroleum Engineering Department

Thanks also go to all Universiti Teknologi PETRONAS's staff's especially lab technologists from block 15 that were so helpful and their warm supports had made this final year project a memorable and an informative one. Not to forget, to all lecturers and friends who have directly or indirectly lent a helping hand here and there. Finally, an honourable mention goes to author's families and friends for their understandings and supports in completing this project. Without helps of the particular that mentioned above, author would face many difficulties while doing this project.

(Nik Mohd Qusyairi Bin Mohd Zulkifli)

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LIST OF EQUATIONS

ABBREVIATIONS AND NOMENCLATURES

IFT	Interfacial Tension
PB	Phase behavior
CMC	Critical micelle concentration
WOR	Water-Oil Ratio
OWR	Oil-Water Ratio
PPM	Part-per-million
PETRONAS	Petroliam Nasional Berhad
DTAB	Dodecyl Trimethyl Ammonium Bromide
SDS	Sodium dodecyl sulfate
Wt%	Weight percentage
HLB	Hydrophilic-Lipophilic Balance
DI	De-ionized water
NaCl	Sodium chloride
$MgCl_2$	Magnesium chloride
ICIPEG	International Conference on Integrated Petroleum Engineering and Geoscience

CHAPTER 1:

INTRODUCTION

1.1 Background studies

This research presented in this work is two-fold. First, experiment was conducted for the purpose of studying the benefits of branched alcohol on surfactant phase behaviour and second (done by my colleague), observations were made through core floods of potential use for field application. The evaluation process builds upon the vast accumulation of knowledge over many decades and is touched upon the literature review chapter. This research focuses on chemical enhanced oil recovery to better understand the processes and mechanism in surfactant flooding.

Most of the chemical design is based off of mass transfer among phases observed in phase behaviour experiment. The primary chemicals studied in this research are branched alcohol. Branched alcohol is chosen because it has lower miscibility in water and its potential for withstanding high temperature and high salinity. Previous research shows that the use of lower concentration of branched alcohol result in **similar amount of interfacial tension reduction** by using alkali.

The systematic chemical evaluation in phase behaviour experiment was used in this research to develop formulation using phase behavior screening method. To improve the chances of success, a salinity gradient has been used by many years as an effective method of making the chemical flooding more robust in the field. This method was inspired by Rice University methodology. The salinity gradient is efficient because it helps to minimize the surfactant retention, makes the design less sensitive to reservoir and fluid property variations and uncertainties and thus reduces both the cost and risk of chemical flooding under field condition. My colleague has done the core flooding to test the candidate formulations and provide sine of the necessary parameters to simulate performance on reservoir scale.

1.2 Problems statement

Surfactant flooding for enhanced oil recovery does not tolerant to (1) high salinity (2) high temperature (3) high hardness. The application of surfactant flooding for enhanced oil recovery is often precluded in reservoir where there is high brine salinity, high temperature and presence of hard water or divalent ions. This is because all these factors will degrade the surfactant to the extent that it will no longer be useful to be used in reducing the interfacial tension of oil-water phase. Therefore additives are usually used as part of the slug mixture to counter the negative effects inflicted by the above factors. A paper from Prof. Dr. Mariyamni Awang showed that sodium carbonate gives better performance than sodium hydroxide, however its use is limited to low salinity conditions and high bivalent-cations are present. Hence, in this research, the author will develop the formulation that is tolerance to high salinity and hardness.

1.3 Objectives

The objectives of this project research are:

i) **To produce low interfacial tension and Winsor type III for the study of phase behavior in surfactant flooding.**

Description: This situation is ideal to achieve low interfacial tension values since only Winsor type III is favourable for EOR research.

ii) **To produce chemical formulation that can withstand high temperature, hard water and high salinity (>50,000 PPM).**

Description: Formulation should be tolerant to salinity and hardness of brine to imitate the formation fluids in which high bivalent-cations are present.

iii) Third objective is to measure the absorption of surfactant formulation above for fluid-fluid study.

Description: This test is to measure how well the branched alcohol will be absorbed into the formation.

1.4 Scope of studies

1.4.1 Branched alcohol:

The potential of the use of branched alcohol was studied. Branched alcohols were chosen because they have lower miscibility in water and its potential for withstanding high temperature and high salinity. Previous research shows that the use of lower concentration of branched alcohol result in **similar amount of interfacial tension reduction** by using alkali.

1.4.2 Phase behaviour:

The oil and water solubilisation ratios were calculated from interface measurement taken from phase behaviour pipettes. These interfaces were recorded over time as the mixtures approached equilibrium and the volume of any microemulsion that initially formed decreased or disappeared. Detailed procedure for creating phase behaviour experiment is discussed in Chapter 3.

1.4.3 Winsor type system

When a surfactant (from dropper) is added to an oil-water system (beaker) and the system is allowed to equilibrate, a microemulsion can form. Surfactant in a Type I case forms a microemulsion with the water phase, leaving excess oil as a separate phase. In a Type II case, the surfactant forms a microemulsion with the oil phase, leaving excess water. Type III describes the case in which the microemulsion is formed with both water and oil in a phase between the excess water and excess oil phases.

1.4.4 High salinity and hardness of brine

The scopes of studies also incorporate the experiment to correct the incompatibility with formation in which high salinity and high bivalent-cations are present. In real field implementation, to be conservative, salinity and hardness of brine should be considered as important affair to be studied.

1.5 Relevancy of project

In terms of the relevancy of this project, it poses a great deal of significance to the oil and gas industry since there were a lot of studies had been done for **primary alcohol** in enhanced oil recovery nowadays, but **not many have been done for branched alcohol.** For this project, the author was applying his theoretical and practical knowledge in petroleum engineering to solve the issue of maximizing hydrocarbon production by means of production enhancement. The basic principle involved ranges from reservoir studies, well completion and production, facilities engineering and production optimization. Hence, the outcome of this project is deemed crucial towards providing energy for the future.

1.6 Feasibility of project

All the objectives stated earlier are achievable and feasible in terms of this project duration and time frame. Since all the chemicals were already here in UTP when author start this project, the experiment was started as soon as the semester start. Previously during industrial internship, the author has already been part of the team for fluid-fluid study in PRSB. Since the author already acquired the basic understanding of SP/ASP flooding, the author is convinced to complete this project. Now, since great findings were achieved, it can be concluded that this research project is feasible and the stated objectives were achieved within the scope of this Final Year Project.

CHAPTER 2:

LITERATURE REVIEW

The purpose of this research project was to study the effect of branched alcohols on phase behaviour and its application to chemical flooding berea to recovery crude oil. Enhanced oil recovery becomes ever more important to the oil industry as the reservoir approaches their economic limit for primary and secondary methods and the price of crude oil justifies the examination of the tertiary recovery methods.

2.1 Branched alcohol

Alcohols can be regarded as organic analogues of water. Alcohols are usually classified as primary, secondary and tertiary.

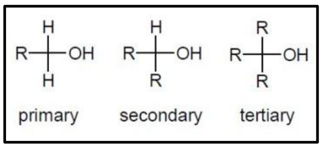

Figure 1: Alcohol general structure

The hydroxyl groups in alcohols can form hydrogen bonds with water, and many low molecular weight alcohols are miscible with water. Alcohols are more polar than hydrocarbons, and are better solvents for polar substances. Formaldehyde is the simplest aldehyde, and reaction with a Grignard reagent created a **primary alcohol,** which contains one more carbon atom than the original Grignard reagent. Reaction of an aldehyde with a Grignard reagent created a **secondary alcohol**.

Branched alcohol is chosen because it has lower miscibility in water and its potential for withstanding high temperature and high salinity. Previous research shows that the use of lower concentration of branched alcohol result in **similar amount of interfacial tension reduction** by using alkali. A recent study also proves that branched alcohol can withstand hardness tolerance of brine up to >1000ppm with CMC approximately at 638 PPM (Carty, 2004) Branched alcohols disturbs interfacial tension to inhibit the formation of gels and liquid crystal (Sanz and Pope, 1995). They also reduce the separation time and improve coalescence of microemulsion. Branched alcohol can be used to regulate the optimal salinity of a formula (Lelanne-Cassou, 1983)

Nelson's most important discovery was using surfactant to raise the optimal salinity to reasonable electrolyte levels and broaden the Winsor type III region. This increase in optimal salinity using surfactant should always be considered **when not using alkali,** because it expanded the oil and water solubilisation curves (Nelson *et al.*, 1984).

Hydrophobe branching dramatically affects foaming, leading to reduced performance in fluid mobility as branching increases. (Carty, 2004)

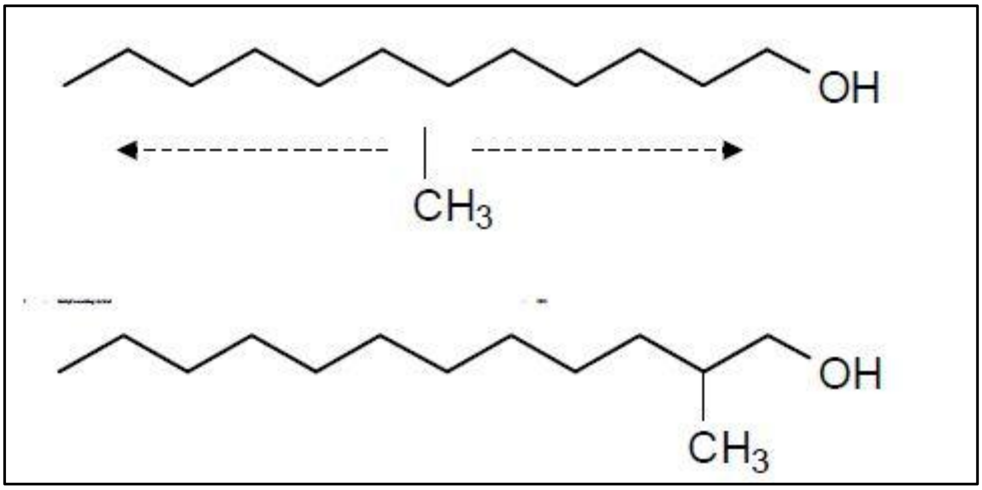

Figure 2: Typical branching type and positioning in OXO-alcohol (studied in this research)

2.2 Surfactant, its classification, and branching effect

Surface-active agents, or surfactants, are molecules that have both a water-soluble and an oil-soluble portion. Since both groups are on the same molecule, they adjust in water to obtain the lowest free energy. Primarily this is at the air/water interface where a properly chosen surfactant can provide wetting and foam. (Anthony, 2007) As the concentration of surfactant is raised above the CMC, aggregations called micelles form. It is interesting to note that despite the presence of micelles in water, if the micelles are small enough, the materials are still considered soluble because the structures are under the size that effects clarity. Solubility and homogeneity of concentration should not be confused. A surfactant present in water below its critical micelle concentration can be said to be soluble, but the concentration within the water is not uniform since most of the surfactant molecules are at the surface (Anthony, 2007). The ratio of water-soluble parts to oil-soluble parts changes as ethylene oxide is added, thus increasing the hydrophilic-lipophilic balance (HLB). One occasionally overlooked structural property that has an effect on surfactant properties is branching. (Anthony, 2007)

Classification:

This was one of the surfactant type used in this research: Anionic. **Anionic Surfactants** are disconnecting in water in an amphiphilic anion, and a cation, which is in general an alkaline metal (Na+, K+) or a quaternary ammonium. They are the most regularly used surfactants. They include alkylbenzene sulfonates (detergents), (fatty acid) soaps, lauryl sulfate (foaming agent), di-alkyl sulfosuccinate (wetting agent), lignosulfonates (dispersants) etc. Anionic surfactants account for about 50 % of the world production. (Jean-Louis, 2002)

Non-ionic Surfactants come as a close second with about 45% of the overall industrial production. They do not ionize in aqueous solution, because their hydrophilic group is of a non-dissociable type, such as alcohol, phenol, ether, ester, or amide (Jean-Louis, 2002). **Cationic Surfactants** are disconnecting in water into an amphiphilic cation and an anion, most often of the halogen type (Jean-Louis, 2002). Both type of surfactant were not tested in this research.

This was one of the surfactant type used in this research: Zwitterionic. When a single surfactant molecule show signs of both anionic and cationic dissociations it is called **amphoteric** or **zwitterionic**. This is the case of synthetic products like betaines or sulfobetaines and natural substances such as aminoacids and phospholipids. Some amphoteric surfactants are not sensitive to pH, whereas others are cationic at low pH and anionic at high pH, with an amphoteric behavior at intermediate pH (Jean-Louis, 2002). For conclusion of surfactant's literature: in this experiment, due to chemical limitation, only 2 type of surfactant will be used: Anionic and zwitterionic

2.3 Chemical design

An organized approach should be used for evaluating surfactant chemical formulation (Schelter & Bourrel, 1998) There is no universal solution; formulations must be created for each specific case study (Austad & Mitler, 1998); (Falls, 1992) (Jayani, 2002). This research focuses on chemical enhanced oil recovery to better understand the processes and mechanisms. Extensive research on surfactants for EOR was done in the 1870s and 1980s including pioneering research by Wade and Schechter at the University of Texas to better comprehend the role of surfactant structure on low interfacial tension (Jackson, 2006). In order for the surfactant to be cost effective, several criterions have to be met. The structure should amplify the chemicals affinity for the interface and create ultra low IFT and it should be sufficiently simple to minimize the number of synthesis steps for commercial production. **Since little can be done to alter fluid and rock properties deep in the reservoir, the IFT poses the most logical node to address.**

2.4 Phase behaviour theory

Phase behaviour experiments have been used to characterize chemical for EOR since late 1950s. There are many benefits of using phase behaviour as a screening method.

Oil solubilisation ratio is defined as the volume of oil divided by the volume of surfactant in microemulsion. All the surfactant is presumed to be in the emulsion phase. The oil solubilisation ratio is applied for Winsor type I and II behaviour. The volume of oil solubilised is found by reading the change between initial aqueous level and excess oil (top) interface level. The oil solubilisation ratio parameter is calculated as follows:

$$\delta o = \frac{vo}{vs}$$

$$\delta o = Oil\ solubilization\ ratio$$

$$vo = Volume\ of\ oil\ solubilized$$

$$vs = Volume\ of\ surfactant$$

Equation 1: Oil solubilization ratio

Water solubilisation ratio is defined as the volume of water divided by the volume of surfactant in microemulsion. All the surfactant is presumed to be in the emulsion phase. The water solubilisation ratio is applied for Winsor type II and type III behaviour. The volume of water solubilised is found by reading the change between initial aqueous level and excess water (bottom) interface level. The water solubilisation ratio parameter is calculated as follows:

$$\delta w = \frac{vw}{vs}$$

$$\delta o = Oil\ solubilization\ ratio$$

$$vw = Volume\ of\ water\ solubilized$$

$$vs = Volume\ of\ surfactant$$

Equation 2: Water solubilization ratio

Optimum solubilisation ratio occurs when the oil and water solubilisation is equal. The course nature of phase behaviour screening often does not include a data point at optimum, so the solubilisation curves are drawn for the oil and water solubilisation and the intersection of these two curves is defined as the optimum. The following is true for the optimum solubilisation ratio:

$$\delta o = \delta w = \delta *$$

$$\delta * = Optimum\ solubilization\ parameter$$

Equation 3: Optimum solubilization ratio

Criteria for selection of best formulation:

1. Solubilization ratio

   Must be high at optimum salinity in order to achieve the ultra low interfacial tension necessary to mobilize oil. Optimum solubilisation ratios that approach or exceed the value of 10 indicate the preferred surfactant and chemical condition.

2. Fluid microemulsion

   Must be able to flow through the reservoir under low pressure gradients between injection and producing wells. If the surfactant forms highly viscous phase such as gel, then it will not be transported long distance under these low gradient. Hence, it is very important that the surfactant rich phase not form gels, liquid crystal structures, or viscous macroemulsions.

3. Coalescence time

   The middle phase microemulsion should be quick to coalesce and equilibrate so that the mixture will approach local equilibrium in the reservoir. Fast equilibration of mixtures is indicative of good performance in oil recovery experiment.

2.5 Interfacial tension

Low interfacial tension was also shown to be possible with low surfactant concentration by Rosen (2005). Borderline CMC values were used (0.01 to 0.05 wt% active surfactant). In order to achieve an ultra-low interfacial tension (<0.01 mN/m) at these concentration, the surfactant must form lamellar micelles (Rosen *et al.*, 2005). The author plan to use the reinforced distortion of drop method: spinning drop to measure the IFT.

Spinning drop method:

Below are other available methods to measure IFT:

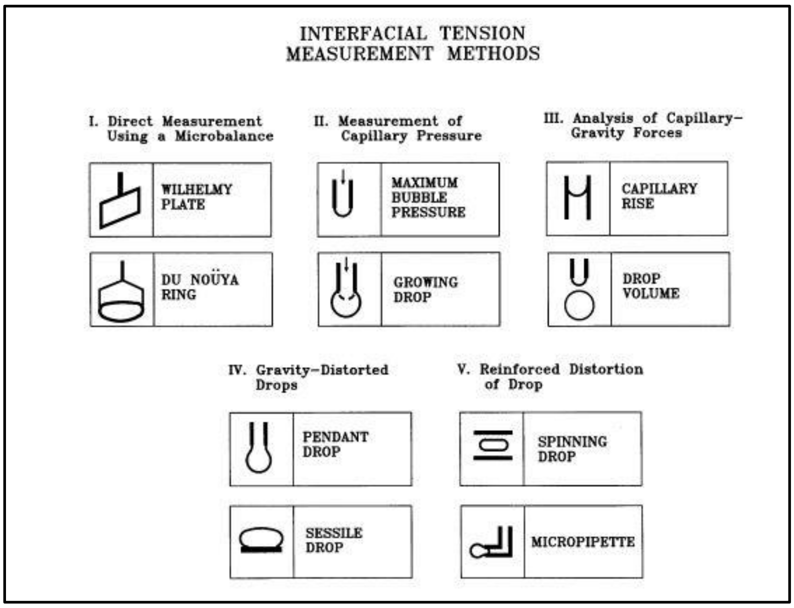

Figure 3: IFT measurement method

An approximate theory was developed by Bernard Vonnegut, in 1942, to measure the surface tension of the fluids, which is based on the principle that the interfacial tension and centrifugal forces are balanced at mechanical equilibrium. In the theory, the shape of the liquid drop at equilibrium is assumed as a circular cylinder.

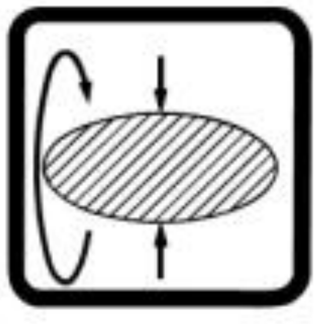

Figure 4: Liquid drop at equilibrium

The relation between the surface tension and angular velocity can be obtained in different ways. One of them is considering the total energy change in liquid drop as the summation of the change in kinetic energy and the surface energy:

$$\boxed{\Delta E = \Delta E_k + \gamma_s}$$

Equation 4: Summation of the change in kinetic energy and the surface energy

The terms in the equation can be replaced by the total kinetic energy change between the stationary fluid and the fluid with an angular velocity, ω, and the surface energy of the circular cylinder that has a length, L, and radius, R:

$$\boxed{E = \frac{\pi}{4} L\omega^2 R^2 \Delta\rho + 2\pi L R\sigma}$$

Equation 5: Total kinetic energy change

Where $\Delta\rho$ is the difference in fluid densities, and σ is the interfacial tension. At mechanical equilibrium, the energy change in radial direction has to be minimum. Differentiating the energy equation with respect to R, and solving for σ yields:

$$\boxed{\sigma = \frac{\Delta\rho\omega^2}{4} R^3}$$

Equation 6: Energy equation with respect to radius

This equation is known as Vonnegut's expression. Interfacial tension of any liquid that gives a shape very close to a cylinder at the equilibrium point can be estimated using this equation.

Relation with Critical Micelle Concentration (CMC)

Micelles are small colloidal particles, relative to the wavelength of light. When micelles form, the aqueous surfactant solution behaves as a micro-heterogeneous medium. The value of the CMC can be determined by the change in the physicochemical properties of the surfactant solution as the surfactant concentration increases. Experimentally, the CMC is found by plotting a graph of a suitable physical property as a function of surfactant concentration. An abrupt change of slope marks the CMC. The CMC can be affected by many variables (*6*), temperature and pressure being of relatively minor importance. It decreases with increasing hydrocarbon chain-length of the polar groups, and for ionic surfactants it also depends on the nature and concentration of counterions in solution.

CHAPTER 3:

METHODOLOGY

3.1 Research Methodology

In summary, these are the research methodology:

1. **Chemical preparation:**
    a) NaCl brine with salinity ranging from 10,000 PPM to 60,000 PPM
    b) Branched alcohol to 0.3 wt%
    c) Surfactant concentration to 0.2 wt%
2. **Phase behavior mixing**

    To prevent adverse effects, pipettes these solutions in this order:
    a) Electrolyte stocks
    b) Distilled water
    c) Surfactant stocks
    d) Branched alcohol
    e) Dulang crude oil
3. **Record emulsion level/characteristic**
    a) Interface level
    b) Emulsion features
    c) Coalescence time
4. **Compatibility test, repeat test at 70 ^{0}C for:**
    a) NaCl salinity (10,000, 20,000, 30,000, 40,000, 50,000 PPM)
    b) Hard water (Mg^+)
    c) Three types surfactants
    d) Two types of branched alcohols
5. **Measure interfacial tension**
6. **Measure absorption rate**

All this research methodology will be elaborate in details below.

3.2 Project Activities

Phase behaviour tests were conducted to investigate the effect of branched alcohol. Since light alcohol always cause the IFT to increase at optimum salinity (Jackson, 2006), so trade-offs included lower solubilisation parameters (increased IFT) and lower optimal salinities. Higher IFT is weighed against the benefit of lower microemulsion viscosities (mostly quantitatively by fluidity of interface) and faster separation of phases. Attempts will be made to find the proper concentration to remain within the criterion of optimum solubilisation ratio. Below are the author's early proposals for the experiments to be conducted:

1. Chemical preparation
2. Phase behavior mixing
3. Record emulsion level/characteristic
4. Compatibility test
5. Measure interfacial tension
6. Measure absorption rate
7. Summary of result and discussion

Phase behaviours as screening method for surfactant-alcohol formulation

This phase behaviours were conducted to find best formulation for specified crude oil: Dulang. This process involves investigating if there is microemulsion formed, how long it took to form and equilibrate if formed, what type of microemulsion formed and some of its properties such as density or refractive index.

Preparation of samples

a. Prepare surfactant stock solutions (at approximately 2.0 wt% active surfactant concentration)

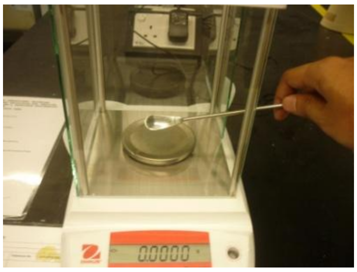

Figure 5: Mass balance

1. Mass of surfactant and de-ionized water (DI) were measured out on a mass balance and mixed in glass jars using magnetic stir bars.

(See surfactant preparation table in APPENDIX 3)

2. The quantity of chemical added was calculated based on activity and measured by weight percent of total solution.

Figure 6: Chemicals were prepared in fume chamber

Prepare brine stock solution (over a range of salinity and hardness)

1. The electrolyte and synthetic brine stocks were created as concentrated mixtures for use in the phase behaviour experiment. The electrolytes used included sodium chloride (NaCl), magnesium chloride ($MgCl_2$). These chemicals were stored in dry environment to prevent the adsorption of water. This reduced the introduction of error when preparing the concentration of electrolytes based on weight.

Figure 7: NaCl stored in dry environment

2. Once the stock solutions were prepared in glass bottles, magnetic stir bars were inserted and solutions mixed on a stir plate until all the components were dissolved into solution.

Figure 8: Branched alcohol was stored in glass

Pipetting solution

1. Phase behaviour components will be added volumetrically into 5 ml pipettes using pipetting instrument. Surfactant and brine stocks were mixed with DI water into labelled pipettes.
2. All of the phase behaviour experiments were created with a water oil ratio (WOR) of 1:1, which involved mixing 2 ml of the aqueous phase with 2 ml of the evaluated hydrocarbon.
3. Typical phase behaviour scan consisted of 10-20 measuring cylinder and each pipette being recognized as data point in the series.

Figure 9: Pipettes were then stored in an oven at 70 celcius

Order of addition

Consideration had to be given to the addition of the component since the concentrations were often several fold greater than the final concentration. Therefore, and order was established to prevent any adverse effects resulting from surfactant coming into direct contact with the concentrated electrolytes.

The desired sample compositions were made by combining the stocks in the following order:

1. Electrolyte stock
2. De-ionized water
3. Surfactant stock (4 types)
4. Branched alcohols (2 types)
5. Hydrocarbon (Dulang crude)

See formulation details (pipette numbering in APPENDIX 4)

Observation

1. Once the components were added to measuring cylinder, sufficient time was allocated to allow all the fluid to drain down the sides.
2. Then the aqueous fluid levels were recorded before addition of oil.
3. Measurement recorded in below sheet:

(See interface measurement in APPENDIX 4)

Tubes were observed for low tension upon mixing by looking at droplet size and how uniform the mixture appeared. Then the solutions were allowed to equilibrate over time and interface levels were recorded to determine equilibration time and surfactant performance. In this experiment, equilibrium time were limited to 3 days due to time constraint

Procedures before repeating the experiment on other parameters:

1. Chemicals were disposed in glass before disposing to the designated area

Figure 10: Disposing chemical

2. The pipettes need to be clean

Figure 11: Using heat from oven and also de-greaser to clean the pipettes

3. Only after completing step 1 and 2 the next compatibility test can be done.

Measurement and observations

Phase behaviour experiments were allowed to equilibrate in ovens that were set to the reservoir temperature for the crude oil being tested (Experimental temperature suggested from Ms Siti from PETRONAS Research Sdn Bhd was 70^0C). The fluid levels in the pipettes were recorded periodically and the trend in the phase behaviour observed over time. Equilibrium behaviour was assumed when fluid levels ceased to change within the margin of error for reading the samples.

Fluid interface

The fluid interfaces are the most crucial element of the phase behaviour experiments. From them, the phase volumes are determined and the solubilization ratios were calculated. The top and bottom interface were recorded as the scan transitioned from oil-in-water microemulsion to a water-in-oil microemulsion. Initial readings were taken after one day depending on the coalescence time. Measurements were taken thereafter at increasing time intervals until equilibrium was reached or the experiment was deemed unessential to continue observation. Graphs in Origin Pro were plotted for the solubilisation ratios as a function of branched alcohol concentration.

Interfacial Tension using Spinning drop tensiometer

Figure 12: Elongated Dulang oil drop due to centrifugal force

In this research the author used spinning drop method to measure the IFT. Measurements were carried out in a rotating horizontal tube which contains a dense fluid. A drop of a less dense liquid or a gas bubble is placed inside the fluid. Since the rotation of the horizontal tube creates a centrifugal force towards the tube walls, the liquid drop will start to deform into an elongated shape; this elongation stops when the interfacial tension and centrifugal forces are balanced. The surface tension between the two liquids (for bubbles: between the fluid and the gas) can then be derived from the shape of the drop at this equilibrium point.

Chemical used:

No	Name	Volume
1	Dodecyl Trimethyl Ammonium Bromide (DTAB) – anionic type-	10 gram
2	Sodium dodecyl sulfate (SDS) – anionic type-	10 gram
3	3-(n,n-dimethylocatadecylamminia) propane sulfonate –zwitterionic type-	10 gram
4	2-methyl 1-butanol	13 ml
5	LIAL	13 ml

Table 1: Chemical used (quantity)

3.3 Key milestone

Week	Key Milestone	Details	Tick
1	Topic selection	Should finalize three most relevant topic relevant to author	Done
2	Project familiarization	Should meet with supervisor to discuss the objective and expected result for the project	Done
3	Submission of draft extended proposal	Information gathering on the project is still ongoing, three draft of extended proposal should be sent to supervisor	Done
4	Submission of extended proposal	Should be able to understand the objective and literature review of other related project	Done
5	Submission of Progress report	Should meet lab technologist, supervisor and expert to get expected outcome of the project	Done
6	Literature review on branched alcohol and surfactant	Should have more than 5 literature review on each branched alcohol and surfactant	Done
7	Literature review on chemical design and branching effect	Should have more than 2 literature review on each chemical design and branching effect to be able to determine best chemical formulation technique	Done
8	Literature review on phase behaviour as screening method and relation with IFT	Should have more than 10 literature review on each phase behaviour and IFT to support the author's objective and compare outcome result	Done
9	Proposal Defence	Should be able to present the project to internal supervisor	Done
10	Meeting with expert to discuss experiment methodology	Should meet with Dr Khaled Abdalla Elraes to get his view on experiment methodology before starting the experiment	Done
11	Equipment and chemical confirmation/gathering	Problems should be identified from the meeting and special equipment was gathered	Done
12	Experiment methodology finalization and lab booking	Should properly book the lab for the experiment from lab executive	Done
13	Submission of draft interim report	Should send 1 draft interim report to supervisor and discuss the appropriate format	Done
14	Submission of interim report	Should submit the binded copy of completed report	Done

Table 2: Key milestone for FYP 1

Milestone for FYP 2:

Week	Key Milestone	Details	Tick
1	Project Work continues	Should borrow the pipettes (5ml) from chemical department and finalize the schedule of experiment and should request for 2-methyl 1-butanol from Mr Sandeep	Done
2	First batch of experiment on 2-methyl 1-butanol	Should start the experiment on first batch of branched alcohol with the three different surfactants and 5 different salinity. First is determining the best concentration should be used in the phase behaviour experiment due to chemical limitation	Done
3	Second batch of experiment on 2-methyl 1-butanol	Should start the experiment on the first batch of branched alcohol three different surfactants and 5 different salinity.	Done
4	Request for second branched alcohol	Need to request second chemical from Mr Arsalan to proceed to next phase behaviour experiment and write the result for first batch of branched alcohol before presenting to Mr Iskandar	Done
5	First batch of experiment on LIAL	Should start the experiment on second batch of branched alcohol with the three different surfactants and 5 different salinities	Done
6	Second batch of experiment on LIAL	Should start the experiment on the second batch of branched alcohol by using three different surfactants and 5 different salinities	Done
7	Submission of paper for ICIPEG 2012	Should write a technical paper for the ICIPEG 2012 and send to Mr Iskandar	Done
8	Submission of Progress report	Should write the progress report for all the result from both alcohol with the variable of three different surfactants and 5 different salinities and their analysis	Done
9	Poster submission on this project	Should produce a poster that consists of problem statement, motivation, objective, methodology, result, discussion and conclusion to be able to present for EDX.	Done
10	Submission of paper for Shell Inter-varsity student paper presentation contest 2012	Should write an abstract to be send to Shell Inter-varsity student paper presentation contest 2012 committees that dues on 14th of April	Done

11	Submission of draft report for the requirement of FYP II	Should write a draft report to be evaluated by Mr Iskandar first before finalizing the report to dissertation.	Done
12	Submission of dissertation for the requirement of FYP II	Should write a dissertation report to be send to three person that will evaluate this project	Done
13	Submission of technical paper for the requirement of FYP II	Should write a technical paper to be send to coordinator	Done
14	Oral presentation	Should make slide and present to supervisor, internal supervisor and external supervisor	Done
15	Submission of Project Dissertation (Hard Bound)	Should submit the hardbound for project dissertation for the requirement of FYP II	Done

Table 3 Key milestone for FYP 2

End of Final Year Project 1 and 2

3.5 Gantt Chart

FYP I

No	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic	■	■						MID-SEMESTER BREAK							
Detail:	(select from 3 best topic)															
2	Preliminary Research Topic		■	■	■	■										
Detail:	(Project familiarization)															
3	Submission of Extended Proposal Defence						■									
Detail:	(Find appropriate literature)															
4	Proposal Defence									■	■					
Detail:	(Discuss the project with expert)															
5	Project Work Continues											■	■	■		
Detail:	(Experiment methodology finalization)															
6	Submission of Interim Draft Report														■	
Detail:	(Finalization of background, literature and methodology of project)															
7	Submission of Interim Report															■
Detail:	(Start of the phase behaviour experiment)															

Table 4: Gantt Chart for FYP 1

FYP 2

No	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Project work continues	■	■	■	■	■	■	■	MID-SEMESTER BREAK							
Detail:	(Experiment continuation)															
2	Submission of progress report									■						
Detail:	(Verify new key milestone)															
3	Project work continues									■	■	■	■	■		
Detail:	(Result compilation and analysis/ may require using simulation)															
4	Pre-EDX												■			
Detail:	(Discuss the project further)															
5	Submission of draft report													■		
Detail:	(Finalize result from the two-fold experiment)															
6	Submission of dissertation (soft bound)														■	
Detail:	(Submit the final report)															
7	Submission of technical paper														■	
Detail:	(Papers for the project finalization)															
8	Oral presentation															■
Detail:	(Last presentation)															
9	Submission of project dissertation (hard bound)															■

Table 5: Gantt Chart for FYP 2

3.4 Tools/Equipment

No	Tools/equipment	Quantity	Purpose
1	Rack	1	To position the pipettes in vertical for observation
2	Pipettes (5ml)	10	Used to see more accurate emulsion separation
3	Mass balance	1	To measure the weight of chemical to be used
4	Spinning drop tensiometer	1	To measure the interfacial tension
5	Spectrophotometer (for absorption measurement)	1	To measure the absorption
6	Densitometer	1	To measure the density of chemicals phases
7	Refractometer	1	To measure the refractive index of chemicals phases
8	Oven	1	To imitate the reservoir temperature for accurate result
9	Syringe	1	To input the brine and distilled water into pipettes
10	Sealing glue	1	To seal the end of the pipettes
11	De-greaser	1	To effectively clean the pipettes to used for next experiment

Table 6: Tools/equipment quantity

CHAPTER 4:

RESULT AND METHODOLOGY

4.1 Data gathering and analysis

Brine salinity	Surfactant	Days	Vo/Vs	Vw/Vs	Comment
10,000	DTAB	1-3	0.5	10.8	Emulsion not fully stabilized
20,000	DTAB	1-3	0.4	12.3	Good coalescence time
30,000	DTAB	1-3	0.4	12.3	Cloudy emulsion
40,000	DTAB	1-3	0.4	12.3	Not a clear emulsion
50,000	DTAB	1-3	0.5	11.8	Good coalescence time
10,000	DTAB	4-5	0.5	10.8	Brownish red emulsion recorded
20,000	DTAB	4-5	1	11.3	Clear separation of brownish red emulsion
30,000	DTAB	4-5	0.4	12.3	Cloudy emulsion
40,000	DTAB	4-5	0.4	12.3	Visible separation
50,000	DTAB	4-5	0.4	12.1	Clear separation
10,000	DTAB	6-7	0.8	10.7	Clear phase behavior
20,000	DTAB	6-7	1.2	11.3	Clear separation
30,000	DTAB	6-7	0.7	12.3	Clear separation
40,000	DTAB	6-7	0.7	12.3	Clear phase behavior
50,000	DTAB	6-7	0.7	11.8	Clear separation
10,000	SDS	1-3	0.3	11	High viscosity emulsion, no separation
20,000	SDS	1-3	0.2	12.5	High viscosity emulsion, no separation
30,000	SDS	1-3	0.2	12.5	High viscosity emulsion, no separation
40,000	SDS	1-3	0.2	12.5	High viscosity emulsion and visible separation

50,000	SDS	1-3	0.3	12	High viscosity emulsion visible separation
10,000	Propane sulfonate	1-3	1	11.5	No separation
20,000	Propane sulfonate	1-3	0.5	11.5	Visible separation of cloudy emulsion
30,000	Propane sulfonate	1-3	0.7	12.5	Repeated scan
40,000	Propane sulfonate	1-3	0.8	11	Visible separation of cloudy emulsion
50,000	Propane sulfonate	1-3	3.7	8	Visible separation of cloudy emulsion
10,000	SDS	4-5	0.3	11	High viscosity emulsion and visible separation
20,000	SDS	4-5	0.8	11.5	Repeated scan
30,000	SDS	4-5	0.2	12.5	High viscosity emulsion and visible separation
40,000	SDS	4-5	0.2	12.5	High viscosity emulsion and visible separation
50,000	SDS	4-5	0.2	12.3	High viscosity emulsion and visible separation
10,000	Propane sulfonate	4-5	0.8	11.7	Visible separation of cloudy emulsion
20,000	Propane sulfonate	4-5	0.3	11.7	Visible separation of cloudy emulsion
30,000	Propane sulfonate	4-5	0.7	12.5	Visible separation of cloudy emulsion
40,000	Propane sulfonate	4-5	0.8	11	Repeated scan
50,000	Propane sulfonate	4-5	0.4	11.3	Visible separation of cloudy emulsion
10,000	SDS	6-7	0.6	10.9	Clear phase behavior
20,000	SDS	6-7	1	11.5	Clear separation

30,000	SDS	6-7	0.5	12.5	Clear separation
40,000	SDS	6-7	0.5	12.5	Clear phase behavior
50,000	SDS	6-7	0.5	12	Clear separation
10,000	Propane sulfonate	6-7	1.5	11	Clear cloudy phase behavior
20,000	Propane sulfonate	6-7	0.5	11.5	Clear cloudy separation
30,000	Propane sulfonate	6-7	-0.5	12.5	Clear cloudy separation
40,000	Propane sulfonate	6-7	0.5	11	Clear cloudy phase behavior
50,000	Propane sulfonate	6-7	0.2	11.3	Clear cloudy separation

Table 7: Summary of phase behavior result for 2-methyl 1-butanol

Date: 10/12/12 to 17/2/12

Objective of experiment:

To find out the best wt% of branched alcohol to be used for further experiment on broader range of salinity (10,000 PPM, 20,000 PPM, 30,000 PPM, 40,000 PPM, & 50,000 PPM). Using different wt% of branched alcohol, 10 pipettes is used to study the microemulsion produced by the formulation

Description:

The experiments were conducted on only one branched alcohol. In one week, 20 pipettes will be used to test the compatibility of the formulation. The results shown in this report highlight the 2-methyl 1-butanol since it is not test to LIAL 123 branched alcohol due to chemical limitation. The branched alcohol was test with 3 different surfactants which are anionic surfactants such as Dodecyl Trimethyl Ammonium Bromide (DTAB) and Sodium Dodecyl Sulfate (SDS) and zwitterionic surfactant such as propane sulfonate to evaluate their compatibility and 5 different branched alcohol concentrations ranging from 0.1-0.3 wt%.

Branched alcohol concentration	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
0.1	2.75	1	2.45	4.1
0.15	3.2	1	2.9	4.15
0.2	3.2	1	2.95	4.2
0.25	3	1	2.9	4.25
0.3	3.25	1	2.85	4.3

Table 8: Difference concentration branched alcohol Day 1-3 (10,000 PPM brine salinity)

Vo/Vs	**Vw/Vs**
3	13.5
3	9.5
2.5	10
1	12.5
4	10.5

Table 9: Solubilization ratio for difference branched oil concentration Day 1-3 (10,000PPM brine salinity)

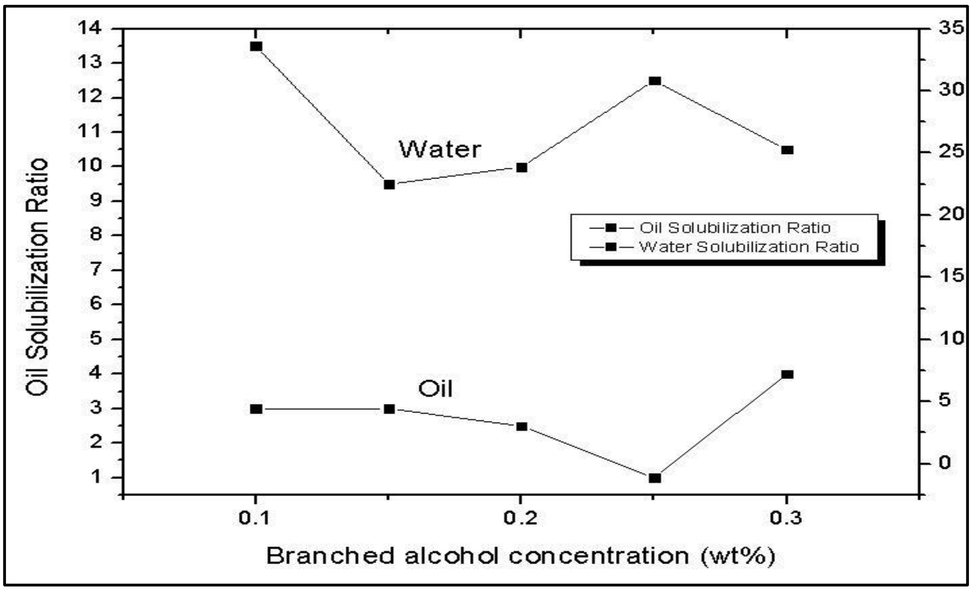

Figure 13: Solubilization Curve for difference concentration of branched alcohol Day 1-3 (10,000 PPM salinity)

Branched alcohol concentration	Aqueous level	Oil Level	Top interface	Bottom interface
0.1	3.15	1	2.95	4.1
0.15	3.4	1	3.05	4.15
0.2	2.8	1	2.7	4.2
0.25	3.15	1	3.05	4.25
0.3	3.45	1	3.32	4.3

Table 10: Difference concentration branched alcohol Day 1-3 (30,000 PPM brine salinity)

Vo/Vs	Vw/Vs
2	9.5
3.5	7.5
1	14
1	11
1.3	8.5

Table 11: Solubilization ratio for difference branched oil concentration Day 1-3 (30,000PPM brine salinity)

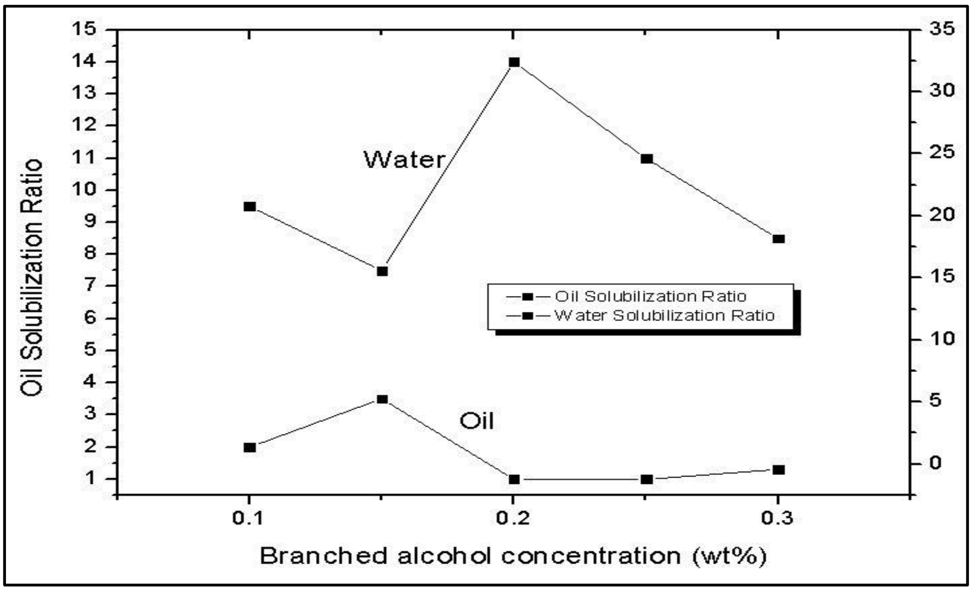

Figure 14: Solubilization Curve for difference concentration of branched alcohol Day 1-3 (30,000 PPM salinity)

Branched alcohol concentration	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
0.1	2.73	1	2.45	4.1
0.15	3	1	2.9	4.15
0.2	3	1	2.95	4.2
0.25	2.92	1	2.9	4.25
0.3	3.05	1	2.85	4.3

Table 12: Difference concentration branched alcohol Day 4-7 (10,000 PPM brine salinity)

Vo/Vs	**Vw/Vs**
2.8	13.7
1	11.5
0.5	12
0.2	13.3
2	12.5

Table 13: Solubilization ratio for difference branched oil concentration Day 4-7 (10,000PPM brine salinity)

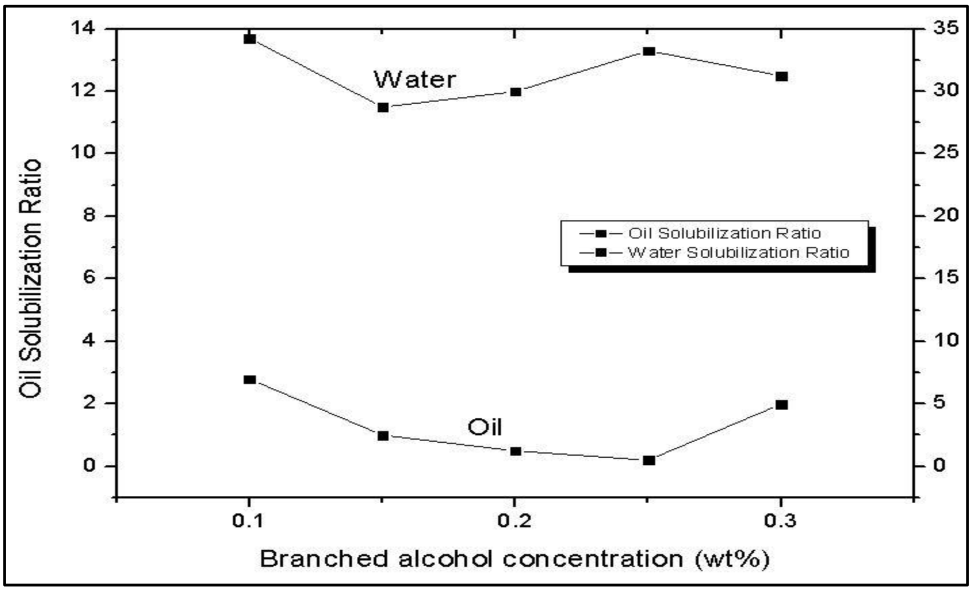

Figure 15: Solubilization Curve for difference concentration of branched alcohol Day 4-7 (10,000 PPM salinity)

Branched alcohol concentration	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
0.1	3.13	1	2.95	4.1
0.15	3.38	1	3.05	4.15
0.2	2.78	1	2.7	4.2
0.25	3.13	1	3.05	4.25
0.3	3.42	1	3.32	4.3

Table 14: Difference concentration branched alcohol Day 4-7 (30,000 PPM brine salinity)

Vo/Vs	**Vw/Vs**
1.8	9.7
3.3	7.7
0.8	14.2
0.8	11.2
1	8.8

Table 15: Solubilization ratio for difference branched oil concentration Day 4-7 (30,000PPM brine salinity)

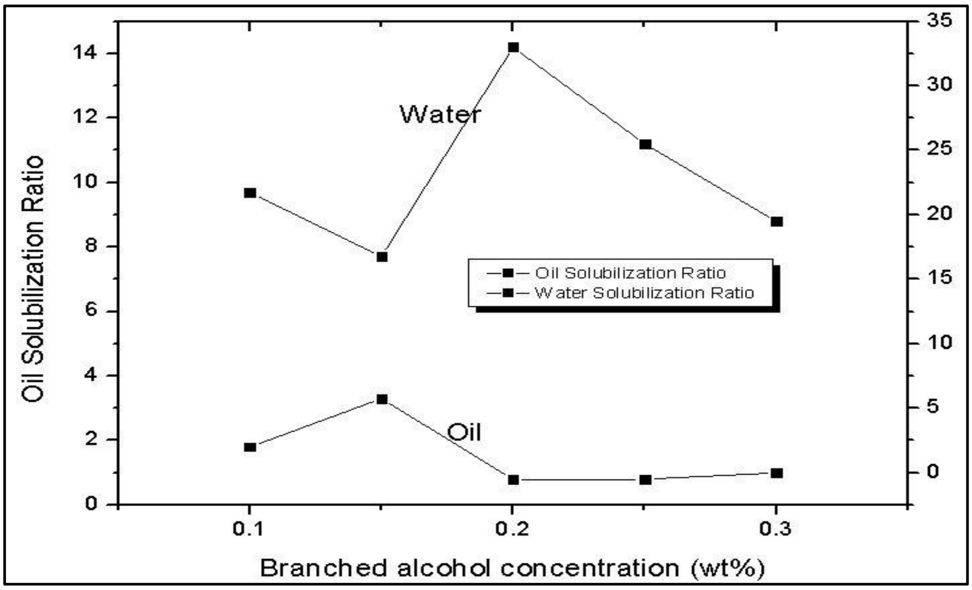

Figure 16: Solubilization Curve for difference concentration of branched alcohol Day 4-7 (30,000 PPM salinity)

Objective of experiment: To find out the best optimum solubilization ratio of using broader range of salinity (10,000 PPM, 20,000 PPM, 30,000 PPM, 40,000 PPM, & 50,000 PPM) with 3 type of surfactants and 2 type of branched alcohol. Using 5 pipettes, salinity of 10,000 PPM, 20,000 PPM, 30,000 PPM, 40,000 PPM, & 50,000 PPM are used for DTAB, SDS and propane sulfonate surfactant for 2-methyl 1-butanol and LIAL 123 branched alcohol

Description:

The experiments were conducted on both branched alcohol. In one week, 20 pipettes were used to test the compatibility of the formulation. To summarize, this experiment have taken 27 weeks by testing using 40 different formulations that generate 27 graphs.

The results shown in this report highlight the 2-methyl 1-butanol and the LIAL result will be attached in the appendix.

For LIAL123 branched alcohol, the emulsion is not clear and most do not have emulsion.

Both of the branched alcohols were test with 3 different surfactants which are anionic surfactants such as Dodecyl Trimethyl Ammonium Bromide (DTAB) and Sodium Dodecyl Sulfate (SDS) and zwitterionic surfactant such as propane sulfonate to evaluate their compatibility and 6 different salinities ranging from 10,000 – 60,000 PPM

Salinity (PPM)	Aqueous level	Oil Level	Top interface	Bottom interface
10000	3.22	1	3.17	4.3
20000	3.07	1	3.03	4.3
30000	3.07	1	3.03	4.3
40000	3.07	1	3.03	4.3
50000	3.12	1	3.07	4.3

Table 16: Result on difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 1-3)

Vo/Vs	Vw/Vs
0.5	10.8
0.4	12.3
0.4	12.3
0.4	12.3
0.5	11.8

Table 17: Solubilization ratio for difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 1-3)

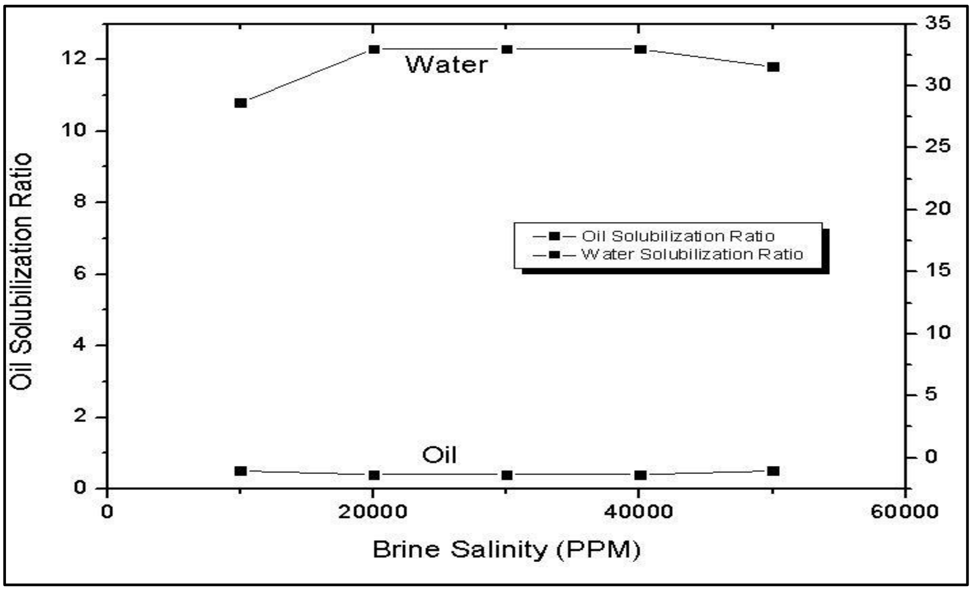

Figure 17: Solubilization curve for difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 1-3)

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.22	1	3.17	4.3
20000	3.17	1	3.07	4.3
30000	3.07	1	3.03	4.3
40000	3.07	1	3.03	4.3
50000	3.09	1	3.05	4.3

Table 18: Result on difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 4-5)

Vo/Vs	**Vw/Vs**
0.5	10.8
1	11.3
0.4	12.3
0.4	12.3
0.4	12.1

Table 19: Solubilization ratio for difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 4-5)

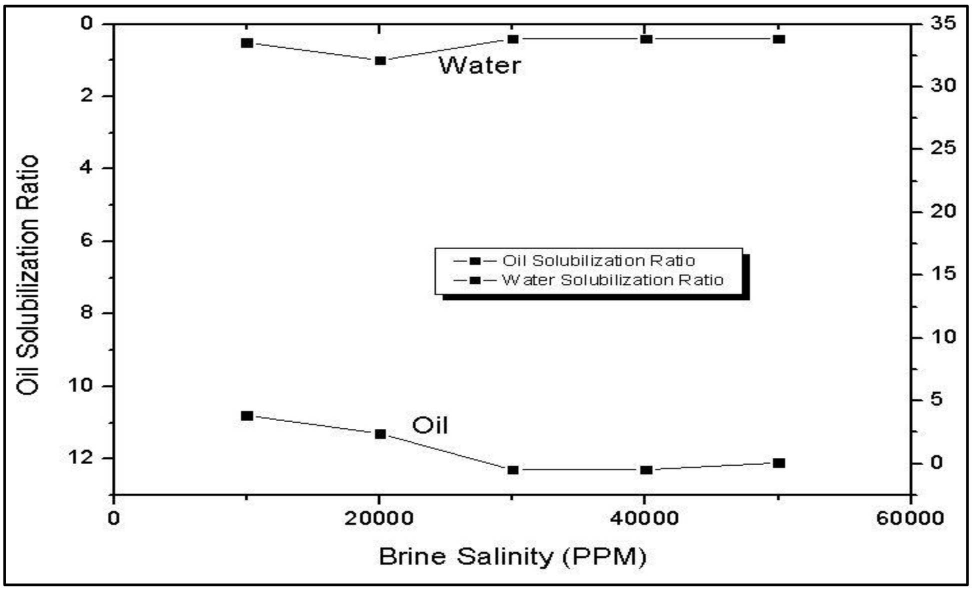

Figure 18: Solubilization curve for difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 4-5)

Salinity (PPM)	Aqueous level	Oil Level	Top interface	Bottom interface
10000	3.23	1	3.15	4.3
20000	3.17	1	3.05	4.3
30000	3.07	1	3	4.3
40000	3.07	1	3	4.3
50000	3.12	1	3.05	4.3

Table 20: Result on difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 6-7)

Vo/Vs	Vw/Vs
0.8	10.7
1.2	11.3
0.7	12.3
0.7	12.3
0.7	11.8

Table 21: Solubilization ratio for difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 6-7)

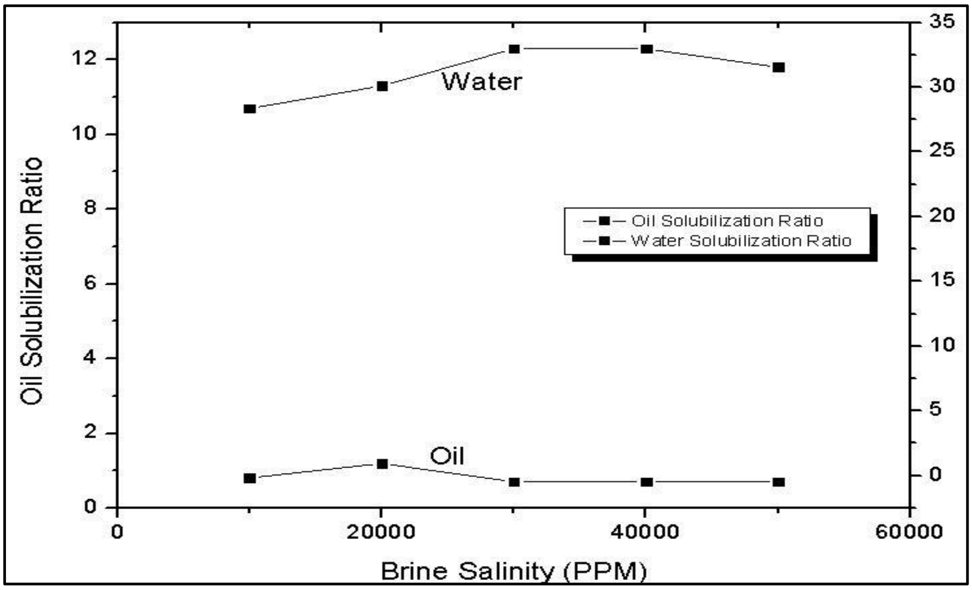

Figure 19: Solubilization curve for difference salinity using Dodecyl Trimethyl Ammonium Bromide (Day 6-7)

Salinity (PPM)	Aqueous level	Oil Level	Top interface	Bottom interface
10000	3.2	1	3.17	4.3
20000	3.05	1	3.03	4.3
30000	3.05	1	3.03	4.3
40000	3.05	1	3.03	4.3
50000	3.1	1	3.07	4.3

Table 22: Result on difference salinity using Sodium dodecyl sulfate (Day 1-3)

Vo/Vs	Vw/Vs
0.3	11
0.2	12.5
0.2	12.5
0.2	12.5
0.3	12

Table 23: Solubilization ratio on difference salinity using Sodium dodecyl sulfate (Day 1-3)

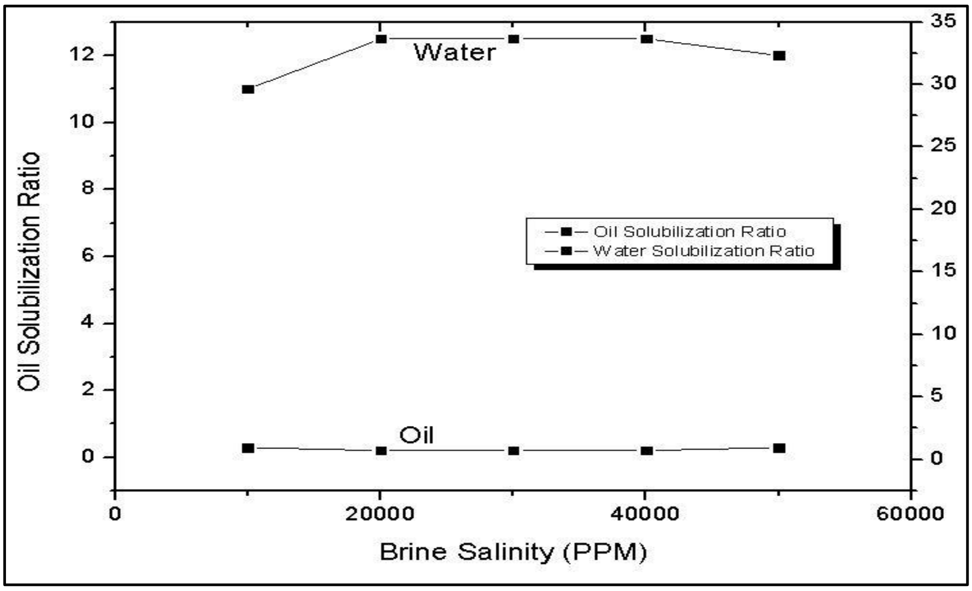

Figure 20: Solubilization curve on difference salinity using Sodium dodecyl sulfate (Day 1-3)

Salinity (PPM)	Aqueous level	Oil Level	Top interface	Bottom interface
10000	3.15	1	3.05	4.3
20000	3.15	1	3.1	4.3
30000	3.05	1	2.98	4.3
40000	3.2	1	3.12	4.3
50000	3.5	1	3.13	4.3

Table 24: Result on difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 1-3)

Vo/Vs	Vw/Vs
1	11.5
0.5	11.5
0.7	12.5
0.8	11
3.7	8

Table 25: Solubilization ratio for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 1-3)

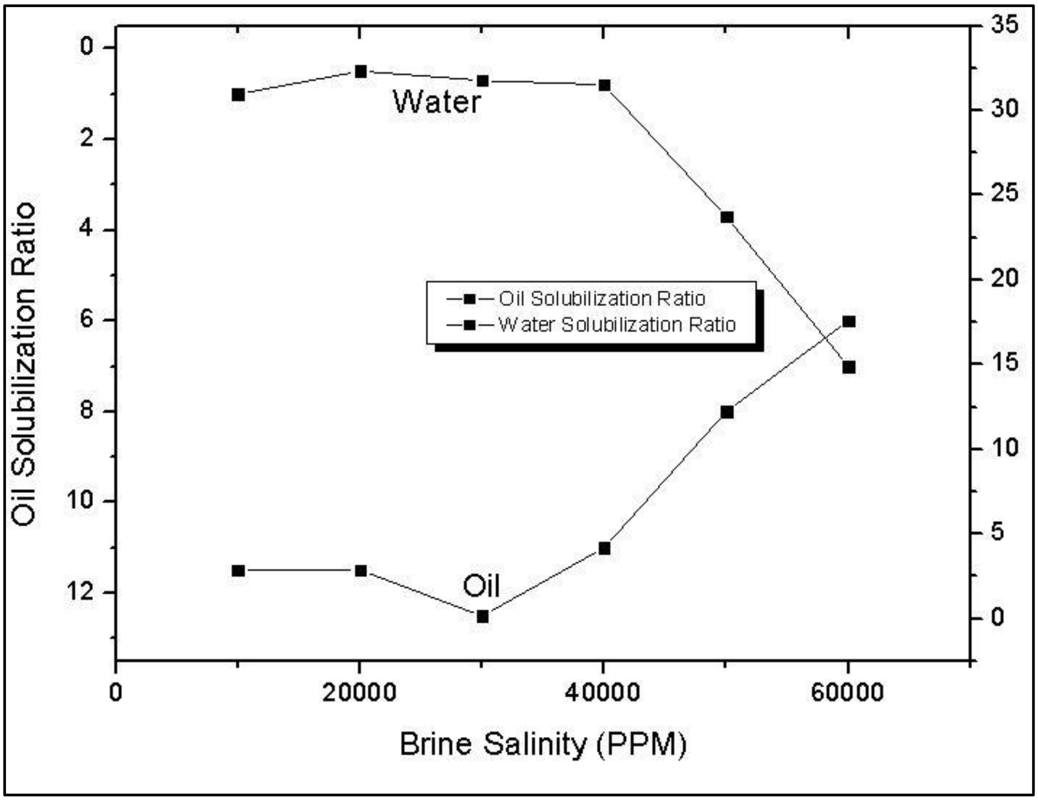

Figure 21: Solubilization curve for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 1-3)

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.2	1	3.17	4.3
20000	3.15	1	3.07	4.3
30000	3.05	1	3.03	4.3
40000	3.05	1	3.03	4.3
50000	3.07	1	3.05	4.3

Table 26: Result on difference salinity using Sodium dodecyl sulfate (Day 4-5)

Vo/Vs	**Vw/Vs**
0.3	11
0.8	11.5
0.2	12.5
0.2	12.5
0.2	12.3

Table 27: Solubilization ratio on difference salinity using Sodium dodecyl sulfate (Day 4-5)

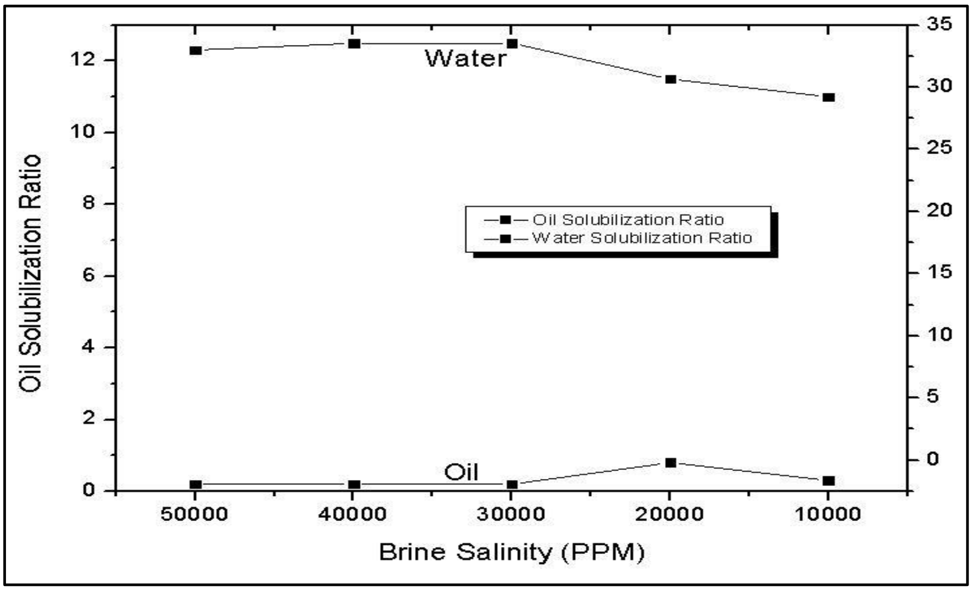

Figure 22: Solubilization curve on difference salinity using Sodium dodecyl sulfate (Day 4-5)

Salinity (PPM)	Aqueous level	Oil Level	Top interface	Bottom interface
10000	3.13	1	3.05	4.3
20000	3.13	1	3.1	4.3
30000	3.05	1	2.98	4.3
40000	3.2	1	3.12	4.3
50000	3.17	1	3.13	4.3

Table 28: Result on difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 4-5)

Vo/Vs	Vw/Vs
0.8	11.7
0.3	11.7
0.7	12.5
0.8	11
0.4	11.3

Table 29: Solubilization ratio for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 4-5)

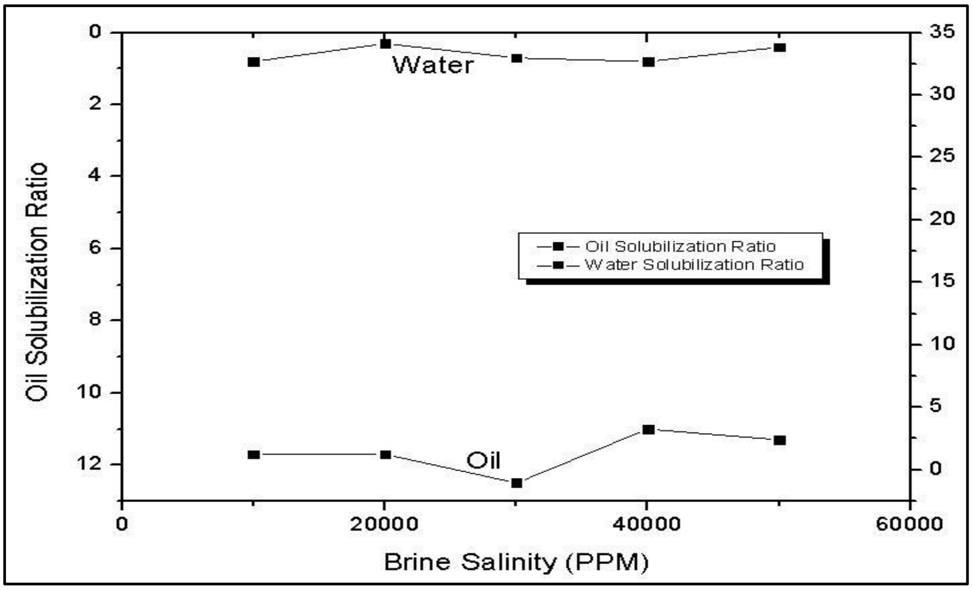

Figure 23: Solubilization curve for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 4-5)

Salinity (PPM)	Aqueous level	Oil Level	Top interface	Bottom interface
10000	3.21	1	3.15	4.3
20000	3.15	1	3.05	4.3
30000	3.05	1	3	4.3
40000	3.05	1	3	4.3
50000	3.1	1	3.05	4.3

Table 30: Result on difference salinity using Sodium dodecyl sulfate (Day 6-7)

Vo/Vs	Vw/Vs
0.6	10.9
1	11.5
0.5	12.5
0.5	12.5
0.5	12

Table 31: Solubilization ratio on difference salinity using Sodium dodecyl sulfate (Day 6-7)

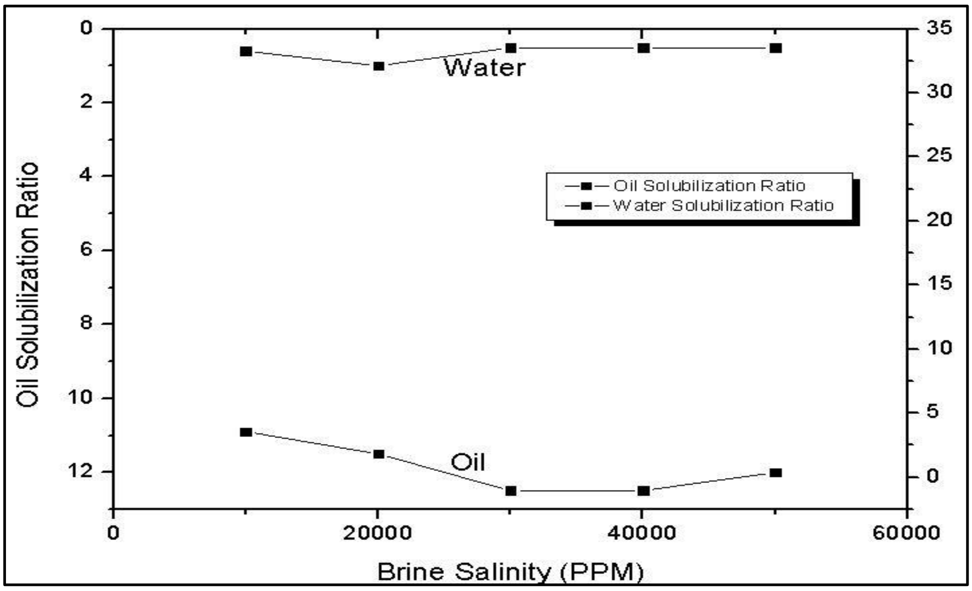

Figure 24: Solubilization curve on difference salinity using Sodium dodecyl sulfate (Day 6-7)

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.2	1	3.05	4.3
20000	3.15	1	3.1	4.3
30000	3.05	1	3.1	4.3
40000	3.2	1	3.15	4.3
50000	3.17	1	3.15	4.3

Table 32: Result on difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 6-7)

Vo/Vs	**Vw/Vs**
1.5	11
0.5	11.5
-0.5	12.5
0.5	11
0.2	11.3

Table 33: Solubilization ratio for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 6-7)

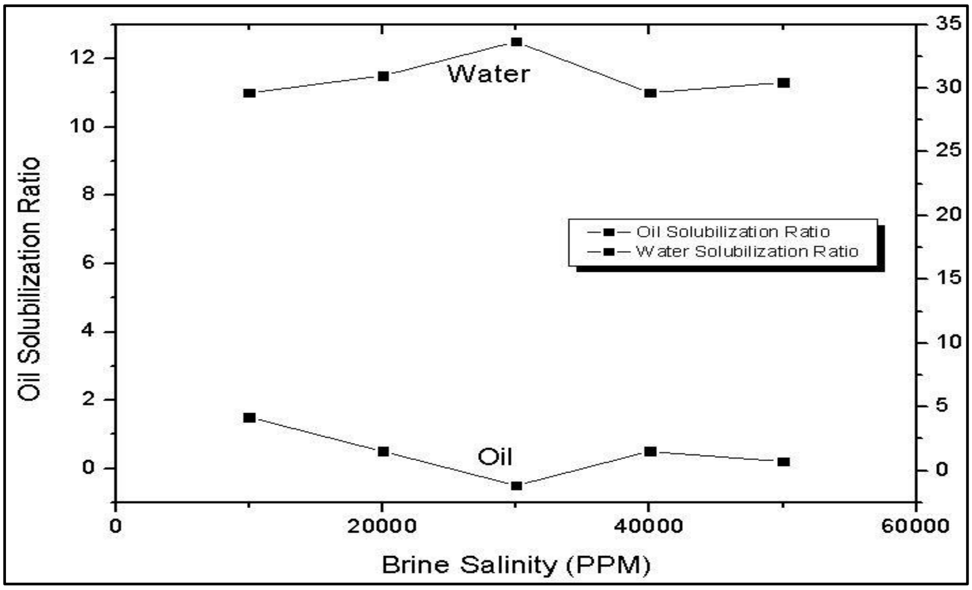

Figure 25: Solubilization curve for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 6-7)

End of 2-methyl 1-butanol experiment

LIAL 123 results:

Most of LIAL 123 phase behavior results do not show any clear separation. The values of the interfaces were measured by the help of using bright light. All the methodology used for 2-methyl 1-butanol was conducted with LIAL 123 and below are one of the result.

Salinity (PPM)	Aqueous level	Oil Level	Top interface	Bottom interface
10000	2.7	1	2.23	4.3
20000	3.03	1	2.9	4.3
30000	3.05	1	2.7	4.3
40000	3.22	1	3.03	4.3
50000	2.98	1	2.95	4.3

Table 34: Interface level for LIAL 123

Vo/Vs	Vw/Vs
4.7	16
1.3	12.7
3.5	12.5
1.9	10.8
0.3	13.2

Table 35: Solubilization ratio for LIAL 123

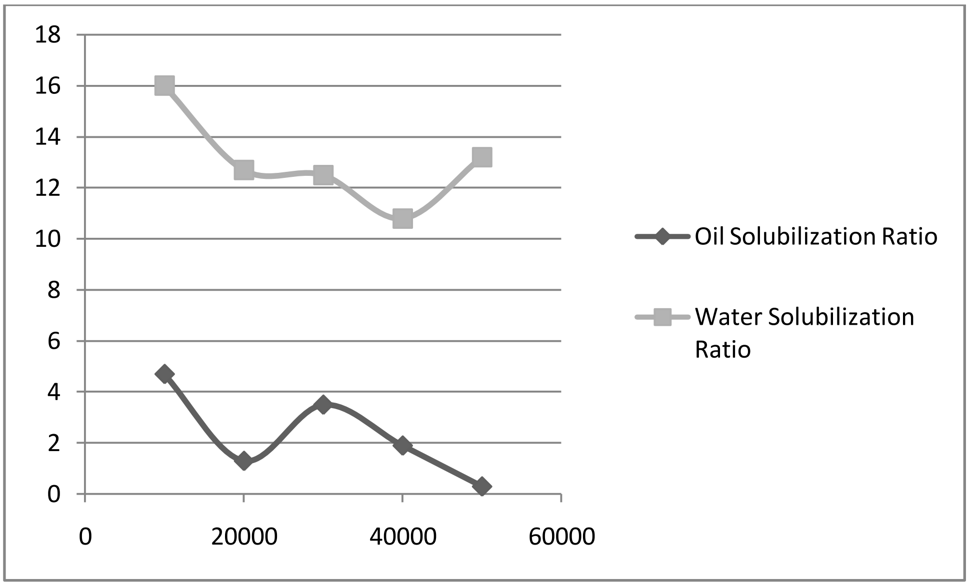

Figure 26: Solubilization ratio graph for LIAL 123

From the graph above, we can see that not only there are not many clear separation for LIAL 123 but the results were also not stable thus prove unreliable for reducing the interfacial tension and definitely cannot form a Winsor type III due to miscibility in oil

4.2 Experimentation/Project deliverables

Interfacial Tension:

Interfacial tension using spinning drop tensiometer by using result from Solubilization curve for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate. The interfacial tension of the formulation is then measured using spinning drop tensiometer (Data physic SVT 20):

Density of phase 1	1.0007 g/cm^3
Density of phase 2	0.87 g/cm^3
Drop type	Full
Spinning speed	3500 RPM
Phase 1 refractive index	1.33741
Interfacial result average	0.11708046

Table 2: Summary of interfacial tension meter test

Figure 27: Picture dated 29/02/12 of Dulang oil drop elongated when applied centrifugal force.

This proves that using branched alcohol can lower the interfacial tension into a low reading (0.11708046) after averaging the four reading for accuracy reason.

Absorption test:

Absorption test using spectrophotometer for difference concentration of 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate surfactant

These were the result of the absorption test using the spectrophotometer (UV-3150 Shimadzu) between surfactant 3-(n, n-dimethylocatadecylamminia) propane sulfonate and chemical formulation.

Scan speed : Fast

Sampling interval : 0.5

Wavelength : 250 nm.

Concentration (wt %)	Absorption	Comments
0.05	1.512	Average reading
0.10	1.789	Average reading
0.15	2.574	Average reading
0.20	-	Average reading

Table 3: Summary of absorption test result

4.3 Prototype

The choice of formulation to be conducted for core flooding and the reasons:

This experiment is a 7 weeks experiment using 40 different formulations that generate 27 graphs. This graph below show an optimum solubilization ratio of 6.2 with the use of 0.3wt% of 2-methyl 1-butanol and 0.2 wt% of 3-(N, n-dimethylocatadecylamminia) propane sulfonate that have a high optimum salinity of 58,000 PPM.

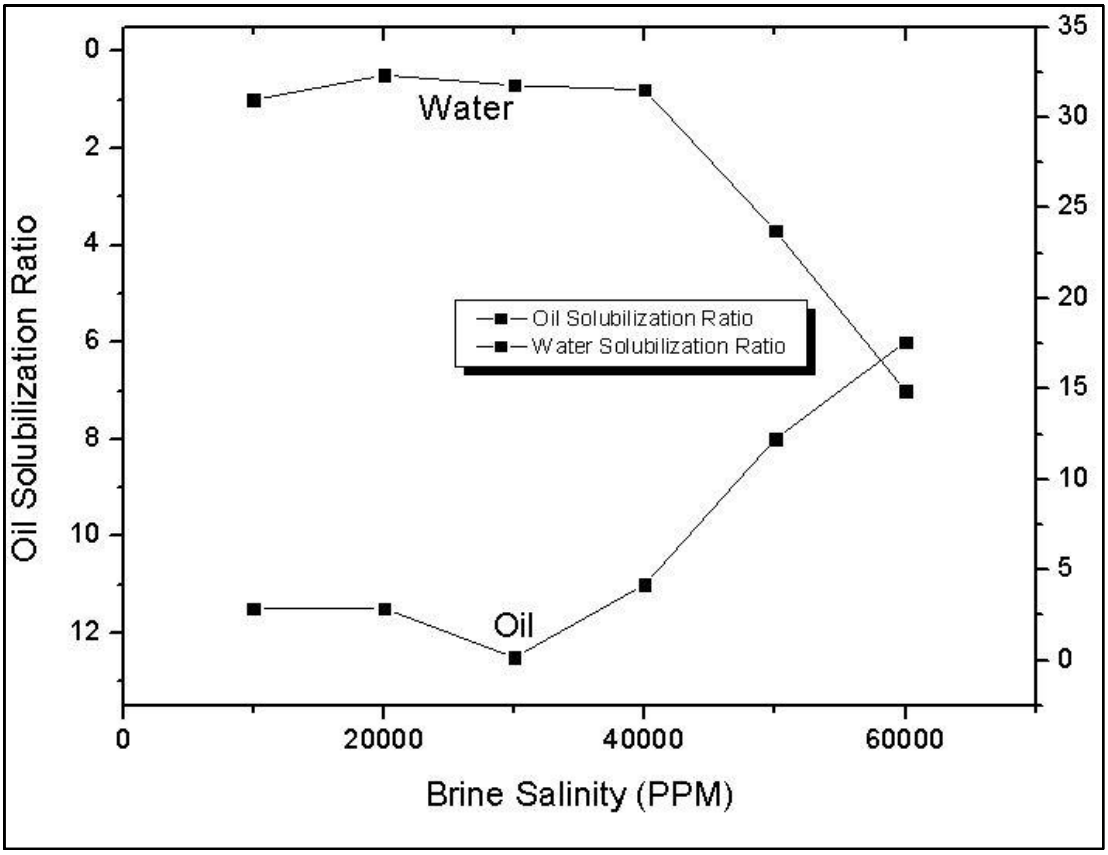

Figure 28: Solubilization ratio graph that shows 2-methyl 1-butanol optimum solubilization ratio 6 at 58,000 PPM of brine salinity by using propane sulfonate

Best result justifications:

This is the best formulation and it is suitable to continue to core flooding experiment to investigate the rock-fluid interaction. Although the optimum solubilization ratio does not exceed 10, the interfacial tension of this formulation is ultra-low and exceeds expectation The fluid microemulsion for 2-methyl 1 butanol are expected to be able to flow through the reservoir under low pressure gradients between injection and producing wells since it is not viscous. For LIAL branched alcohol, the emulsion is not clear and most don't have any emulsion at all. Previous researcher demonstrates that LIAL is miscible in oil. This show that more experiment should be conducted to investigate this branched alcohol.

From this experiment, the author found out that the coalescence time differ from each formulation, but most of the formulation need a minimum of three days to stabilize and some may take weeks. Because of the time constraint the author limits the experiment one week duration in order to investigate different formulation

CONCLUSION & RECOMMENDATIONS

CONCLUSION:

The conclusions of this project research are:

Throughout two semesters given to complete this project, two branched alcohol samples, 2-methyl 1-butanol and LIAL 123 were tested with anionic surfactants such as Dodecyl Trimethyl Ammonium Bromide (DTAB) and Sodium Dodecyl Sulfate (SDS) and zwitterionic surfactant such as propane sulfonate to evaluate their compatibility. The formulations were optimized in order to withstand high temperature, hard water (> 500 ppm Mg^{2+}) and high brine salinity (>50,000 PPM). Phase behaviour study were conducted to obtain low interfacial tension (<1.0 mN/m) and Winsor type III microemulsion suitable for surfactant flooding. In this work it was found that the formulation of 0.3 wt% of 2-methyl 1-butanol and 0.2 wt% of 3-(n, n –dimethylocatadecylamminia) propane sulfonate would form a Winsor Type III microemulsion. This will give an optimum salinity of 58,000 PPM with low interfacial tension of 0.12 mN/m, thus fulfill the objectives of this study.

RECOMMENDATION:

Suggested future works for expansion and continuation:

1. From the previous paper, it shows that, some benefits of faster equilibration are not well known or established in literature and this should be further investigated.
2. An expert in phase behavior experiment, Dr Khaled Abdalla Elraes, give his comment that future experiment should fully imitate the reservoir condition and reaction, the composition of the brine should be from an actual reservoir.
3. Repeat this test using the selected formation on other two types of surfactants (non-ionic and cationic) that have not been tested in this project to compare result and increase reliability of this study.

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APPENDICES

APPENDIX 1: CHUN HUH RELATION TO COMPARE IFT RESULT

APPENDIX 2: SURFACTANT PREPARATION

APPENDIX 3: FORMULATION DETAILS (PIPETTE NUMBERING)

APPENDIX 4: INTERFACE MEASUREMENT

APPENDIX 5: EXPECTED GRAPH RESULT

APPENDIX 6: LIAL 123 RESULTS

APPENDIX 7: PICTURES OF EXPERIMENT

APPENDIX 1: CHUN HUH RELATION TO COMPARE IFT RESULT

A correlation between oil and water solubilization ratios and IFT was suggested by Healy and Reed (1976) and a theoretical relationship was later derived by Chun Huh (1979). Lowest oil-water IFT occurs at optimum solubilisation as shown by the Chun Huh theory. This is equated to an IFT through the Chun Huh equation, where IFT varies with the inverse square of the solubilisation ratio:

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

For most crude oil emulsion, C=0.3 is a good approximation. Therefore, a quick and convenient way to estimate IFT is to measure phase behaviour and the use of Chun Huh equation to calculate IFT. The

IFT between microemulsion and water and/or oil can be very difficult and time consuming to measure and is subject to larger errors, so using the phase behaviour approach to screen is not only simpler and faster, but avoids the measurement problems and errors associated with measuring IFT especially of combination that show complex behaviour (gel etc) and will be screened anyway.

APPENDIX 2: SURFACTANT PREPARATION

Component	Name	Wt% desired	Mass calc. (grams)
Surfactant			
Surfactant			
Surfactant			
Surfactant			
	DI water		
	Total		
		pH	

Table 36: Surfactant preparation

APPENDIX 3: FORMULATION DETAILS (PIPETTE NUMBERING)

Pipette number	Branched alcohol (wt)	Surfactant (ppm)	Bran. Alcohol (ppm)	NaCl (ppm)	DI (ppm)
1	0.20%	1000	100	300	600
2	0.25%	1000	125	300	575
3	0.30%	1000	150	300	550
4	0.35%	1000	175	300	525
5	0.40%	1000	200	300	500
6	0.45%	1000	225	300	475
7	0.50%	1000	250	300	450
8	0.55%	1000	275	300	425
9	0.60%	1000	300	300	400
10	0.65%	1000	250	300	375

Table 37: Formulation details (Pipette number)

APPENDIX 4: INTERFACE MEASUREMENT

Branched Alcohol	Aqueous Level	Oil Level	Top Interface	Bottom Interface	Comments
0.20%					
0.25%					
0.30%					
0.35%					
0.40%					
0.45%					
0.50%					
0.55%					
0.60%					
0.65%					

Table 38: Interface measurement

APPENDIX 5: EXPECTED GRAPH RESULT

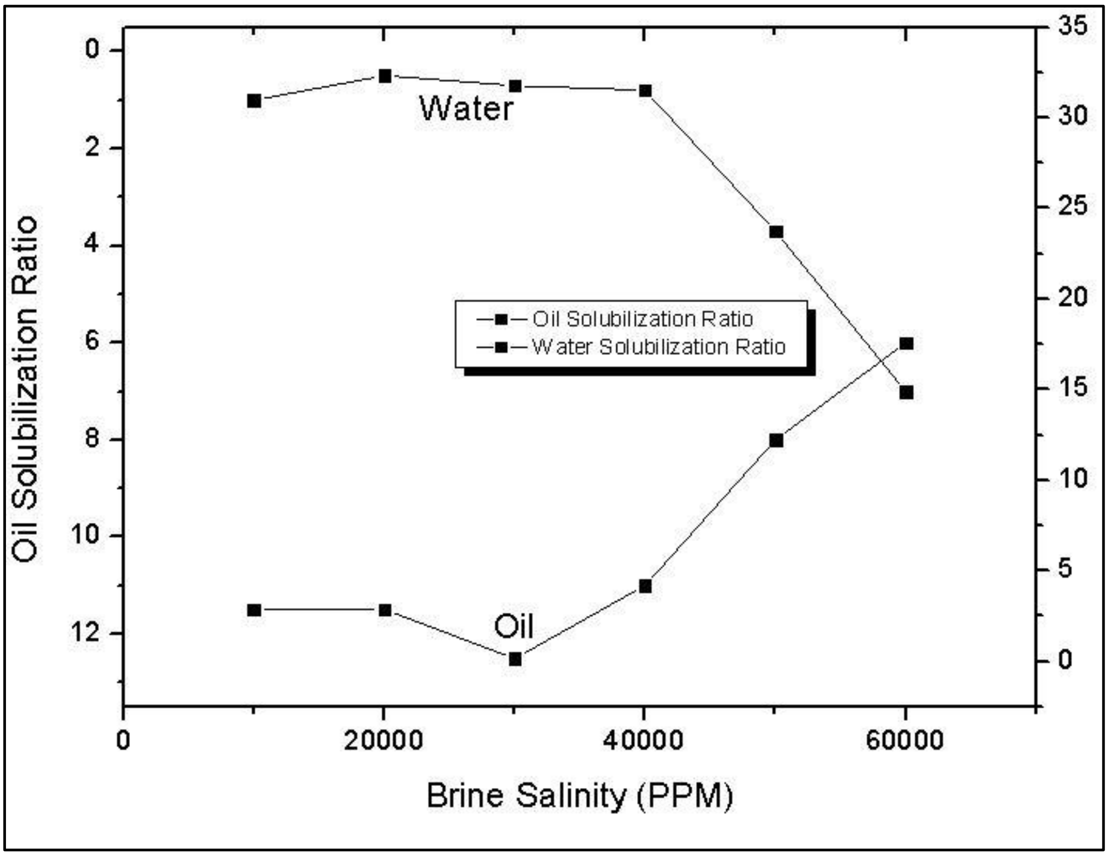


Figure 29: Example of solubilisation ratio graph

APPENDIX 6: LIAL 123 RESULTS

Most of LIAL 123 phase behavior result does not show any clear separation. The value of the interface is measured by the help of using bright light.

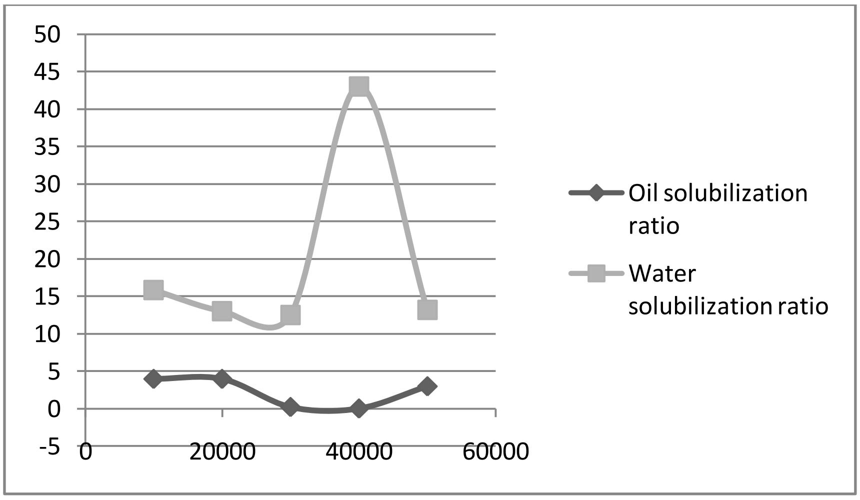
From the graph below, we can see that not only there are not many clear separation for LIAL 123 but the results were also not stable thus prove unreliable for reducing the interfacial tension and definitely cannot form a Winsor type III due to miscibility in oil

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	2.72	1	2.32	4.3
20000	3	1	2.6	4.3
30000	3.05	1	3.03	4.3
40000		1		4.3
50000	2.98	1	2.68	4.3

Table a: Result on difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 1-3

Vo/Vs	**Vw/Vs**
4	15.8
4	13
0.2	12.5
0	43
3	13.2

Table b: Solubilization ratio for difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 1-3



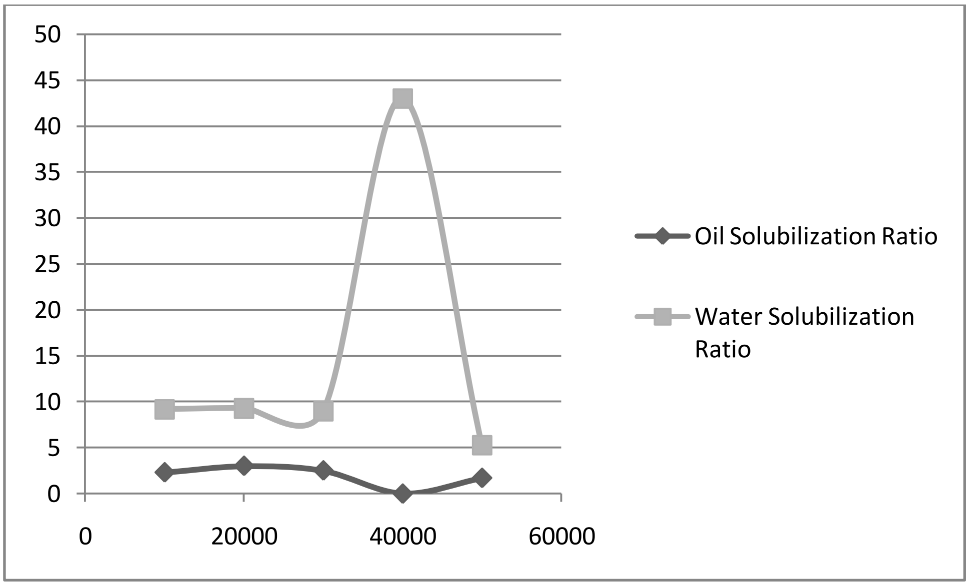
Graph a: Solubilization curve for difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 1-3

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.38	1	3.15	4.3
20000	3.37	1	3.07	4.3
30000	3.4	1	3.15	4.3
40000		1		4.3
50000	3.77	1	3.6	4.3

Table c: Result on difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 4-5

Vo/Vs	**Vw/Vs**
2.3	9.2
3	9.3
2.5	9
0	43
1.7	5.3

Table d: Solubilization ratio for difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 4-5



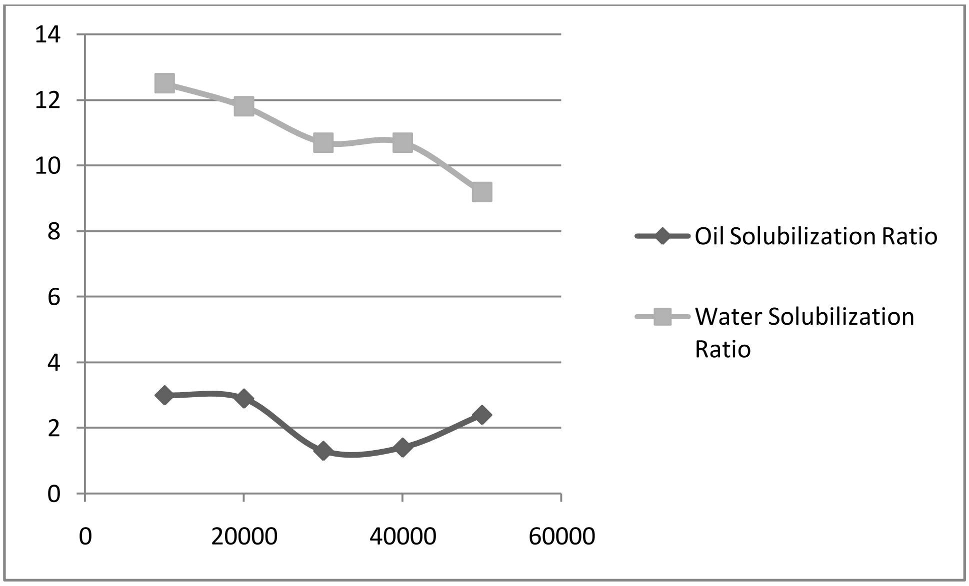
Graph b: Solubilization curve for difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 4-5

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.05	1	2.75	4.3
20000	3.12	1	2.83	4.3
30000	3.23	1	3.1	4.3
40000	3.23	1	3.09	4.3
50000	3.38	1	3.14	4.3

Table e: Result on difference salinity using using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 6-7

Vo/Vs	**Vw/Vs**
3	12.5
2.9	11.8
1.3	10.7
1.4	10.7
2.4	9.2

Table f: Solubilization ratio for difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 6-7



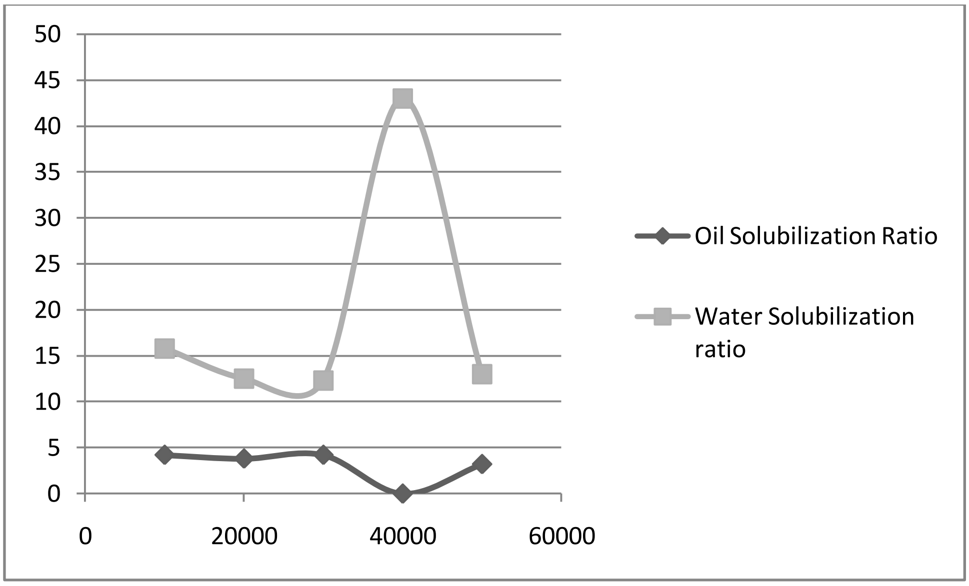
Graph c: Solubilization curve for difference salinity using Dodecyl Trimethyl Ammonium Bromide (DTAB) Day 6-7

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	2.72	1	2.3	4.3
20000	3.05	1	2.67	4.3
30000	3.07	1	2.65	4.3
40000		1		4.3
50000	3	1	2.68	4.3

Table g: Result on difference salinity using Sodium dodecyl sulfate (SDS) Day 1-3

Vo/Vs	**Vw/Vs**
4.2	15.8
3.8	12.5
4.2	12.3
0	43
3.2	13

Table h: Solubilization ratio for difference salinity Sodium dodecyl sulfate (SDS) Day 1-3



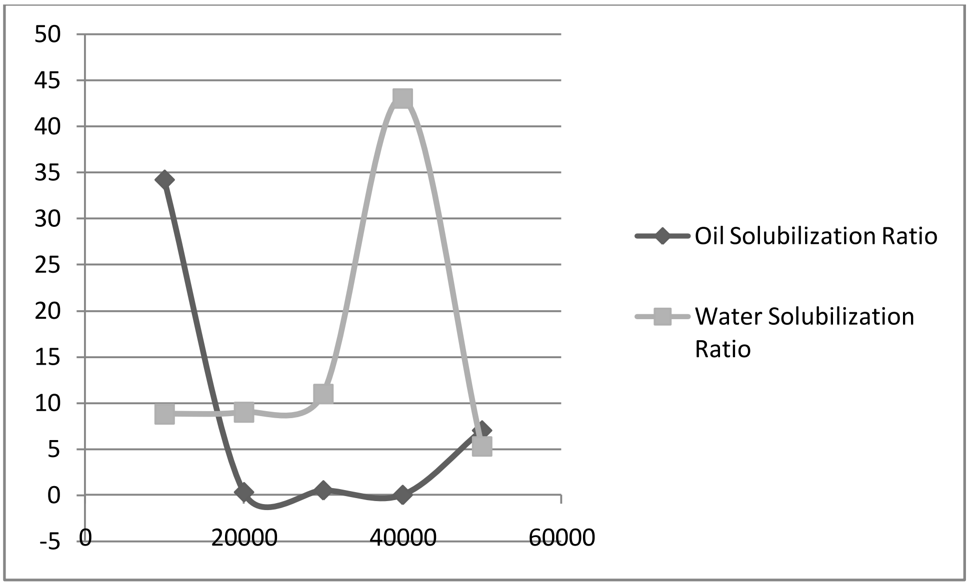
Graph d: Solubilization curve for difference salinity Sodium dodecyl sulfate (SDS) Day 1-3

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.42	1		4.3
20000	3.4	1	3.37	4.3
30000	3.2	1	3.15	4.3
40000		1		4.3
50000	3.77	1	3.07	4.3

Table i: Result on difference salinity using Sodium dodecyl sulfate (SDS) Day 4-5

Vo/Vs	**Vw/Vs**
34.2	8.8
0.3	9
0.5	11
0	43
7	5.3

Table j: Solubilization ratio for difference salinity using Sodium dodecyl sulfate (SDS) Day 4-5



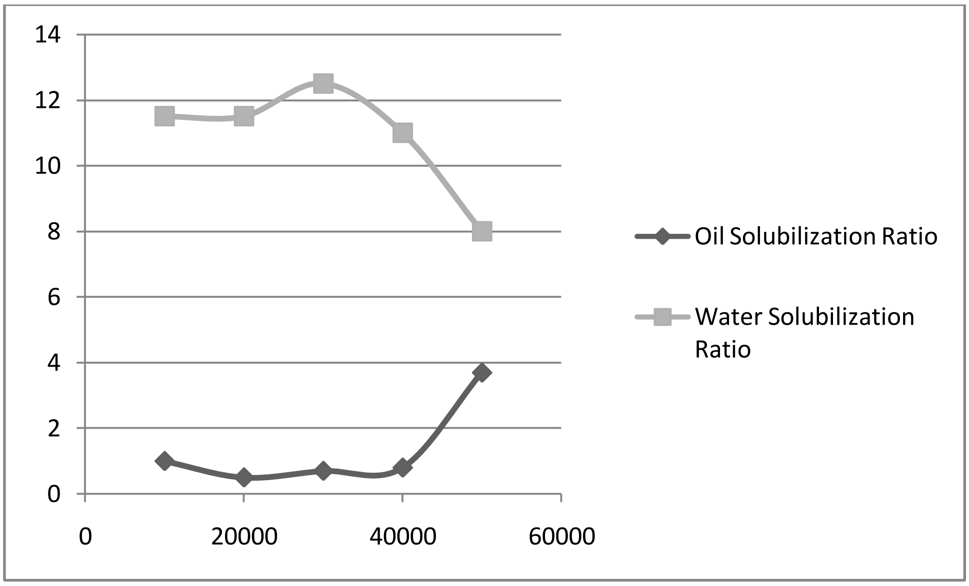
Graph e: Solubilization curve for difference salinity using Sodium dodecyl sulfate (SDS) Day 4-5

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.15	1	3.05	4.3
20000	3.15	1	3.1	4.3
30000	3.05	1	2.98	4.3
40000	3.2	1	3.12	4.3
50000	3.5	1	3.13	4.3

Table k: Result on difference salinity using Sodium dodecyl sulfate (SDS) Day 6-7

Vo/Vs	**Vw/Vs**
1	11.5
0.5	11.5
0.7	12.5
0.8	11
3.7	8

Table l: Solubilization ratio for difference salinity using Sodium dodecyl sulfate (SDS) Day 6-7



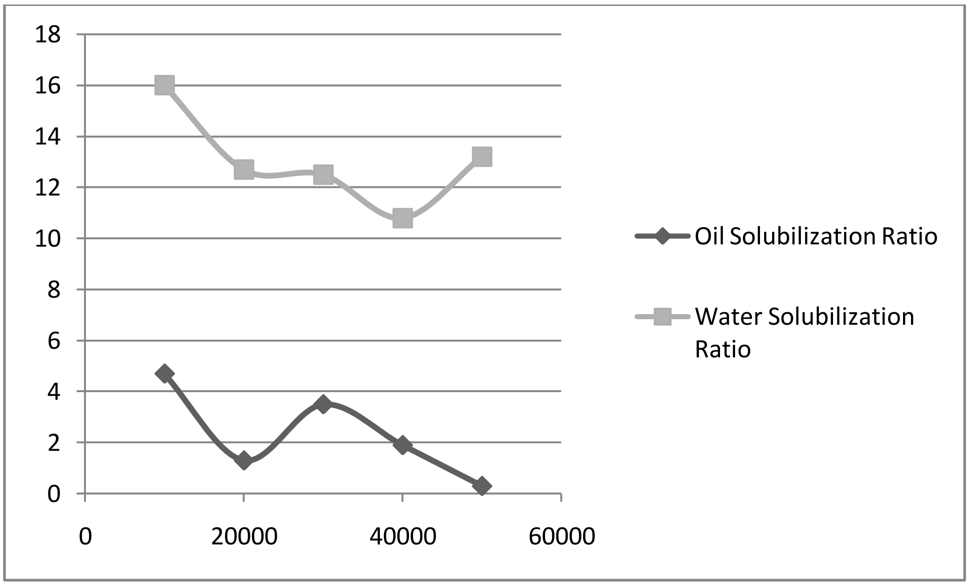
Graph f: Solubilization curve for difference salinity using Sodium dodecyl sulfate (SDS) Day 6-7

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	2.7	1	2.23	4.3
20000	3.03	1	2.9	4.3
30000	3.05	1	2.7	4.3
40000	3.22	1	3.03	4.3
50000	2.98	1	2.95	4.3

Table m: Result on difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 1-3)

Vo/Vs	**Vw/Vs**
4.7	16
1.3	12.7
3.5	12.5
1.9	10.8
0.3	13.2

Table n: Solubilization ratio for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 1-3)



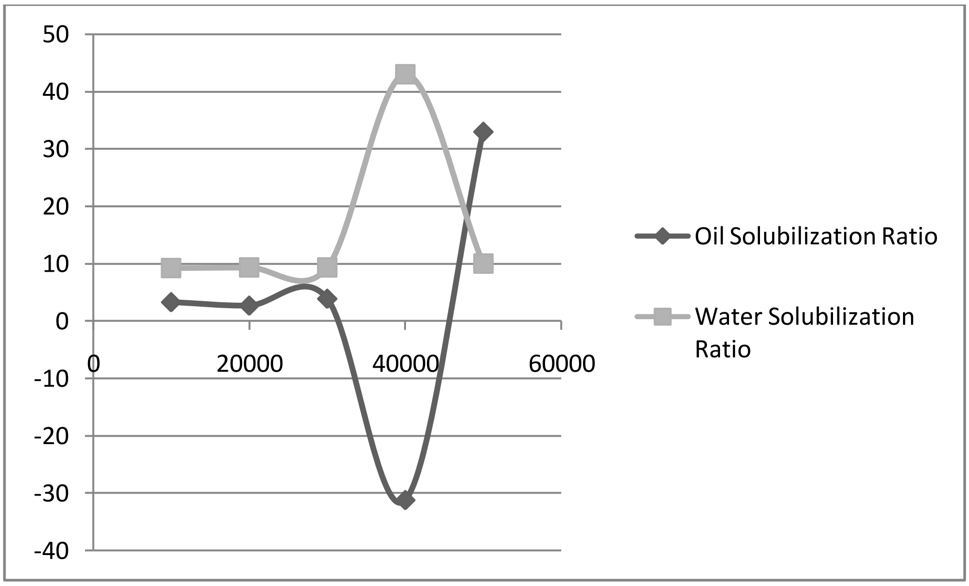
Graph g: Solubilization curve for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 1-3)

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.38	1	3.05	4.3
20000	3.37	1	3.1	4.3
30000	3.37	1	2.98	4.3
40000		1	3.12	4.3
50000	3.3	1		4.3

Table o: Result on difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 4-5)

Vo/Vs	**Vw/Vs**
3.3	9.2
2.7	9.3
3.9	9.3
-31.2	43
33	10

Table p: Solubilization ratio for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 4-5)



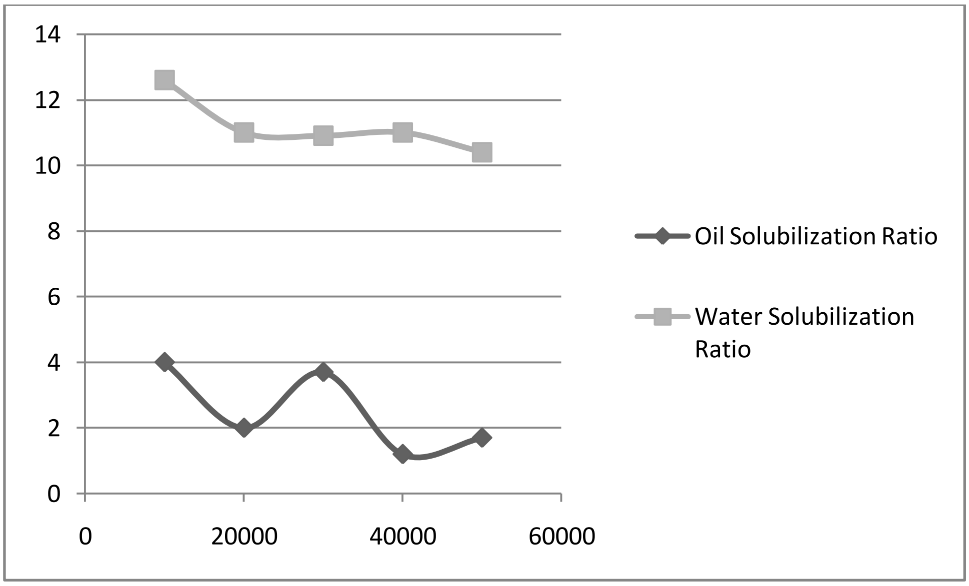
Graph h: Solubilization curve for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 4-5)

Salinity (PPM)	**Aqueous level**	**Oil Level**	**Top interface**	**Bottom interface**
10000	3.04	1	2.64	4.3
20000	3.2	1	3	4.3
30000	3.21	1	2.84	4.3
40000	3.2	1	3.08	4.3
50000	3.26	1	3.09	4.3

Table q: Result on difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 6-7)

Vo/Vs	**Vw/Vs**
4	12.6
2	11
3.7	10.9
1.2	11
1.7	10.4

Table r: Solubilization ratio for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 6-7)



Graph i: Solubilization curve for difference salinity using 3-(N, N-Dimethyl octadecylammonio) Propane sulfonate (Day 6-7)

APPENDIX 7: PICTURES OF EXPERIMENTS

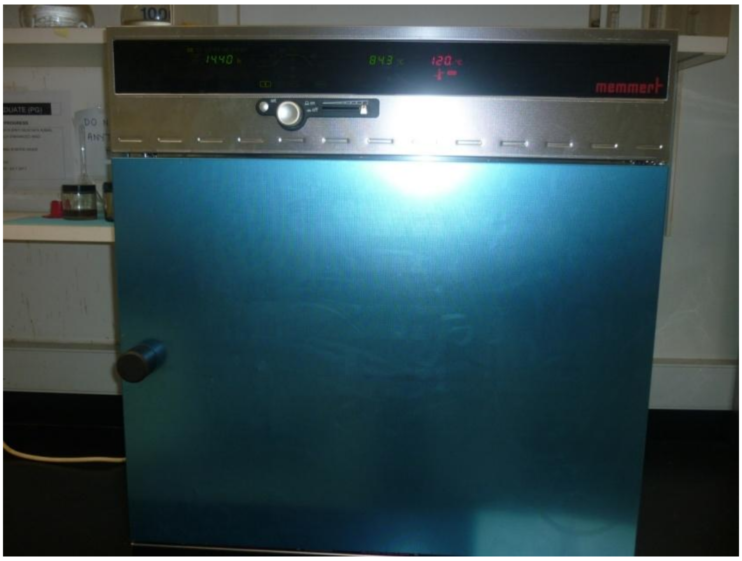
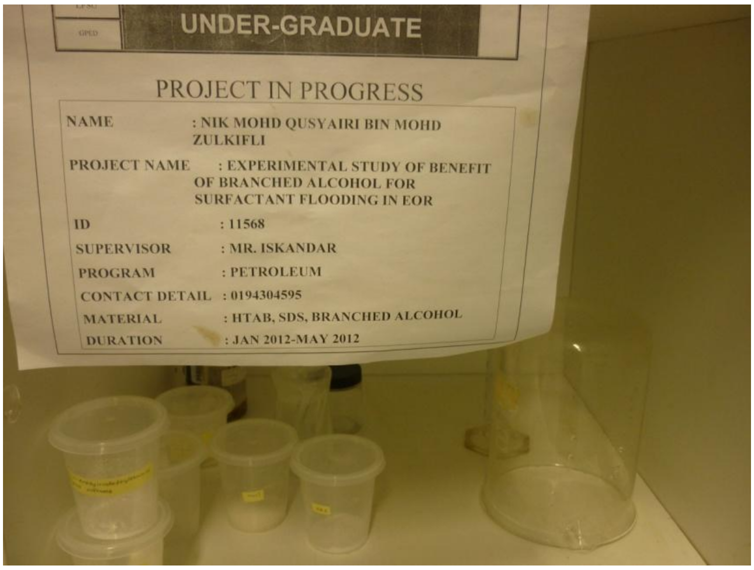


Figure A: Oven used to imitate the reservoir temperature at 70^{o} Celsius

Figure B: Storage of phase behavior experiment in the oven



UNDER-GRADUATE

PROJECT IN PROGRESS

NAME	: NIK MOHD QUSYAIRI BIN MOHD ZULKIFLI
PROJECT NAME	: EXPERIMENTAL STUDY OF BENEFIT OF BRANCHED ALCOHOL FOR SURFACTANT FLOODING IN EOR
ID	: 11568
SUPERVISOR	: MR. ISKANDAR
PROGRAM	: PETROLEUM
CONTACT DETAIL	: 0194304595
MATERIAL	: HTAB, SDS, BRANCHED ALCOHOL
DURATION	: JAN 2012-MAY 2012

Figure C: Workspace given by the lab technologist

Figure D: Densitometer used to measure the density of oil and phase formulation

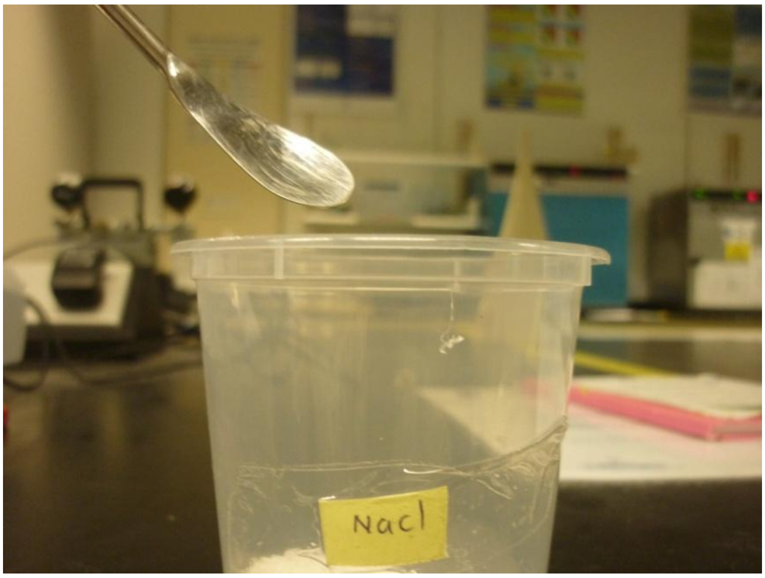


Figure E: Brine preparation using Sodium chloride (Nacl)

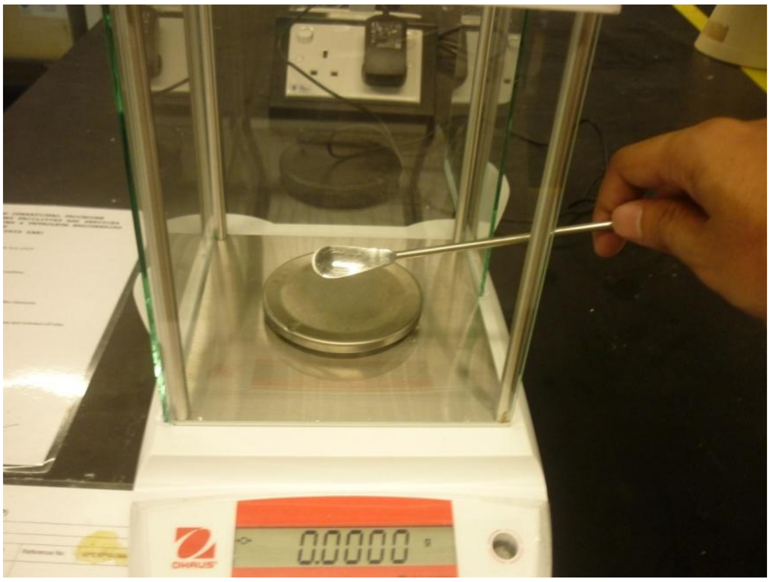


Figure F: Using high accuracy device to measure weight of chemical

Figure G: Waste is being properly disposed

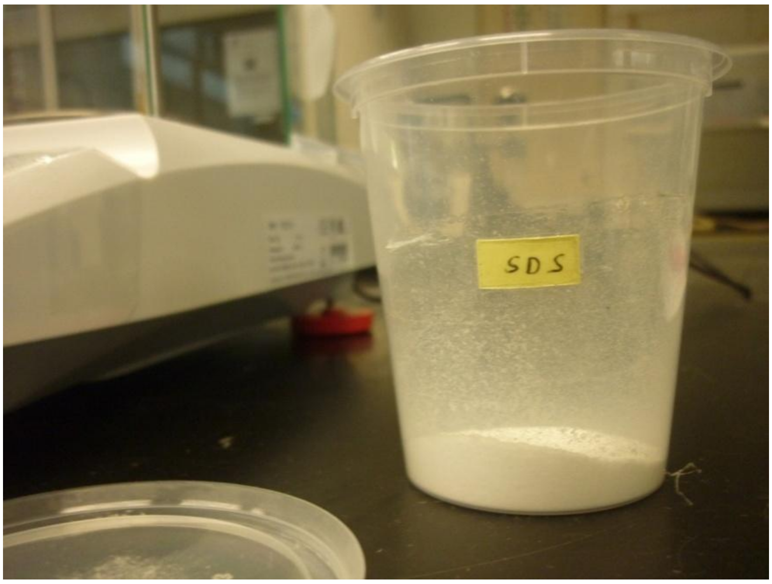


Figure H: Ionic surfactant used in phase behavior experiment: Sodium dodecyl sulfate (SDS)

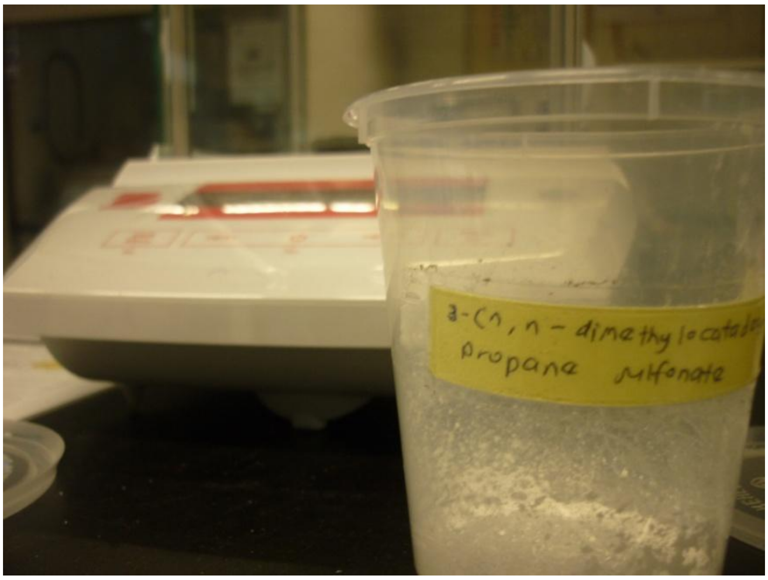


Figure I: Zwitterionic surfactant used in phase behavior experiment: 3-(n, n-dimethylocatadecylamminia) propane sulfonate

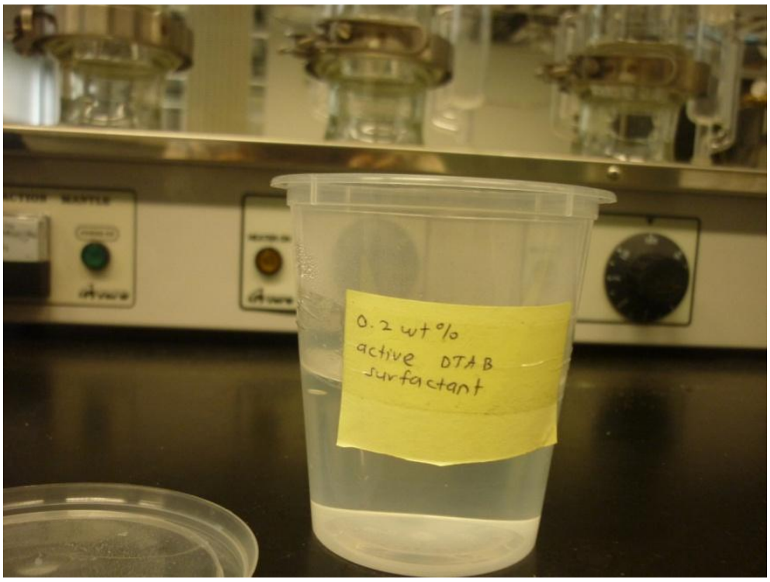


Figure J: Ionic surfactant used in phase behavior experiment: Dodecyl Trimethyl Ammonium Bromide (DTAB)

Figure K: Glass container used to store the branched alcohol of 2-methyl 1-butanol and LIAL 123

Figure L: Degreaser used to clean the pipettes before each experiment

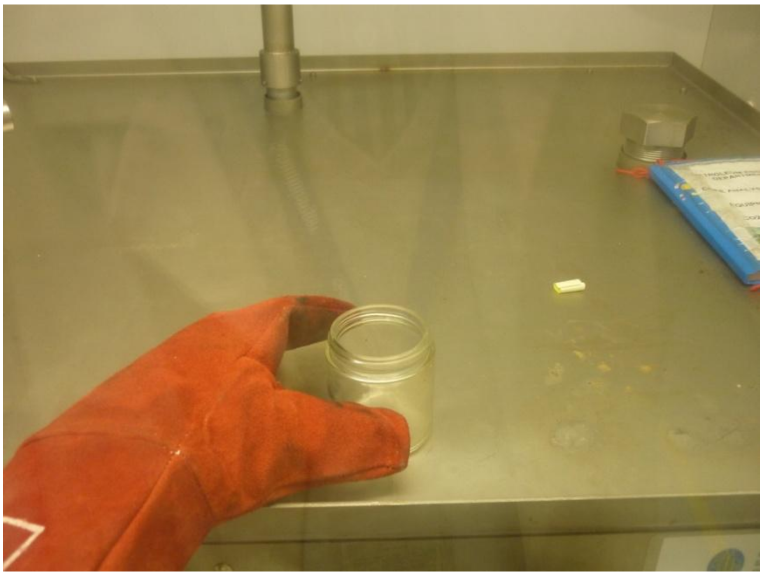


Figure M: Using proper apparatus to transfer chemical

Figure N: All chemical transfer is done in fume chamber in the lab