

CERTIFICATION OF APPROVAL

**SCREENING OF SURFACTANTS FOR MICELLAR FLOODING AND
FOAM FLOODING**

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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 the original work contained herein have not been undertaken or done by specified sources or persons.

Ong Chong Yew

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ABSTRACT

A great deal of information has been published on the use of surfactants in chemical Enhanced Oil Recovery methods as it holds particular attraction for recovering the residual oil left in the reservoir after water flooding. The application of surfactants leads to the mobility control mechanism in foam flooding whereas interfacial tension reduction is the important mechanism in micellar flooding. When foam flooding is conducted, some portion of the surfactant tends to adhere on the surface of the rocks due to the adsorption characteristic of the surfactants themselves. This will consequently result in less amount of surfactant to be injected for the micellar flooding as remaining surfactant down hole will react with the injected solution to form microemulsion.

Hence, this study aims to screen the best surfactant which can be performing in both foam flooding and micellar flooding by investigating the surfactant behaviour in foam and microemulsion. Foam stability is the main concern in foam displacement as the foam is needed to be stable to improve the sweep efficiency of the driving fluid whereas Type-III microemulsion had been proven in previous work as the most suitable for achieving ultra-low interfacial tension.

Individual laboratory experiments were conducted to investigate the behaviour of the surfactant in foam stability, adsorption characteristic on sandstone and the ability to form Type III microemulsion. The experiments were conducted by varying the concentration of the surfactants. Two surfactants which were able to perform well in the experiments were then further analysed by repeating the experiment in a continuous way where the same solutions were being tested one after another experiment. The surfactants examined were Triton X-100, Adogen 464, Niaproof 4 and Triton QS-15.

Under the limited condition of the conducted experiments, Triton QS-15 and Triton X-100 showed a good result which were able to have a good foam stability and able to generate Type-III microemulsion. However, Triton QS-15 is more preferable and outstands as a whole because it showed high adsorption behaviour and high volume of Type-III microemulsion to form with only low concentration of surfactant.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The declining trend of new findings in line with the high demand for energy, industry has been focused on the importance of enhanced oil recovery (EOR) processes in low recovery efficiency reservoir. Ultimately, overall oil displacement efficiency is the main goal or target of EOR processes which can be viewed into two different scope of displacement including microscopic and macroscopic. Microscopic efficiency is defined as the oil displacement or mobilization at the pore scale and the efficiency is measure in term of the ability of displacing fluid in sweeping the oil at those places (Green & Willllhite, 1998). For instance, microscopic efficiency is inversely proportional to the two factors which are oil viscosity and capillary forces or more likely refer to the interfacial tension (IFT) between the displacing fluid and oil (Satter et al., 2008). In the other hand, macroscopic displacement efficiency is referring to the effectiveness of the displacing fluid in contacting the reservoir in a volumetric sense.

Capillary forces which present in the reservoir are responsible for entrapping a large amount of oil during water flooding of petroleum reservoir. Foam flooding was introduced as an improvement of the gas injection processes which use to control the highly mobile gas and thus avoid the forming of gas channels upon contact with the more viscous oil. The injection of foam into a porous medium takes place at a lower mobility ratio than that of gas or liquid injection alone and a large number of resilient interfaces are created which tend to exert a piston-like force on the oil to be displaced (Latil, 1980). Surfactants are required in foam flooding to form bubbles and stabilized the generated foam, thus the selection of right surfactants need to be highly concerned as it may affect the foam stability.

The ultralow IFT plays a dominant role in the capillary forces which can be achieved by surfactant flooding but hurdle in the implementation of this method is the high cost of chemicals. Hence, micellar flooding which also known as microemulsion refer to the injection of a chemical mixture that contains water, a suitable hydrocarbon, and surfactant and a suitable alcohol. A microemulsion is a thermodynamically stable

dispersion of oil and water which contains substantial amounts of oil and water stabilized by surfactant. The IFT between microemulsion and excess phase can be extremely low (Green and Willhite, 1998; Flaaten et al., 2008; Sheng, 2010). Winsor (1954) characterized the microemulsion into three types including Type I (lower phase), Type II (Upper phase) and Type III (middle phase). Type I is defined as oil in water where microemulsion forms at low salinity by the solubilisation of some amount of oil in aqueous phase. In contrast, Type II is water in oil microemulsion which forms at high salinity by solubilisation of some amount of water in oil (Healy et al., 1976; Flaaten et al., 2010). In the other hand, Type III microemulsion begins to form at intermediate optimal salinity which has been found quite suitable for reducing residual oil saturation due to its unique properties such as ultralow IFT, large interfacial area, thermodynamically stability and the ability to solubilize both oil and water (Healy et al., 1976; Nelson and Pope, 1978; Pope et al., 1982; Flaaten et al., 2008). Recovery efficiency in surfactant EOR highly depends on the degree of surfactant retention. Surfactant retention reduces concentration of surfactant in the injected surfactant slug and results in less oil recovery. Loss of surfactant in the reservoir takes place due to several mechanisms such as surfactant adsorption, precipitation, degradation and partitioning (Donaldson et al., 1989).

Surfactant is a group of chemicals, including soap and soap like substances that have hydrophobic and hydrophilic tails that alter the surface activity of aqueous media. Moreover, surfactants which made of oil-soluble and water soluble groups plays an important role in enhanced oil recovery by lowering the interfacial tension between two immiscible liquids and thus improve the displacement efficiency. During a waterflood where oil can be produced until the residual waterflood oil saturation S_{or} is achieved, the capillary number N_{cap} is around $10^{-8} - 10^{-7}$ and has to be increased by an order of at least two or three magnitudes (Abrams, 1975; Green and Willhite, 1998) to allow additional oil recovery. In chemical flooding, development of surfactant system in today emerging trends are required to have the capabilities on reducing the IFT between oil and water to ultra-low values in the range of 0.01-0.001 dyne/cm in order to significantly mobilize residual oil and thus allowing both oil and water flow as continuous phase.

1.2 Problem Statement

Capillary forces or the interfacial tension between the oil and the displacing fluid is affecting the displacement sweep efficiency for micellar flooding. In foam flooding, the ability of the surfactant in improving mobility control of the foam significantly affects the volumetric sweep efficiency. When foam flooding is conducted, some portion of the surfactant tends to adhere on the surface on the rocks due to surfactant adsorption behaviour. Thus, the remaining surfactant down hole is able to react again with the following injected solution to form micellar solution. Hence, surfactant which is good at both foaming and reducing IFT is needed for the proposed EOR methods.

All prior research has focused on examining the effect of formulation based parameters such as surfactant type, concentration, generation pressure and viscosity in improving the recovery through only one type of chemical EOR method. In order to find the suitable surfactant to perform both flooding, there are some problems which lead to this project investigation:

- What are the types of surfactants which can be performing well in foam flooding micellar flooding?
- How is the adsorption behaviours of the surfactants when propagate within the pore space of the rocks affect the efficiency of the flooding?
- Which surfactant is able to show a good result when both experiments are conducted one after another by considering the adsorption behaviour?

1.3 Objectives

- To identify the behaviour of different classes of surfactants in
 - a) Foam stability
 - This is essential during the foam flooding because the stability of the foam is able to ease the propagation of the driving fluid through the formation.
 - b) Adsorption behaviour onto the sandstone
 - Some portion of the surfactant tends to adhere on the surface of the rocks during foam flooding and thus the adhered surfactants can be used to form micellar solution on the following flooding.
 - c) Ability to form Type III microemulsion
 - This bi-continuous phase of microemulsion which is equilibrium with upper excess oil and lower excess water is proven to be effectively reducing the interfacial tension of oil and displacing fluid.
- To suggest the surfactant that is suitable for the proposed floodings.

1.4 Scope of Study

- i. Research and literature review on the properties and characteristics of the surfactant and area of application in enhanced oil recovery.
- ii. Surfactants examined in this project are Triton X-100, Adogen 464, Niaproof 4 and Triton QS-15 with the concentration limit from 0.1wt% to 0.5wt% for foam stability experiment whereas 1wt% to 4wt% concentration for micellar solution.
- iii. Isopropyl Alcohol is used as the solvent in forming the microemulsion for the experiments with the concentration of 6.0wt% for all the micellar solution.
- iv. All experiments are carried out at ambient condition.
- v. The results are based on the outcome of static behaviour experiments instead of the core flooding experiments.

CHAPTER 2

LITERATURE REVIEW

2.1 Enhanced Oil Recovery

Oil production can be divided into three stages including primary, secondary and tertiary oil production. Primary recovery mechanism is defined as the production assisted by the natural flow of the reservoir as well as the artificial lift (Lake, 1989). Factors such as reservoir rock and fluid properties, and geological heterogeneities are influencing the primary recovery; worldwide basis shows that the range from 20% and 40% is the most common primary oil recovery factor which approximately equivalent to an average around 34%, and thus the remainder of hydrocarbon is still unrecoverable (Satter et al., 2008). Secondary recovery mechanism which likely referring to immiscible processes of water flooding and gas injection or gas-water combination floods are aimed to introduce artificial energy into the reservoir for pressure maintenance purpose. However, water definitely becomes the most common fluid injected due to its availability, high specific gravity and economical (Dake, 1978;; Satter et al., 2008). Lake (1989) mentioned that the injection of fluids and energy not normally found in the reservoir is considered as tertiary recovery mechanism or known as Enhanced Oil Recovery (EOR). The injected fluids must have the ability to enhance the natural energy in the reservoir and react with reservoir rock or oil system to create situation which favourable for residual oil recovery (Green & Willhite, 1998). This included:

- Increment in capillary number
- Increment in drive water viscosity
- Provide mobility control
- Alteration of the reservoir rock wettability
- Minimization of the IFT between the displacing fluid and oil

2.2 Foam flooding

The application of foam flooding in EOR was studied extensively by many researchers such as Schramm, Falls, Rossen and many more. Foam is defined as multi-phase mixture of gas and liquid that exhibits a unique set of properties which make them suitable for a large number of applications (Stevenson, 2012). Foam is a metastable

dispersion of a relatively large volume of gas in a continuous liquid phase that constitutes a relatively small volume of the foam (Sydansk & Romero-Zeron, 2011). The foam properties which are most notable including:

- High specific surface area
- Finite yield stress which enables it to support large finite shear stress before exhibiting strain
- Large expansion ratio that is attained through the high liquid to gas volume ratio

The injection of foam into a porous medium takes place at a lower mobility ratio than that of gas or liquid injection alone and a large number of resilient interfaces are created which tend to exert a piston-like force on the oil to be displaced (Latil, 1980). Foam exhibits higher apparent viscosity and lower mobility within a formation than its separate constituents. This lower mobility may often be achieved by the inclusion of less than 0.1% surfactant in the liquid phase of the foam (Craig, 1965). In oilfield application, Hirasaki et al. (2011) reported that foams play an important role as mobility control agent in surfactant flooding, especially at high temperature, in few combination flooding including alkaline-surfactant flooding and surfactant/polymer projects.

2.3 Foam Stability

In fact, surfactants are required in foam flooding to form bubbles and stabilize the generated foam, thus the selection of right surfactants need to be highly concerned as it may affect the foam stability. Some of the early developments in the field of foam stability were derived from early studies made on the stability of oil water emulsions (Iglesias et al, 1995). Foam stability in the presence of oil can be better understood by looking at the early studies on emulsions. Generally there are four theories approximating the behaviour of foams in the presence of oil. First there is the “spreading coefficients” which assumes that the surface tension between the surfactant solution and oil phase can be described by a specific coefficient which is called as spreading coefficient. The value of that coefficient depends on the stability of the mixture. The second theory is the lamellae number theory which correlates the lamellae number less than 1 is called class A foams and it was found to be the most stable with results conforming to those obtained from previous theories. The third theory is the pseudo

emulsion film theory which suggests that the stability of the water film separating the gas and oil, is directly responsible for the stability of the foam structure, because by being stable it isolates the oil at the lamellae. Lastly, the bridging theory which regards oil as an antifoaming agent, and hence it describes oil content as a function of stability.

2.4 Micellar Flooding

Micellar flooding refers to the injection of a chemical mixture that contains water, a suitable hydrocarbon, and surfactant, together with smaller quantities of a salt and a suitable alcohol. Latil (1980) reported that these fluids are possible to pass continuously from the case of a micro-emulsion in which oil is the continuous phase to one in which water is the continuous phase and thus a true miscible displacement of oil by water may be possible. For a successful displacement process, the range of 0.001 to 0.01mN/m which is considered as ultralow IFT must be achieved by the injected surfactant slug in order to sweep the residual oil and generate an oil bank where continuous phases can be occurred for the flow of both oil and water (Hirasaki et al., 2011). Satter et al. (2008) highlighted some problems which limit the widely application and effectiveness of micellar flooding which including:

- Large adsorption of surfactants onto rock surfaces.
- Surfactants are very expensive chemical
- Large volume of surfactant are needed to reach ultralow values of interfacial tension between water and oil
- Degradation of the chemical mixture at high temperature

2.5 Microemulsion

Microemulsions or swollen micellar systems represent an intermediate state between micellar solutions and true emulsions as shown in the hypothetical phase diagram:

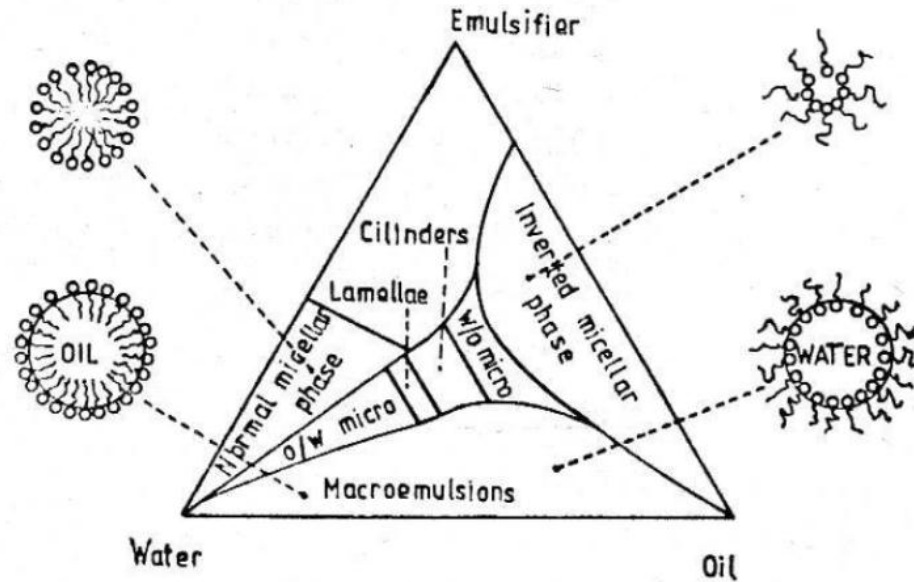


Figure 1: Hypothetical Phase Equilibria Diagram Showing Regions of Microemulsions and Micellar Solutions.

Formation of microemulsions is encouraged by the addition of a co-surfactant such as alcohol possibly because of the geometric requirements for appropriate curvature in the interfacial region. Once the conditions are right, spontaneous formation occurs. As for simple aqueous systems, microemulsion formation is dependent on surfactant type and structure. If the surfactant is ionic and contains a single hydrocarbon chain (e.g., sodium dodecylsulphate, SDS) microemulsions are only formed if a co-surfactant (e.g., a medium size aliphatic alcohol) and/ or electrolyte (e.g., 0.2M NaCl) is also present. With double chain ionic and some non-ionic surfactants a co-surfactant is not necessary. This results from one of the most fundamental properties of microemulsions that are an ultra-low interfacial tension between the oil and water phases. A well-known classification of microemulsions is that of Winsor who identified four general types of phase equilibria:

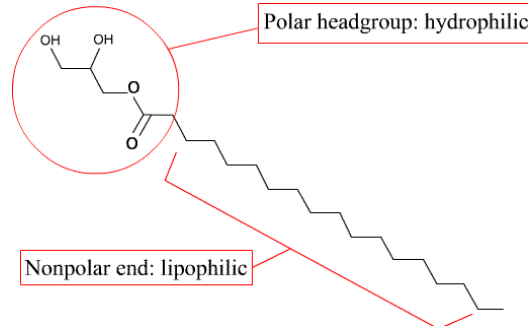
- Type I: the surfactant is preferentially soluble in water and oil-in-water (o/w) microemulsions form (Winsor I). The surfactant-rich water phase coexists with the oil phase where surfactant is only present as monomers at small concentration.
- Type II: the surfactant is mainly in the oil phase and water-in-oil (w/o) microemulsions form. The surfactant-rich oil phase coexists with the surfactant-poor aqueous phase.
- Type III: a three-phase system where a surfactant-rich middle-phase coexists with both excess water and oil surfactant-poor phases.
- Type IV: a single-phase (isotropic) micellar solution, that forms upon addition of a sufficient quantity of amphiphile (surfactant plus alcohol).

2.6 Surfactant

The involvement of surfactant in Enhanced Oil Recovery has been investigated and studied for many years and yet, a lot of experiments are still being conducted for the advancement and improvement of the technique. Surfactant generally is a group of chemical that contained a hydrophilic head, a hydrophobic hydrocarbon tail and possible intermediate neutral groups. Varieties of the groups are common in surfactant design and certain structures of surfactants have been proven can increase oil recovery performance (Green & Willhite, 1998). Types of surfactant are based on the characteristics of the head group:

- Anionic: Negative charge on the head groups
- Cationic: Positive charge on the head groups
- Non-ionic: Does not ionize, head group is larger than tail group
- Zwitterionic: Surfactant contains two groups of opposite charge

Figure 1: Schematic of surface-active molecule: Glycerol Monostearate



Latil (1980) mentioned that petroleum sulphonates derived from crude oil is the surfactant which being used to date due to reasons: economical, easily obtainable and effective in attaining low IFT. In chemical flooding, surfactant is used to displace residual oil that is trapped by high capillary forces within the porous media but the elimination or reduction in capillary force and the increase in fluid flow viscous flow are able to improve the displacement efficiency. In order to be considered as successful in chemical flooding, the surfactant must has the ability to reduce the interfacial tension between oil and aqueous phase to ultralow values. In general surfactants do not exceed 0.17 of the constituting solution composition. One of the key challenges is to identify the optimal surfactant type and concentration needed for creating stable foam, in the presence of electrolytes, brine salinity and oil emulsifying effects. The surfactant structure effect on foam performance was also investigated by examining 40 surfactant types, it was found that parameters such as hydrophole size and chain length had the greatest effect on foam stability (Borchardt et al, 1985).

2.7 Several Definitions and Concepts

Below are definitions of some terms that may have been used in the project.

2.7.1 Interfacial Tension (IFT)

Healy et al. (1976) reported that a huge number of anionic surfactant system showed good correlation between IFT and solubilization parameter. A theoretical relationship between the solubilization parameter and IFT for a middle phase micro-emulsion is developed (Huh, 1979):

$$\sigma_{mw} = \frac{C_{Hw}}{(V_{wm}/V_{sm})^2} \quad , \quad \sigma_{mo} = \frac{C_{Ho}}{(V_{om}/V_{sm})^2}$$

where V_{wm} , V_{sm} , V_{om} are the volume of surfactant, volume of water, and volume of oil in the microemulsion phase, C_{Hw} and C_{Ho} are the empirical constants determined experimentally, mN/m which normally ranges from 0.1 to 0.35 and a typical value may be 0.3 if experiment data are available.

2.7.2 Mobility Ratio

Mobility ratio is defined as the ratio mobility of displacing phase divided by the mobility of displaced phase (Chon & Yu, 2001). Cheek and Donald (1955) studied the effect of mobility ratio on the areal sweep efficiency. Mobility ratio is able to identify the stability of a displacement process, for instance when $M > 1.0$, it indicates that the flow becoming unstable with non-uniform displacement front or viscous fingering. The condition which leads to the water fingering towards the more viscous oil is during the huge viscosity different between the displacing fluid and the displaced fluid and thus will reduce the oil recovery efficiency.

2.7.3 Capillary Number

Capillary number is a dimensionless grouping of parameters which expressing the ratio of viscous to capillary (interfacial) forces as follows:

$$N_c = \frac{\text{viscous forces}}{\text{capillary forces}} = \frac{v\mu}{\sigma}$$

Where v refers to the pore flow velocity of the displacing fluid in their derivation, μ means the viscosity of the displacing fluid, and σ_{ow} is defined as the IFT between the displacing and the displaced phases. Sydansk and Romero-Zeron (2011) suggested that the application of surfactant or alkaline flooding is a significant alternative to increase the capillary number.

2.7.4 Surfactant Adsorption

Surfactant retention is caused by several mechanisms such as surfactant adsorption, precipitation of surfactant in the presence of divalent ions, diffusion of surfactant into dead-end pores and surfactant partitioning into the oil phase. These mechanisms all result in retention and deterioration of the composition of the chemical slug, leading to poor displacement efficiency. Among them, surfactant adsorption is the major factor that controls the quality of surfactant loss into a porous medium. In the most common form,

surfactants consist of a hydrocarbon tail and a polar head. The mechanism responsible for the surfactant adsorption is mainly the electrostatic attraction between the charged surface of the solid and the charged head group of the surfactant molecule. This is a process of transfer of surfactant molecules from the bulk solution phase to the surface interface. The mechanisms by which surface-active molecules may adsorb onto the solid substrates from an aqueous solution have been well identified, and in general involve single ions rather than micelles of ion exchange, ion pairing, hydrophobic bonding, and dispersion forces (Somasundaran et al., 2000; Santamu et al., 2006; Zhang et al., 2006).

CHAPTER 3

METHODOLOGY

3.1 Methodology Outline

Three individual experiments were conducted to investigate the surfactant characteristics including foam stability, adsorption behaviour and the microemulsion phase behaviour study. Based on the result, the surfactants which performed well were then further analysed and investigated through the continuous experiment in order to select the most suitable surfactant for foam flooding and then followed by micellar flooding.

In foam stability experiment, 3 different concentrations which were 0.1wt%, 0.3wt% and 0.5wt% of each surfactant were used to identify the surfactant which is good in foaming. Evaluation parameters for this experiment were calculated in term of the half time and the foam height changes in 20 minutes. The decaying process was monitored by plotting the foam height against time. The half time is defined as the time needed for the foam to dewater of half of its initial height. This experiment aim to find out the surfactant which was able to generate foam and meantime the foam was able to have a good stability.

For the adsorption behaviour experiment, the calibration graph of refractive index against the concentration of the surfactant was needed to obtain the pattern of the behaviour. Refractive Index is defined as the measure of the bending of a ray of light when passing from one medium into another. Theoretically, the refractive index will increase with the increase of concentration. 1wt% concentration of each surfactant was prepared and left for equilibration for one week after mixing with the sandstone powder. The equilibrium surfactant concentration was analysed in order to determine the amount of chemical absorbed.

Next, the phase behaviour study was conducted to investigate the ability of the surfactants to form Type-III microemulsion. The concentration of the solvent is constant whereas the concentration of surfactant varied at 1wt%, 2wt%, 3wt% and 4wt%. The volume of Type-III microemulsion was monitored and graphs of volume against time were plotted in order to find the equilibrium time.

The surfactants which showed good result in both foam stability and microemulsion phase behaviour experiments were then further investigate through continuous experiments by considering the adsorption behaviour of the surfactant.

3.2 Materials and apparatus:

- Surfactants
 - a) Triton X-100 (Non-Ionic Type)
 - b) Adogen 464 (Cationic Type)
 - c) Niaproof 4 (Anionic Type)
 - d) Triton QS-15 (Anionic Type)
- Sodium chloride
- Crude oil
- Isopropyl alcohol
- Berea sandstone
- Mechanical stirrer
- Magnetic Stirrer
- Refractometer
- Glassware

3.3 Procedures

3.3.1 Foam Stability Experiment

Surfactant solution: All types of surfactant were used with varies concentration at 0.1wt%, 0.3wt% and 0.5wt%. The solutions were prepared at temperature of 23 °C and atmospheric pressure. Salinity of 10gram/Litre was made by dissolving 10grams of sodium chloride in the deionised water. The dissolution process was accelerated by using a standard laboratory stirrer without heating.

Foam generation: Foam was generated by stirring the surfactant solution for a certain time. All the runs had the same generation procedure, with differences in the surfactant concentration. The solutions were stirred for 180 seconds with 60 seconds intervals, followed by 10 seconds short stops. The foamability of the foam was measured by the initial volume of the generated foam. After that the decaying process was monitored by

plotting the foam height against time. The half time needed for dewatering of half of the initial height was recorded as an evaluation parameter for the foam stability. Three types of measurement were monitored including:

- Initial foam height
- Foam height against time
- Half time

3.3.2 Surfactant Adsorption Experiment

Surfactant solution: All types of surfactant were used with varies concentration from 0.1wt% to 1wt% in order to construct the calibration curve of refractive index value against the concentration. The surfactant solution with 1wt% was then prepared again to show the adsorption characteristic onto sandstone. The solutions were prepared at temperature of 23 °C and atmospheric pressure. Salinity of 10gram/Litre was made by dissolving 10grams of sodium chloride in the deionized water. The dissolution process was accelerated by using a standard laboratory stirrer without heating.

Adsorption investigation: Berea sandstones powders are heated in an oven at 120Deg for 2 hours in order to remove water and any other absorbed materials. Sandstones samples and surfactant solutions are mixed at 1:20 mass ratio in a beaker and then stirred. They were then left for equilibration for a minimum of 3 days at ambient condition. The beaker is next centrifuged to separate the solution and sandstone. The new refractive index was measured and compared to the calibration graph in order to determine the amount of chemical absorbed.

3.3.3 Microemulsion Phase Behaviour Experiment

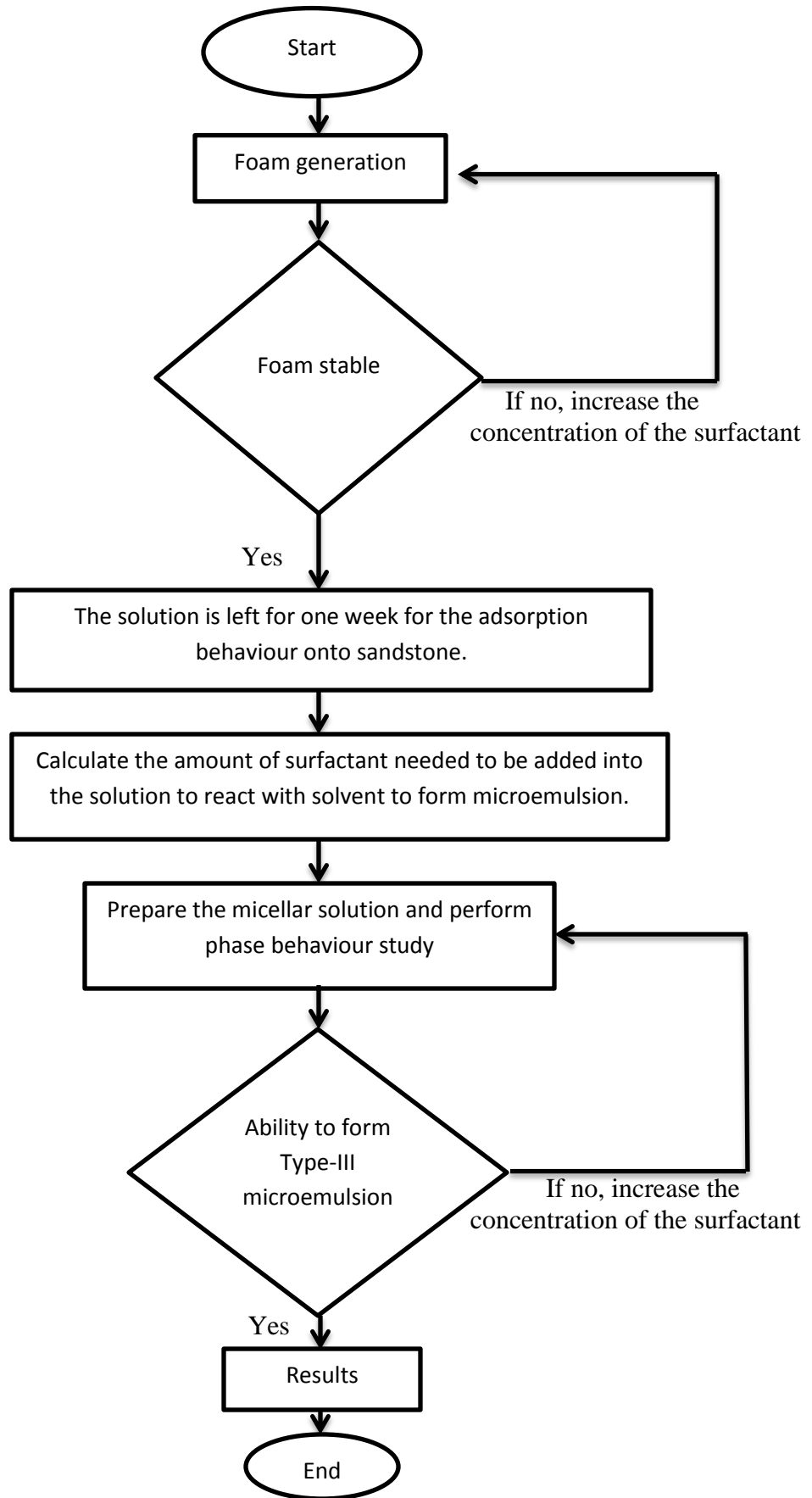
Surfactant solution: Chemical mixtures containing surfactant, solvent and brine were tested and optimised in this experiment. All types of surfactant were used with varies concentration at 1wt%, 2wt%, 3wt% and 4wt% were prepared. The micellar solutions were then formed by mixing with Isopropyl alcohol with constant concentration at 6wt%. The solutions were prepared at temperature of 23 °C and atmospheric pressure. Salinity of 10gram/Litre was made by dissolving 10grams of sodium chloride in the deionized

water. The dissolution process was accelerated by using a standard laboratory stirrer without heating.

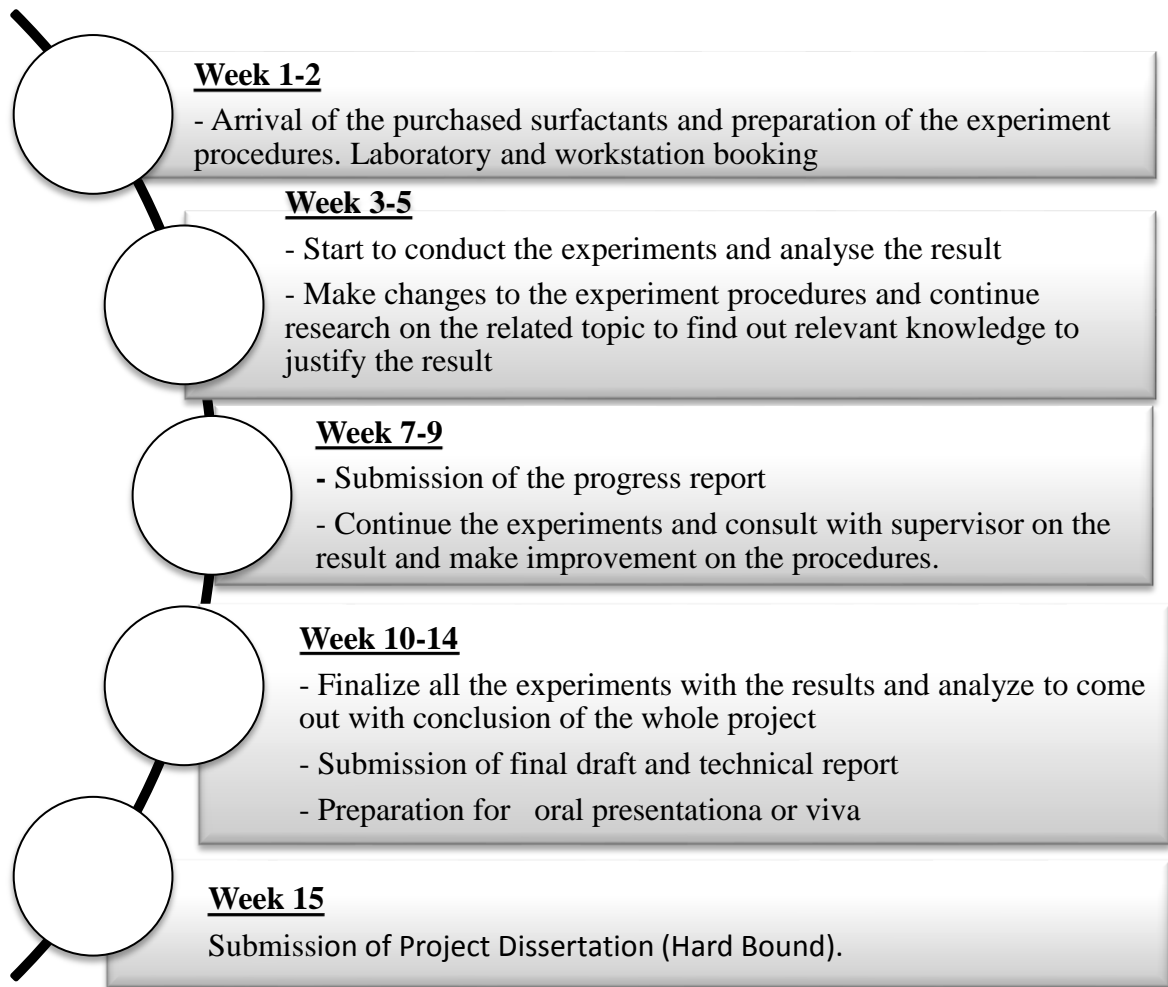
Phase behaviour: Each phase behaviour experiment used an array of burette with incremental surfactant concentration to obtain Type-III microemulsion data versus equilibrium time. Aqueous phases prepared for phase behaviour experiments contain surfactant, solvent and electrolyte. Equal volume of aqueous phase and oil phase were added in burettes and burettes were heated-sealed, cooled and slowly inverted several times to allow oil and aqueous phase mixing and then allowed it to settle for few hours till equilibrium time. The fluids in the phase behaviour array were visually inspected and the equilibrium time of Type-III microemulsion was recorded by noting volume change from time to time.

3.3.4 Continuous Experiment

The surfactants that able to perform well in both foam stability and microemulsion phase behaviour experiment were further investigated in a continuous way by considering the adsorption behaviour in order to suggest the most suitable surfactant which able to be applied in both foam flooding and followed by micellar flooding.



3.4 Key Milestones



3.5 Gantt Chart

No.	Details/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work Continues	█	█	█	█	█	█									
2	Submission of Progress Report							█								
3	Project Work Continues								█							
4	Pre-SEDEX									█						
5	Submission of Draft Report										█	█				
6	Submission of Dissertation (soft bound)												█			
7	Submission of Technical Paper													█		
8	Oral Presentation														█	
9	Submission of Project Dissertation (hard bound)															█

CHAPTER 4

RESULTS AND DISCUSSIONS

The results and discussion were based on the proposed flooding methods where foam flooding was conducted first followed by micellar flooding. Thus, three main individual parts were discussed earlier including the investigations on the surfactant behaviour in foam stability, adsorption onto Berea sandstone and microemulsion phase behaviour study. Each of these parts is further divided based on the types of surfactant. This consequent to the result of continuous experiment where the surfactants which performed well in the individual experiments were then further discussed and analysed to suggest the best performing surfactant in foam flooding and micellar flooding.

4.1 Foam Stability of the Surfactant

The two evaluation criteria used to access foam stability are half time value and initial foam height (foamability). The foamability of the foam was measured by the initial volume of the generated foam. After that the decaying process was monitored by plotting the foam height against time. In the other hand, half time was measured on the time needed for the generated foam to dewater of half of the initial height. The stirring period for all the surfactant is the same which is 180 seconds and each surfactant are tested with 3 different concentrations in order to have more accurate and consistent behaviour result.

Table 1: The Changes of Foam Height In 20 Minutes for All the Surfactants In Three Different Concentrations

Types of Surfactant	Surfactant Name	Surfactant Concentration (wt%)	Foam Height (cm) at Different Time Intervals (minutes)										
			0	2	4	6	8	10	12	14	16	18	20
Non-Ionic	Triton X-100	0.1	5.40	4.86	4.32	3.60	3.24	2.70	2.52	2.16	1.80	1.62	1.44
		0.3	5.40	5.40	5.22	5.22	5.22	5.22	5.04	5.04	5.04	4.68	4.50
		0.5	5.40	5.40	5.40	5.22	5.22	5.22	5.22	5.22	5.04	5.04	4.86
Cationic	Adogen 464	0.1	No Foam										
		0.3											
		0.5											
Anionic	Niaproof 4	0.1	5.40	4.50	2.16	0.90	0.18	0.09	0.00	0.00	0.00	0.00	0.00
		0.3	5.58	5.40	4.50	3.42	1.80	1.44	0.90	0.54	0.36	0.18	0.09
		0.5	5.58	5.22	4.50	3.60	2.70	1.80	1.44	0.90	0.90	0.72	0.54
	Triton QS-15	0.1	4.50	4.14	2.70	1.80	1.08	0.90	0.72	0.72	0.54	0.54	0.54
		0.3	5.04	4.50	3.42	1.80	1.26	1.08	0.90	0.90	0.90	0.90	0.90
		0.5	5.04	4.50	4.14	3.06	2.16	1.26	0.90	0.90	0.90	0.90	0.90

The results showed that all the surfactants were able to generate foam once being stirred except Adogen 464 which was from Cationic group of surfactant. Although Cationic surfactant was claimed to be infrequently used in oilfield foam due to the strong tendency in absorbing on the surface of clays and sand, but the result was contradicting as the surfactant solutions turned into cloudy water only for all the concentrations tested. For Adogen 464, it is clearly know that this group of surfactant does not have the behaviour in satisfying the foaming agent as it does not generate foam regardless its concentration In the other hand, other surfactants were easily to generate foam and the height of the foam increased along with the stirring period.

4.1.1 Foam Decline Pattern

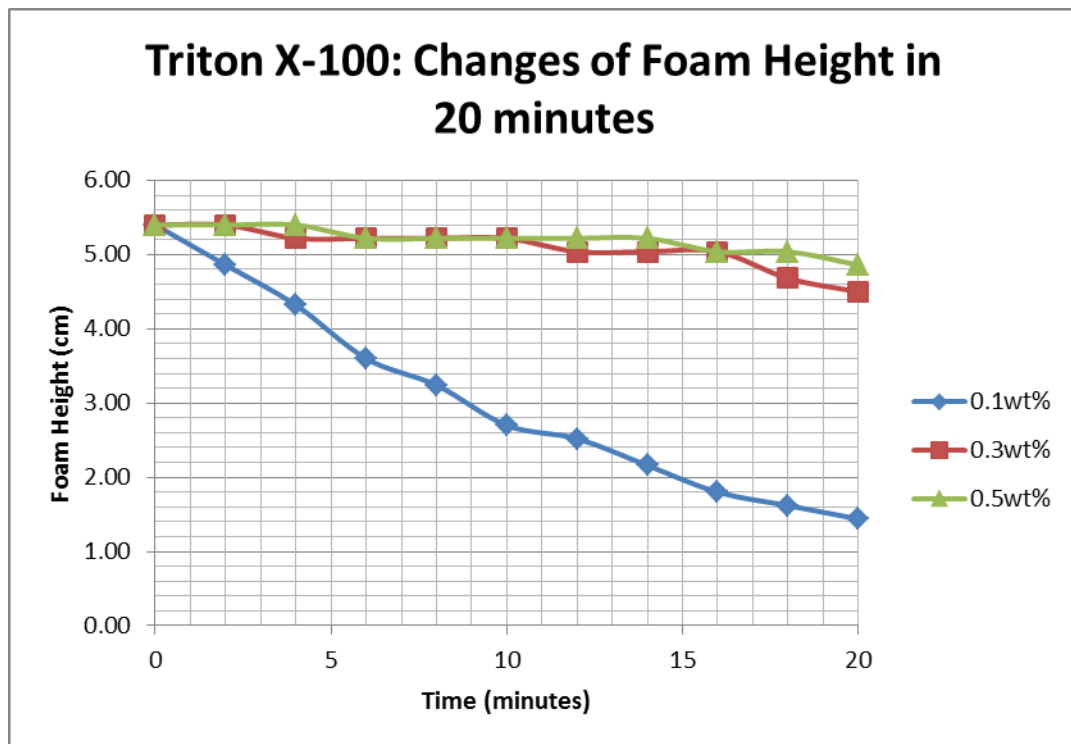


Figure 2: Foam Decline Pattern for Triton X-100 at 3 Different Concentrations

The figure above showed that the initial height of the foam regardless of the concentration had the same value at 5.40cm but the trends for the foam to dewater were different. With slightly increase in concentration from 0.1wt% to 0.3wt% and 0.5wt%, the foam stability was hugely increased as the foam was able to maintain close to the original height for 20 minutes.

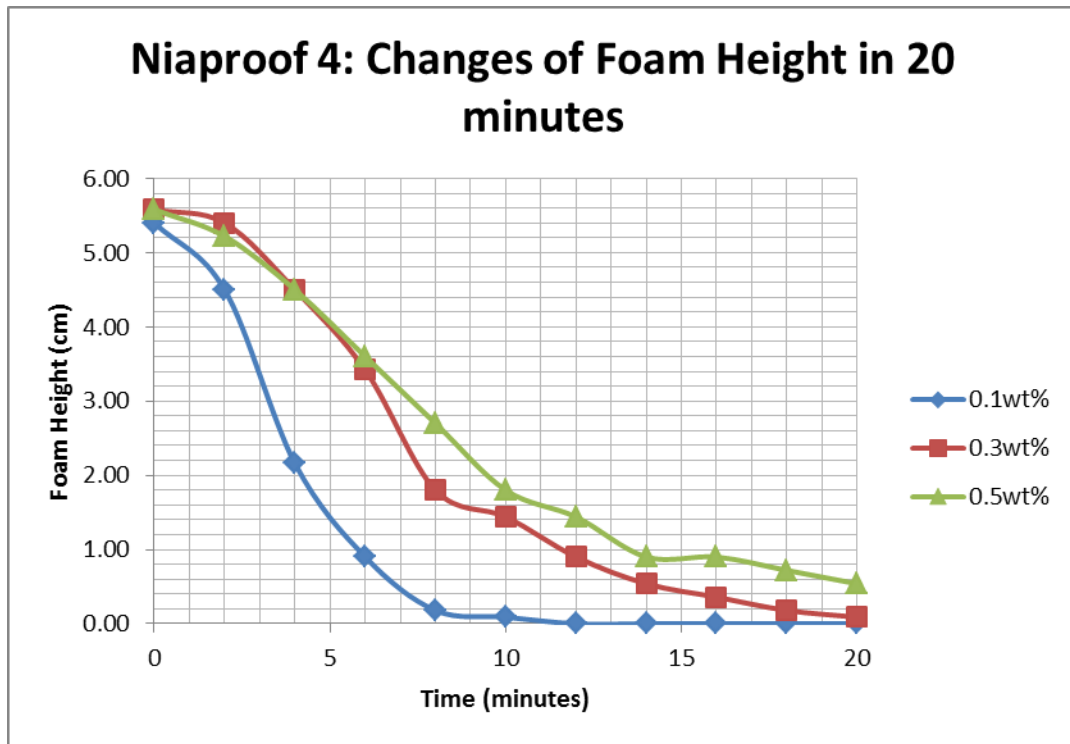


Figure 3: Foam Decline Pattern for Niaproof 4 at 3 Different Concentrations

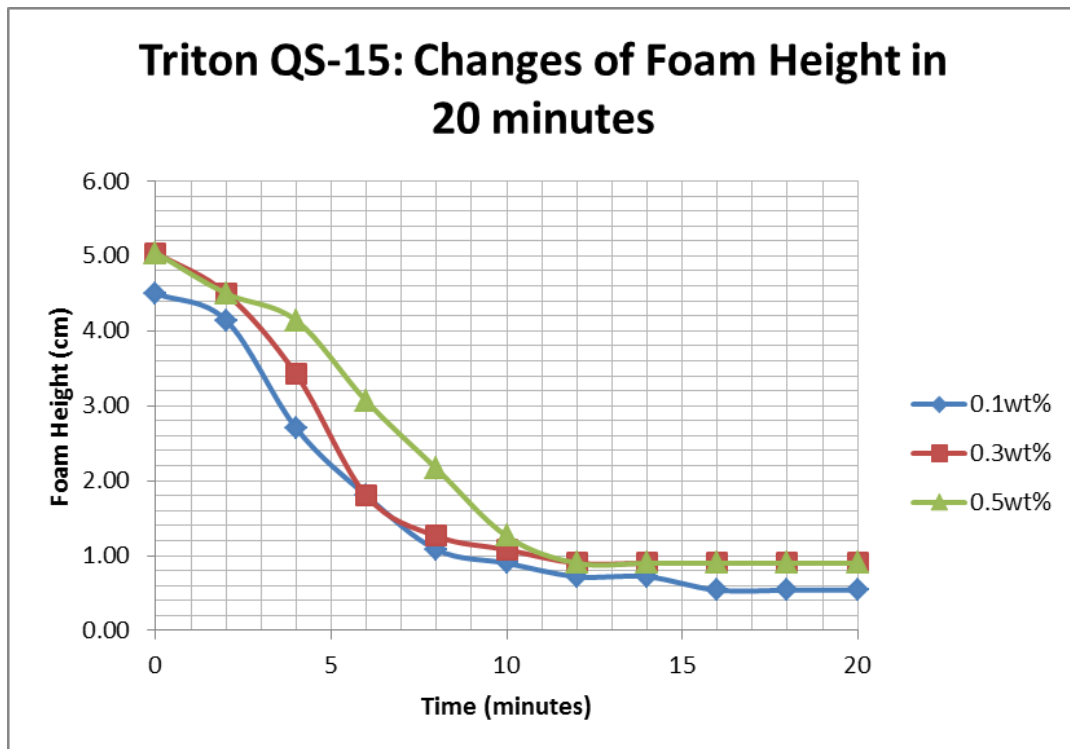


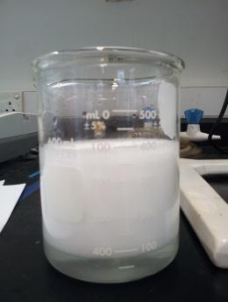






Figure 4: Foam Decline Pattern for Triton QS-15 at 3 Different Concentrations

In addition to that, both Niaproof 4 and Triton QS-15 which from the Anionic group of surfactant showed a good result in foaming. In industry, Anionic surfactants are often used in oilfield foams because they are relatively good surfactants, generally resistant to retention, quite chemically stable and available on a commercial scale. However, foam height of both surfactants did not able to maintain close to original foam height and showed the same dewatering trend for all the concentration tested. However, Triton QS-15 showed different foam behaviour where the foam height was able to remain at the same height after 12 minutes whereas the foam height of Niaproof 4 kept decreasing with time until only the surfactant solution was observed. Hence, in order to enhance the mobility control of the displacing fluid during foam flooding, Triton QS-15 was more preferable compared to Niaproof 4 because the foam was able to stable at a low volume but yet still can ease the propagation of the driving fluid through the formation.

4.1.2 Foam Texture

Table 2: Initial Foam Height and Foam Texture Observations for all the Surfactants at 0.1wt% Concentration

Surfactant	Triton X-100	Adogen 464	Niaproof 4	Triton QS-15
Initial foam height				
Foam Texture		No Foam		

Previous research had proven that the stirring period play an important role in affecting the foamability and texture of the foam. It is proven as all types of surfactants generate the same initial foam height as the stirring period is kept constant at 180 seconds except for Adogen 464 which had no foaming characteristic. However, the stability in term of half time contribute to the screening decision as there is a huge different time for different group of surfactants. In term of foam texture, it can be observed that the foam had the bubble density and as the stirring period goes on, the foams started to look more homogeneous and coarser foam structures had become finer foam. After the stirring period, it is observed that only fine foam existed and compacted in the beaker and it happened to all the generated foam.

4.1.3 Half Time

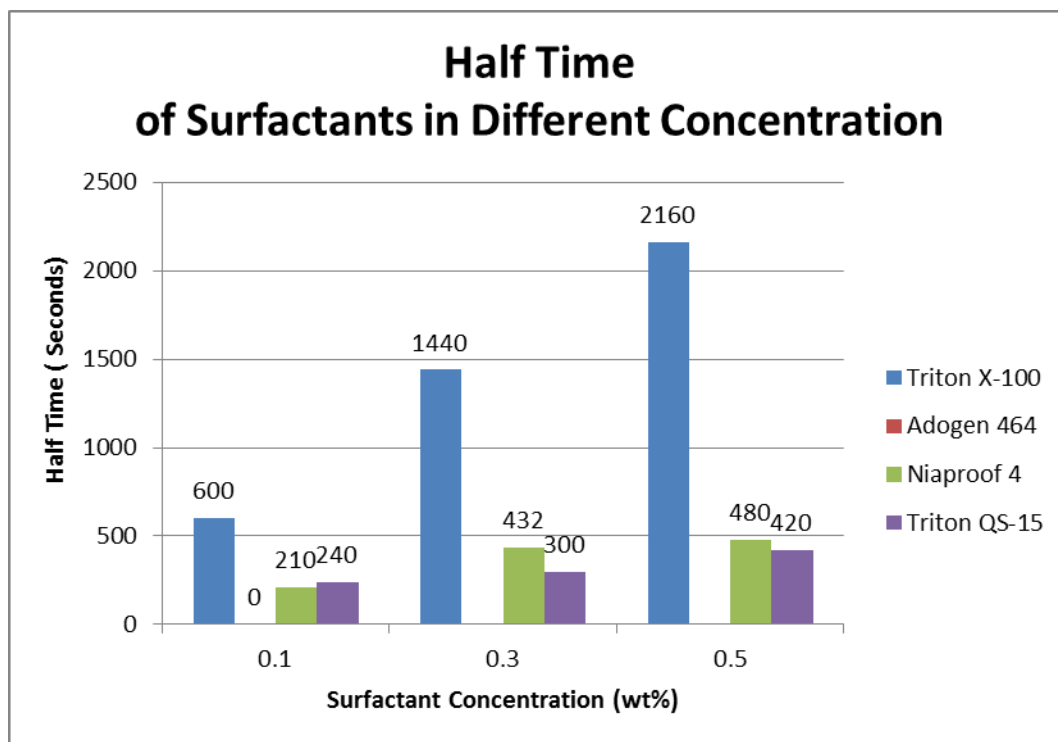


Figure 5: Half Time Result for all the Surfactants in Different Concentrations

All the surfactants shared the same behaviour where the half time increased with the increase of concentration. For Triton X-100, it showed a high half time value as compared to the other surfactants and the value increased in a trend with increase in concentration. However for Niaproof 4 and Triton QS-15, although the half time value

increased with concentration but that is only a slightly increment for concentration 0.3wt% and 0.5wt%. Overall, Triton X-100 from the Non Ionic surfactant group is the most performing foaming agent in term of foam stability as its half time measurement had shown a huge difference and reliable results as compared to the other group of surfactants.

4.2 Adsorption Behaviour onto Sandstone

Surfactant adsorption behaviour played an essential role in reducing the amount of surfactants to be injected for the second flooding method because when foam flooding is conducted in the beginning, some portion of the surfactant will tend to adhere onto or within the pore surface of the rocks throughout the formation. In order to fully utilize the adhered surfactant down hole, micellar flooding is introduced by injecting the solvent, electrolyte and less amount of surfactant in order to form microemulsion and lead to reduction of interfacial tension between the displacing fluid and trapped oil. Refractive index method was introduced to construct the calibration curve because theoretically as the concentration of any particular solution increase, there will be an increase in refractive index.

Table 3: Refractive Index of all the Surfactant in Different Concentrations

Types of Surfactant	Surfactant Name	Surfactant Concentration (wt%)	Initial Refractive Index			Final Refractive Index		
			1st Reading	2nd Reading	Average	1st Reading	2nd Reading	Average
Non-Ionic	Triton X-100	1.00	1.35313	1.35323	1.35318	1.34577	1.34581	1.34579
		0.60	1.34822	1.34824	1.34823			
		0.20	1.34288	1.34284	1.34286			
		0.00	1.34008	1.34010	1.34009			
Cationic	Adogen 464	1.00	1.34952	1.34963	1.349575	1.34266	1.34281	1.34274
		0.60	1.34837	1.34847	1.34842			
		0.20	1.34654	1.34667	1.346605			
		0.00	1.34007	1.34010	1.340085			
Anionic	Niaproof 4	1.00	1.33952	1.34871	1.344115	1.34201	1.34203	1.34202
		0.60	1.34341	1.34342	1.343415			
		0.20	1.34210	1.34208	1.34209			
		0.00	1.34032	1.34032	1.34032			
	Triton QS-15	1.00	1.36102	1.36048	1.36075	1.3398	1.33985	1.339825
		0.60	1.35808	1.35793	1.358005			
		0.20	1.34108	1.34108	1.34108			
		0.00	1.34000	1.34010	1.34005			

Hence, the graphs of refractive index against concentration are plotted for all the surfactant to find the trend line for further analyse on the surfactant being adsorbed.

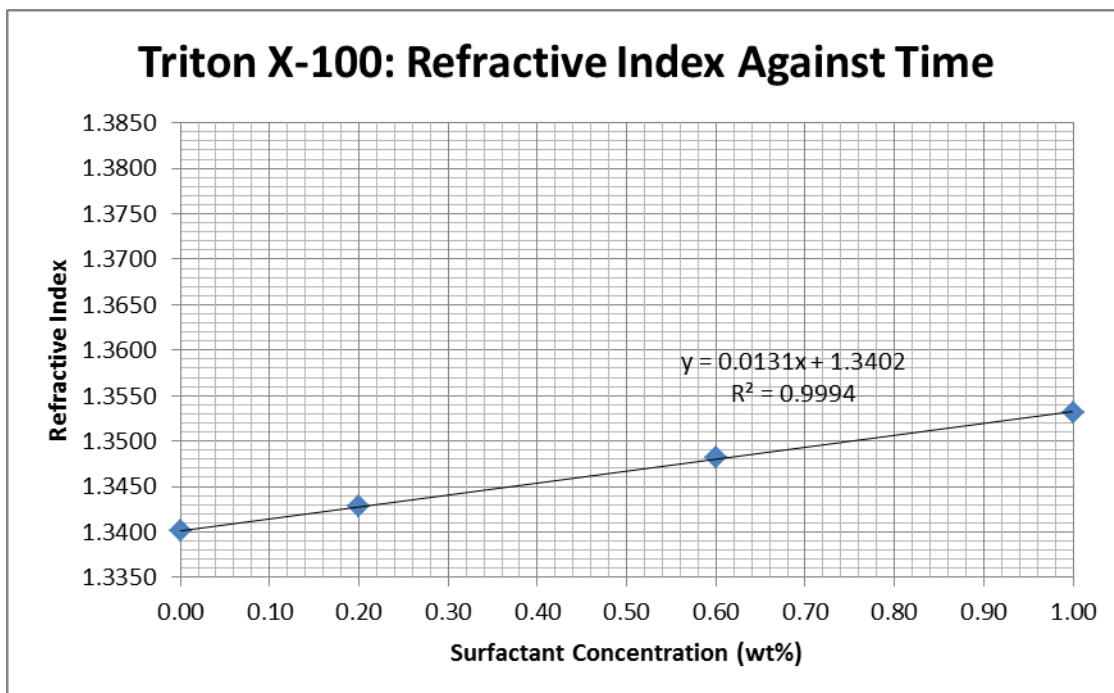


Figure 6: Refractive Index of Triton X-100 in Different Concentrations

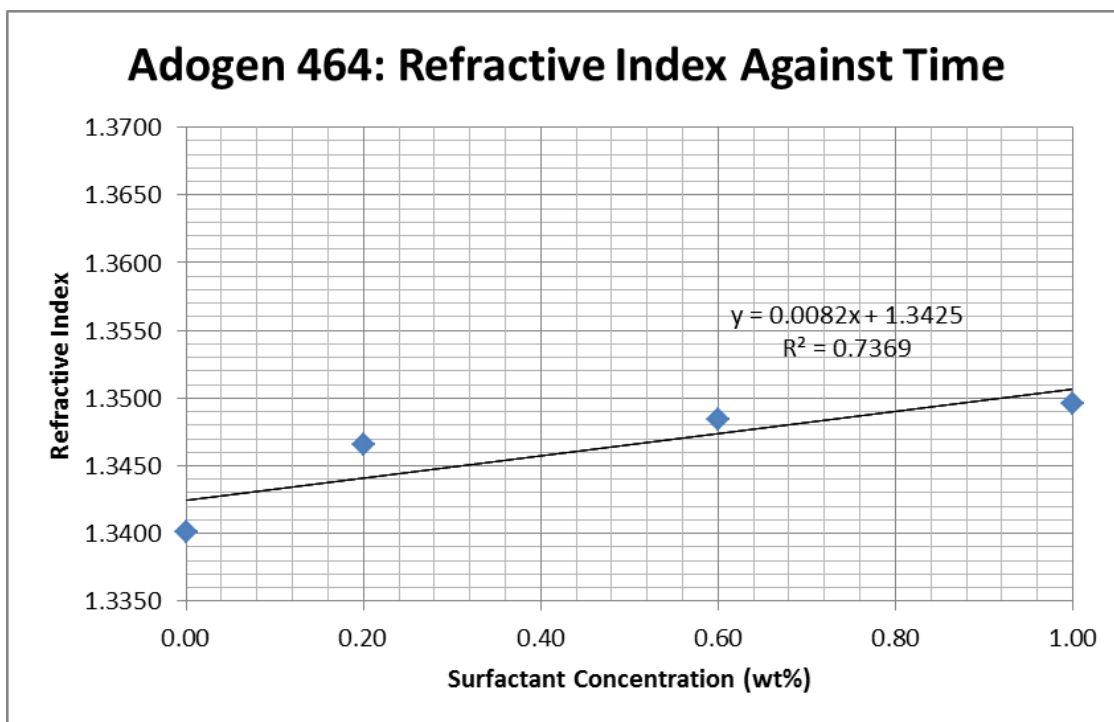


Figure 7: Refractive Index of Adogen 464 in Different Concentrations

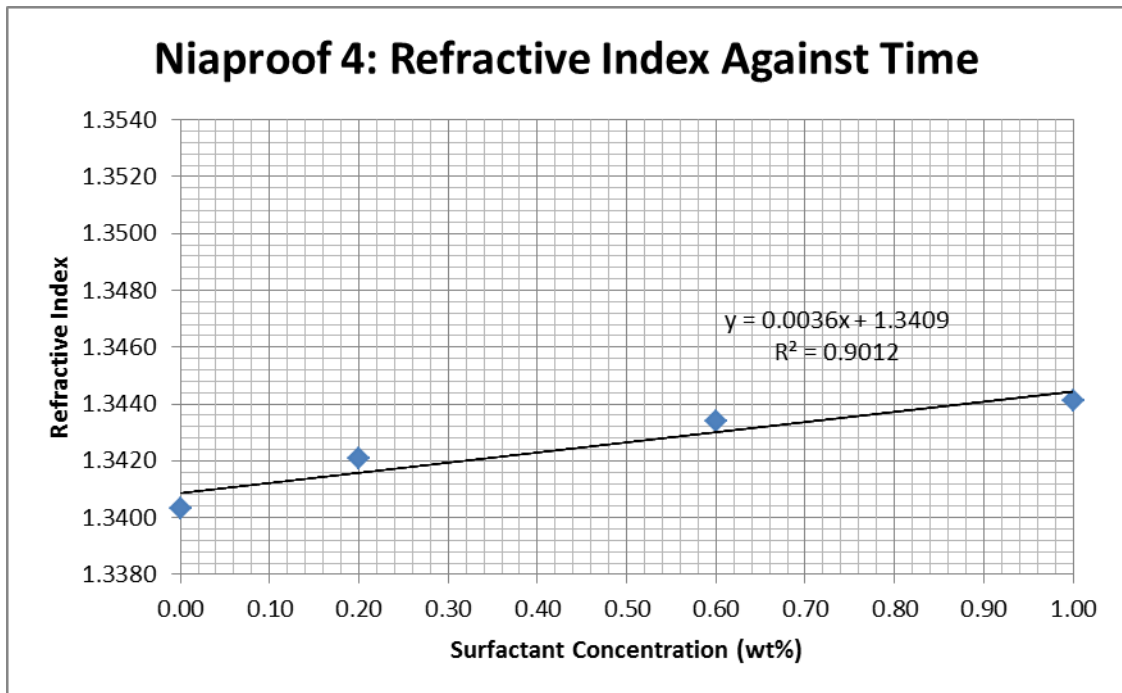


Figure 8: Refractive Index of Niaproof 4 in Different Concentrations

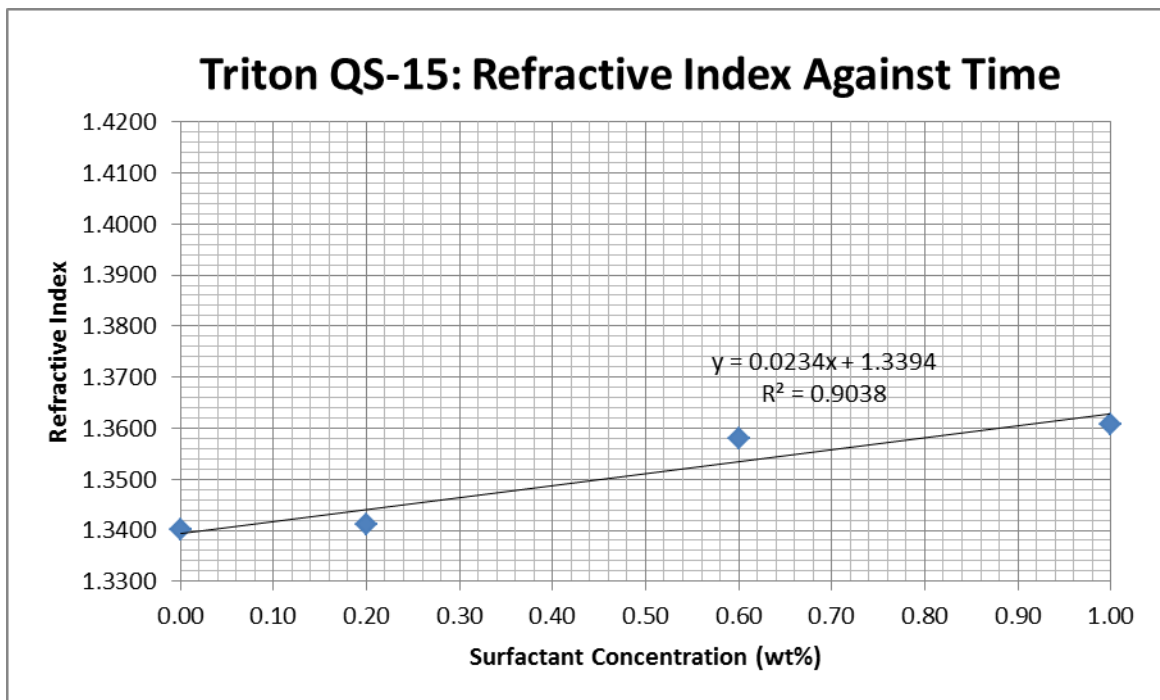


Figure 9: Refractive Index of Triton QS-15 in Different Concentrations

Table 4: Adsorption Results for the Surfactants onto the Sandstone

Types of Surfactant	Surfactant Name	Mass of surfactant adsorbed per gram sandstone (mg/g)
Non-ionic	Triton X-100	115
Cationic	Adogen 464	194
Anionic	Niaproof 4	138
	Triton QS-15	196



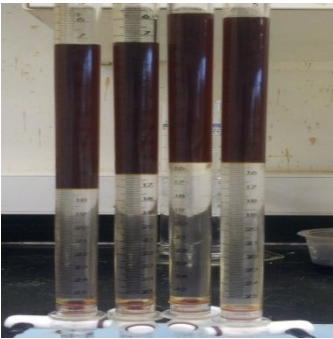


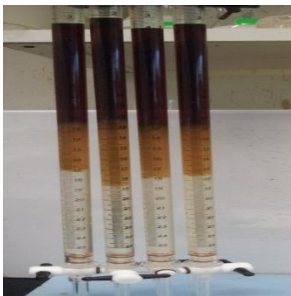
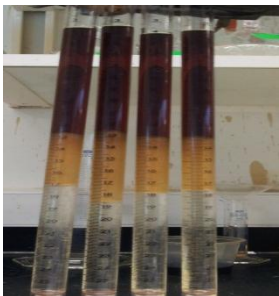
From the table above, Triton X-100 showed the lowest surfactant adsorption behaviour where only 115mg/g, followed by Niaproof 4 with the amount of 138mg/g and then Adogen 4. Triton QS-15 achieved the highest adsorption behaviour among the others which was 196mg/g. As this project aimed to utilised the adhered surfactant for creating micellar solution after the foam flooding, the higher adsorption value was preferable as less amount of surfactant was needed to be included to form micellar solution for injection in order to create microemulsion when react with the crude oil.

4.3 Microemulsion Phase behaviour Study

Number of phase behaviour experiments was performed by changing surfactant concentration for achieving Type-III microemulsion. All the surfactants including Triton X-100, Adogen 464, Niaproof 4 and Triton QS-15 with concentration ranging from 1-4wt% were used in phase behaviour experiments. All the surfactants were able to generate Type-III microemulsions after mixing with crude oil except Adogen 4. Phase behaviour burettes of Type-III microemulsions achieved by four different surfactant formulation (i.e., 1wt%, 2wt%, 3wt% and 4wt%) at same solvent concentration (i.e., 6wt%) are shown in Table 5. In a Winsor Type III system, the surfactant forms a microemulsion in a separate phase between the oil and aqueous phases in which this phase is a continuous layer containing surfactant, water and dissolved hydrocarbon. This situation is ideal to achieve ultralow interfacial tension values and is favourable for EOR. However, Adogen 464 is definitely unfavourable because all the surfactant had been solubilize into the light crude oil phase and resulting only two types of phases exist in the burettes. This is known as Type-II microemulsion as the surfactant is mainly in the

oil phase and resulting water-in-oil (w/o) microemulsion to form. This surfactant-rich oil phase coexists with the surfactant-poor aqueous phase.

Table 5: Result of Phase Behaviour Study for Surfactant Concentration range from 1wt% to 4wt%

Surfactant Name	Initial State of Type-III Microemulsion	Equilibrium State of Type-III Microemulsion
Triton X-100 (Non-Ionic)		
Adogen 464 (Cationic)		
Niaproof 4 (Anionic)		
Triton QS-15 (Anionic)		

4.3.1 Type-III Microemulsion Phase Volume and Equilibrium Time

Volumes of Type-III microemulsion at various concentration were recorded at different time period and plotted versus time.

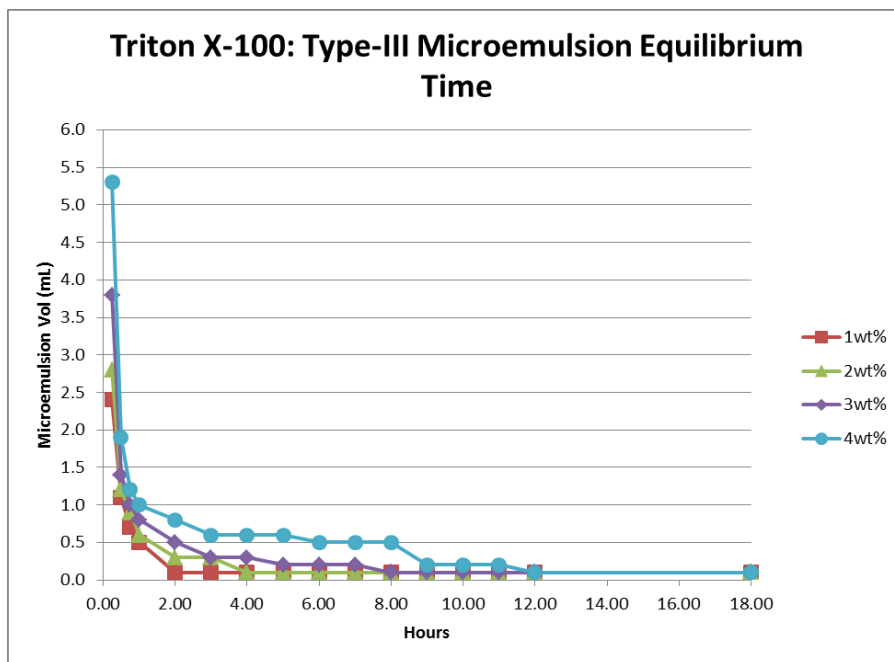


Figure 10: Type-III Microemulsion volume vs Time of Triton X-100 at 1-4wt% surfactant

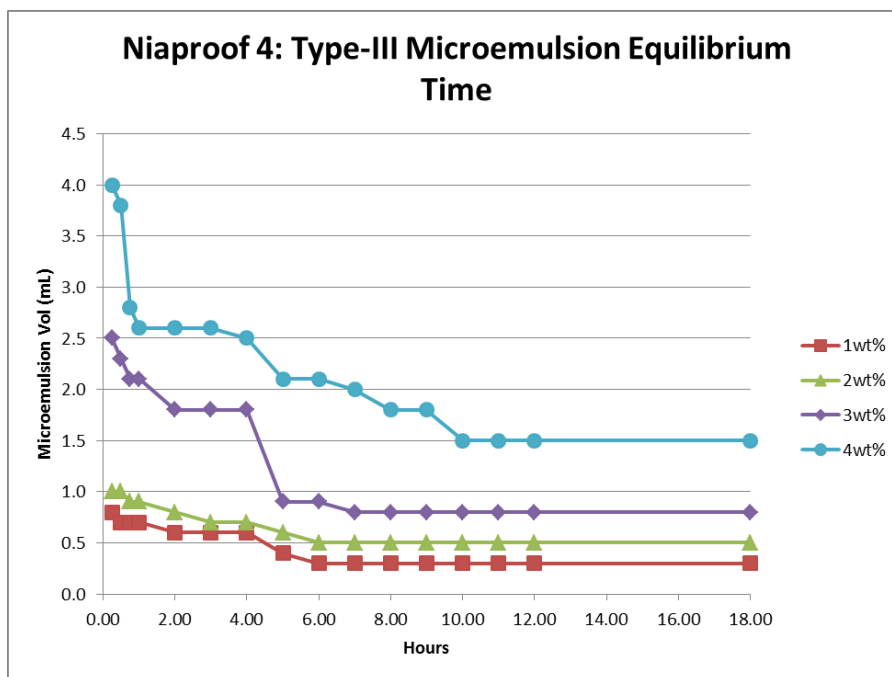


Figure 11: Type-III Microemulsion volume vs Time of Niaproof 4 at 1-4wt% surfactant

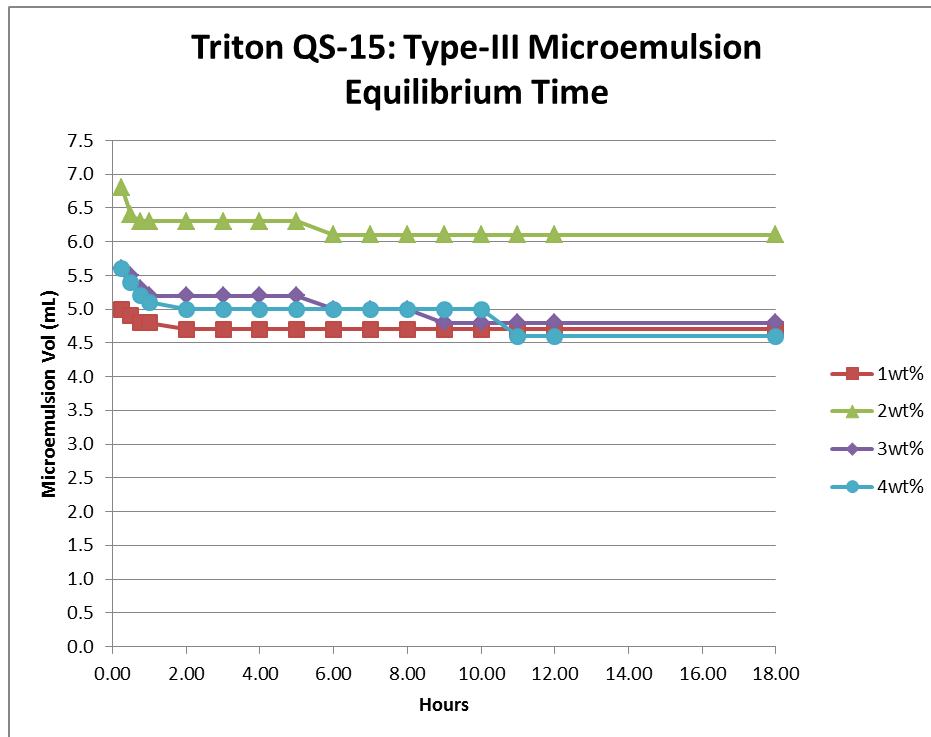


Figure 12: Type-III Microemulsion volume vs Time of Triton QS-15 at 1-4wt% surfactant

Figures 10, 11 and 12 are the plot of microemulsion volume versus time of Type-III microemulsions prepared with 1-4wt% surfactant and 6wt% solvent. In figure 10, it can be seen that microemulsion volume at different surfactant concentration was decreasing with time and remain constant after 12 hours which is its equilibrium time for this particular formulation (Flaaten et al., 2009). For Figure 11, the volume of microemulsion remain constant after 10 hours whereas for Triton QS-15 is 11 hours. However, all of them shared the same trend where the higher the surfactant concentration, the time required for the microemulsion to achieve equilibrium is longer. From the figures above, it can be concluded that stabilization time for the formulation having high surfactant concentration is high and vice versa. It can also be seen that Triton QS-15 is more preferable as large volume of Type-III microemulsion can be generated at 1wt% compared to others and achieved equilibrium in a short time period.

4.4 Continuous Experiment

Based on the foam stability, adsorption result and phase behaviour study, two of the surfactants which performed well were further investigated in terms of the efficiency of the adhered surfactant due to adsorption for the proposed flooding sequence. Hence, Triton X-100 and Triton QS-15 were selected as the most effective foaming agents as well as the ability to generate Type-III microemulsion. Firstly, the concentration of the surfactant to achieve stable foam was determined for both of the surfactants.

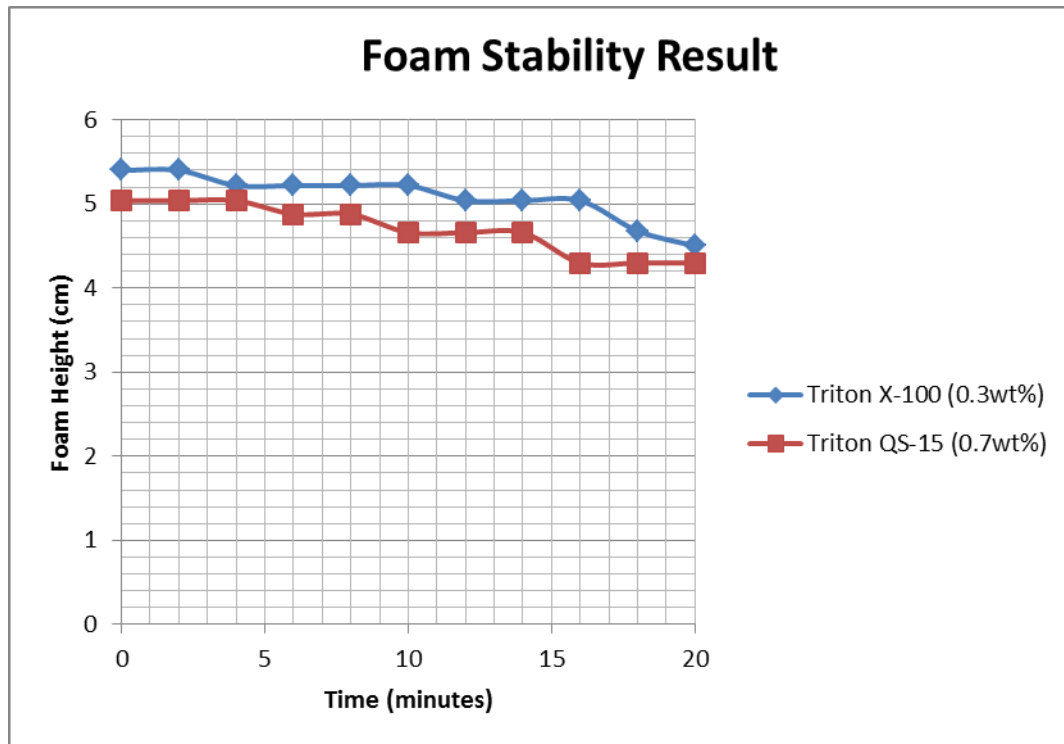


Figure 13: Foam Stability Result for Both Triton X-100 and Triton QS-15

As figure above shown, Triton X-100 was able to have a good foam stability at a lower concentration which is 0.3wt% whereas for Triton QS-15, few incremental of surfactant concentration were tested in order to obtain a good stability foam and finally with the 0.7wt% surfactant, the foam height was able to remain close to the original height. Although the foam height of Triton QS-15 was much lower than Triton X-100, but the stability of the foam will able to ease the propagation of the driving fluid in the foam through the formation.

Next, both of the surfactant solutions were mixed with sandstone and then left for equilibration approximately about one week to allow for the adsorption behaviour to occur. The beakers were then centrifuged to separate the solution and the sandstone. Based on the previous adsorption experiment, amount of surfactant that needed to form microemulsion was calculated. Besides, previous microemulsion phase behaviour study had shown that both surfactants were able to generate Type-III microemulsion at 1wt% concentration, hence this will be used as the reference concentration.

Table 6: The concentration of surfactant needed to add in the micellar solution to form Microemulsion

Surfactant Name	Concentration That Adhered Onto The Surface Of Sandstones	Concentration That Need To Be Added To Form Microemulsion
Triton X-100	0.172 wt%	0.828 wt%
Triton QS-15	0.687 wt%	0.313 wt%

The micellar solutions were then prepared in the beaker containing the sandstone with the solvent concentration (i.e., 6wt%) and electrolyte to form aqueous phases. The solutions were stirred to allow the adhered surfactant to react with each other before the centrifuge process was carried out.

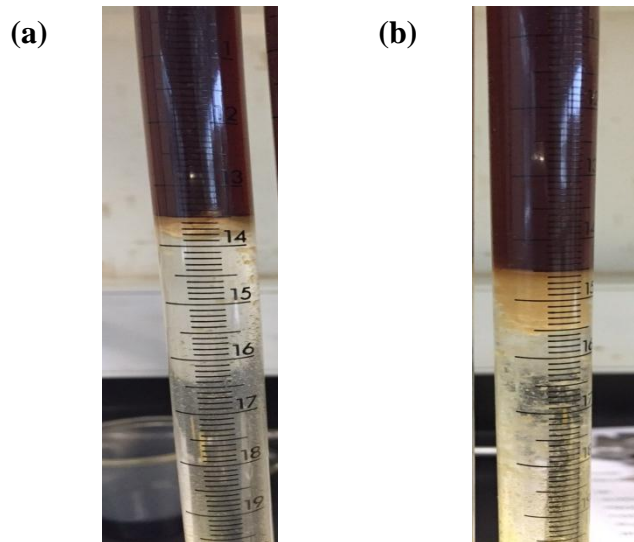


Figure 14 (a-b): Type-III microemulsion prepared by (a) Triton X-100 and 6wt% solvent (b) Triton QS-15 and 6wt% solvent after equilibrium time

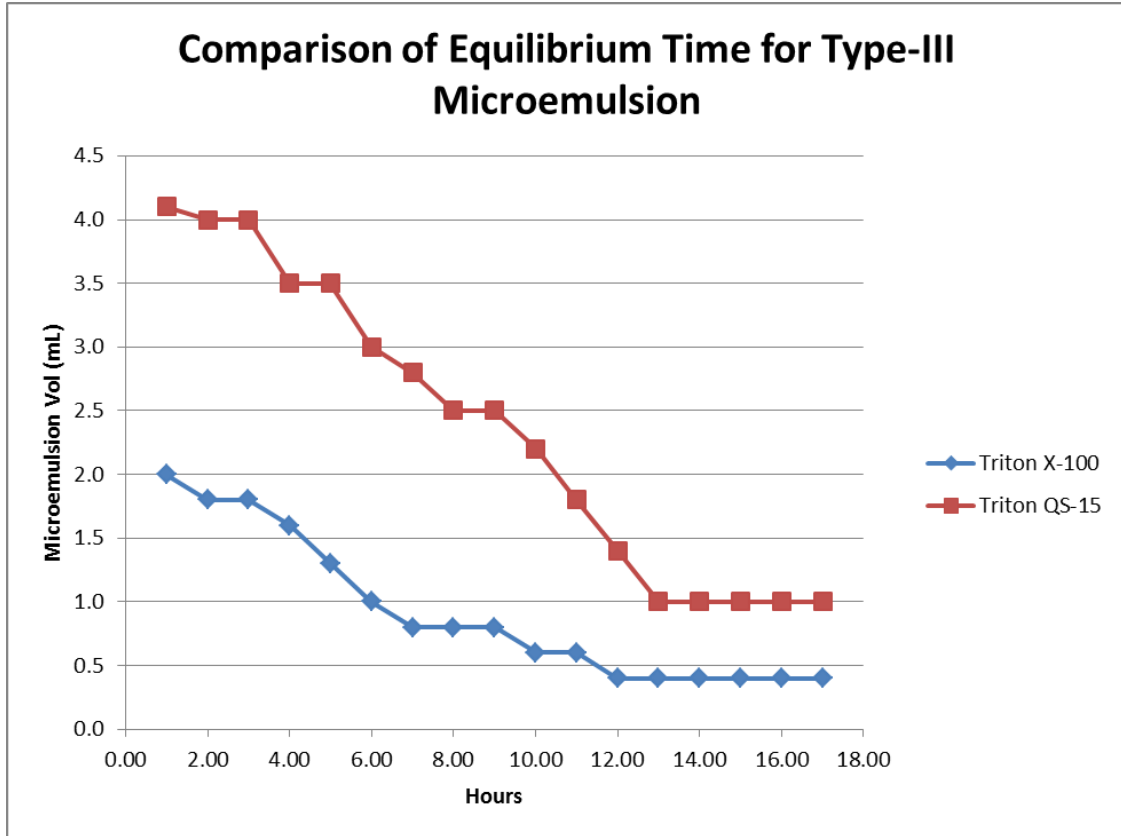


Figure 15: Comparison of Equilibrium Time for Type-III Microemulsion between Triton X-100 and Triton QS-15

Figure 15 showed the volume of Type-III microemulsion generated in the burettes for both the surfactants after achieving equilibrium time. Although Triton X-100 took a shorter time period which is 12 hours to achieve equilibrium as compared to Triton QS-15 which took 14 hours, but the volume of generated Type-III microemulsion was obviously preferable to help in reducing interfacial tension between the displacing fluid and the oil. The final Type-III microemulsion volume for Triton X-100 was 0.4 ml whereas for Triton QS-15 was 1ml.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Foam stability and microemulsion phase behaviour of several surfactants including non-ionic, anionic and cationic were investigated for the proposed flooding sequence which is foam flooding and the followed by micellar flooding . Foam characteristics and microemulsion were studied at different surfactants concentrations. Based on the individual experiment screening, two of the surfactants which were able to perform well in all experiments were further investigated to test their performance by considering the adsorption behaviour. The following are the outcomes of the work:

1. All surfactants except Adogen 464 showed good formability and had the ability to generate Type-III microemulsion but Triton X-100 provided the most stable foam at lowest concentration whereas Triton QS-15 provided the highest volume of Type-III microemulsion at the same concentration.
2. Foamability and foam stability enhances as surfactants concentration increases.
3. Microemulsion volume is directly related to the surfactant concentration.
4. Proposed flooding sequence should be foam flooding first prior to micellar flooding because less concentration of surfactant is needed to generate foam as comparing to generate microemulsion.
5. Triton QS-15 is the best performing and suitable surfactant for this project even though higher concentration of surfactant which is 0.7wt% is needed to achieve good foam stability, the lesser the amount of surfactant needed to form micellar solution due to the high adsorption characteristic.

5.2 Recommendations

- Future experiments should include more types of surfactants to be screened in order to have a more suitable and accurate surfactant.
- Core flooding should be introduced instead of static experiments as it will give a more real situation of the flooding methods.
- Future experiments should be planned and conducted in reservoir condition (Pressure and Temperature) because some of the surfactant characteristic may change instead of ambient condition.

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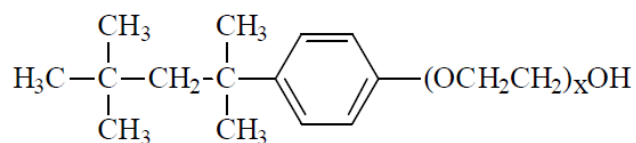
APPENDICES

Appendix 1-A : Product Details of Triton X-100

TRITON X-100Product Number **X-100, T9284, T8787, T8532**

Storage Temperature 25°C

CAS #: 9002-93-1

Synonyms: X-100; TRITON X-100¹; Octylphenol ethylene oxide condensate¹**Product Description**

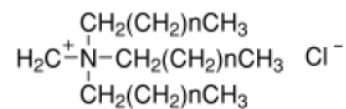
x = 9-10

Appendix 1-B: Product Details of Adogen 464

Product Name:
Adogen® 464

Product Number:	856576
CAS Number:	72749-59-8
MDL:	MFCD00132827

Formula Weight: 1,338 g/mol



Appendix 1-C: Product Details of Niaproof 4

Niaproof 4	
Product Code 4	
Store at Room Temperature	
Product Description	
Molecular Formula: $C_{14}H_{29}NaO_4S$	
Molecular Weight: 316.4	
CAS Number: 139-88-8	
Boiling Point: 100 °C	
Density: 1.06	
pH: 10.5 (neat), 8.6 (0.1% in water)	
Critical Micelle Concentration (CMC): 2.1 mM (water)	
Synonym: Niacet Anionic 4, Tergitol 4	

Appendix 1-D: Product Details of Triton QS-15

Product identifiers	
Product name	: Triton QS-15
Product Number	: QS15
Brand	: Sigma
CAS-No.	: 11105-10-5
Relevant identified uses of the substance or mixture and uses advised against	
Identified uses	: Laboratory chemicals, Manufacture of substances

Appendix 1-E: Schematic Representation and Corresponding photographs of a Series of Microemulsion controlled by Hydrophile-Lipophile Balance (HLB)

