

**Effect of Agitated Turbulence on Demulsification of Crude Oil Emulsions in
Production Separator**

by

Muhammad Amirul Luthfi Bin Zainalabidin

16230

**Dissertation submitted in partial fulfillment of the requirements for the
Bachelor of Engineering (Hons)
Mechanical Engineering**

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Universiti Teknologi PETRONAS

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CERTIFICATION OF APPROVAL

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Approved by,

(Assoc Prof Dr Shaharin Anwar Bin Sulaiman)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2016

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 reference and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMMAD AMIRUL LUTHFI BIN ZAINALABIDIN

ABSTRACT

Water-in-oil (W/O) emulsion is a common production operation problem faced by well operators in the oil and gas industry. The presence of water in oil will induce corrosion, leaching of additives, raise in conductivity and occupy space in the pipeline and separator ineffectively. Emulsion are formed when oil and water are produced along under great amount of agitation or turbulence. Agitation or turbulence is needed for crude oil emulsion to form, but if the agitation could be controlled, it may help in de-emulsifying. The agitation or turbulence have to be kept at average to achieve a good coalescing conditions. However, excessive agitation or turbulence may result intense emulsification and retard the water droplet settling. Therefore, range of agitated turbulence, operating temperature and emulsion treatment duration have to be assessed to study the stability behaviour of crude oil emulsion. Twelve emulsion samples were prepared with water-to-oil ratio of (30:70) and treated with varying parameters that includes the agitation speed, operating temperature and emulsion treatment duration. Three tests were conducted to study on the characteristic of the each emulsion samples for more extensive analysis. The three test were the emulsion stability by bottle test, water content analysis by Karl Fischer titrator and droplet size distribution by cross – polarized microscopy. The results showed that the emulsion samples treated with medium agitation speed of 100 rpm, temperature of 50°C and emulsion treatment duration of 30 minutes, contribute to highest reduction in volume percentage of emulsion by as much as 65% from the original emulsion volume. Thus also resulted in the lowest water content percentage of 0.53% in oil layer and the highest water droplet mean size of 81 μm in the emulsion layer taken after 8 hours bottle test. However, there were no water layer formed in the end of the bottle test for each emulsion samples which resulted in high water content percentage in emulsion layer taken after the treatment and after 8 hours bottle test. From the studies conducted, the agitated turbulence, operating temperature and emulsion treatment duration have a great impact on the stability behaviour of crude oil emulsion. The water-in-oil emulsion problems can be reduced and optimized by performing these parameter, as it is practically feasible for the field operations in oilfield.

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CHAPTER 1

INTRODUCTION

This chapter discussed the background of the project which includes the rationale of the study, problems statement from the project along with the goals and research objectives of the study. This chapter also presents the scope of studies for this work.

1.1 Background of the Project

Crude oil is rarely produced alone. Most of the oil reservoirs in the world nowadays are producing a mixture of oil and water, and when the reservoir matures, it may produce more water than the oil. The oil will always commingle with water, which later will create a number of problems during the oil productions. These liquids are sheared as they flow through pressure-reducing equipment such as pumps or lifting mechanism in oil production which result in emulsion formation (Abdel-Raouf, 2012). Figure 1.1 shows crude oil flowing from the subsurface to surface equipment, flowing through the pressure-reducing equipment causing severe emulsion formation.

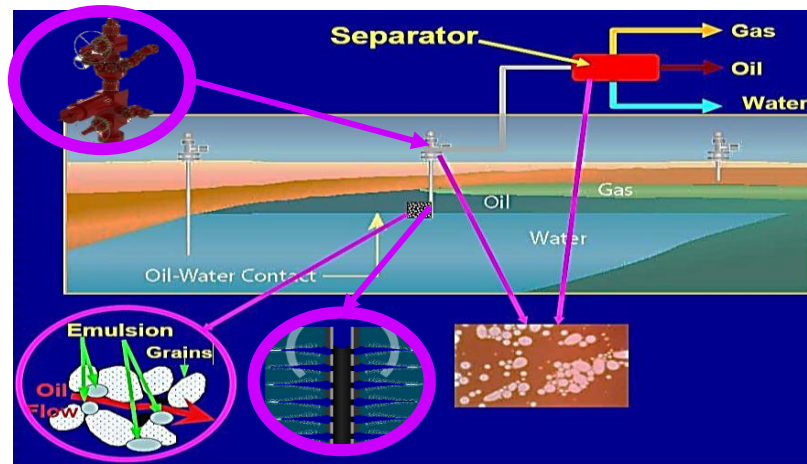


Figure 1.1: Formation of emulsion in oil field (Kokal, 2008)

Almost every oil field produces water-in-oil (W/O) emulsion form in its petroleum production. The stability of emulsions results from the presence of interfacial barrier such as polar components, thus preventing the dispersed water droplets to coalesce in crude oil (Dosunmu, Otikiri, & Fekete, 2012). During the transportation and refining crude oil, it is crucial to separate crude oil from the water for purpose of economical and operational consideration. The presence of water in crude oil will induce corrosion in the pipeline and maximize the space in the pipeline ineffectively. The industrial approaches in tackling these problems is by using the most effective method by demulsifying crude using various type of demulsifier (Opawale & Osisanya, 2013). The usage of demulsifier will destabilize the interfacial barrier between the droplets, thus concluded that the usage of several demulsifier will produces an effectiveness in crude oil and water separation process.

Crude oil emulsion problem in petroleum production and processing have been taken into a serious consideration for its fundamental and industrial aspects by the oil companies especially petroleum operator; and academia researcher in the effort to resolve crude oil emulsion problems (Abdel-Raouf, 2012). To some extent, the petroleum operators have to sell their crude oil in the form of emulsions at very low price because of high treatment cost for the emulsions. Crude oil emulsions are very undesirable due to its space consumption on volume of the dispersed water in the processing equipment such as separator; and pipelines, thus increasing the operating expenditure (OPEX) and capital expenditure (CAPEX) (Mosayebi & Aberdini, 2013). The presence of water-in-oil (W/O) emulsion can reduce the efficiency of refining operations, induce corrosion of the material, increase the heat capacity and reduce the handling capacity of refining equipment and pipelines (Warren, 2007).

Several cases on formation of emulsions are due to poor practices of petroleum operations, thus it is possible to resolve crude oil emulsion by improving the operation practices (Thro, 2007). Crude oil emulsion is quite predicable but, nevertheless the producing water during the oil recovery from well and prevention of agitation from the pressure-reducing equipment are hard to predict due to its limitation, and must be treated (Warren, 2007). Resolving emulsion is an important element in handling the petroleum product, from the moment it is produced until it reaches the refinery for extraction of

petroleum products. Therefore, the evaluations on the emulsion treatment have to be made to decide on the most optimum and effective operating conditions in treating crude oil emulsion in the petroleum system, thus creating the demulsification formulation to treat crude oil emulsion (Wan Razak, et al., 2014).

1.2 Problem Statement

Agitation or turbulence is needed for crude oil emulsion to form, unless it is controlled, it can help in treating the emulsion. Agitation can increase the collision of dispersed water in crude oil, thus increase the possibility for dispersed water to coalesce and settle from the emulsion. However, excessive agitation or turbulence may result in intense emulsification and retard the water droplet settling. The agitation or turbulence have to be kept at average to achieve a good coalescing conditions. Therefore, the range of agitated turbulence, operating temperature and emulsion treatment duration have to be assessed to study stability behaviour of crude oil emulsion. The optimum operating conditions for the demulsification of the crude oil emulsion in the laboratory conditions considered the agitated turbulence, operating temperature and emulsion treatment duration based on the maximum oil layer separated from the emulsion, minimum emulsion layer and minimum water content in oil layer resulted from the experimentation.

1.3 Objectives

The main objective of the project is to study the effect of agitated turbulence, operating temperature and emulsion treatment duration on stability behaviour of crude oil emulsions. The secondary objective is to devise an optimum operating conditions in the laboratory conditions. The optimization of crude oil demulsification process considers the technical engineering aspects which evaluate the effectiveness of demulsification process by considering the maximum oil layer separated from the emulsion, minimum emulsion layer and minimum water content in oil layer result from the experimentation.

1.4 Scope of Studies

The project is a part of the upstream flow assurance project under the deepwater technology category at Universiti Teknologi PETRONAS. This extensive research is conducted to resolve the emulsion problems during the oil production effectively and economically. Within this project, the scopes of studies are stated as follow;

- a) The tests conducted are limited to RE-110 crude oil from Terengganu Crude Oil Terminal (TCOT), Malaysia.
- b) The research studies is fully experimentation in the laboratory condition. No simulation was used in this research studies.

CHAPTER 2

LITERATURE REVIEW

This chapter discussed literature review on the introduction to crude oil emulsion, formation of crude oil emulsion in oil and production systems, effects of emulsion formation, nature of emulsifier, emulsion stability mechanism and various emulsion treatment methods used in the oil and industry to tackle crude oil emulsion problems.

2.1 Introduction to Crude Oil Emulsion

Abdel-Raouf (2012) through his publication ‘Crude Oil Emulsions – Composition Stability and Characterization’ technically defined emulsions as dispersion of immiscible liquids with one another in the form of the droplet. Udonne (2012) stated that the emulsion do not usually exist in the production formation, however emulsion are formed when oil and water are producing along under great amount of agitation or turbulence. When water and crude oil in reservoir flows into the wellbore through the perforated hole in the casing, the large pressure differences are induced that severely mixed the produced liquid consist of oil and water together thus, forming crude oil emulsion. Tang (2005) notified that increases in watercut in the petroleum production will result in emulsification of oil which significantly increased the viscosity that will hinder the transportation of crude oil, thus limiting the wellbore and pipeline delivery capabilities.

The emulsion can be classified by the amount of liquid in one another (Abdel-Raouf, 2012). Abdel-Raouf’ statement was supported by Kokal (2005) that crude oil emulsion are separated into three groups which are classified into water-in-oil (W/O) emulsion, oil-in-water (O/W) emulsion and complex emulsions or known as water-in-oil-in-water (W/O/W) emulsions. Warren (2007) mentioned that crude oil emulsions formed most of the time, is water droplets that are dispersed in crude oil and known as a “normal” emulsion or water-in-oil (W/O) emulsion. Warren (2007) also explained that an “inverse” or “reverse” emulsions is the dispersion of oil droplets in the water to form oil-in-water (O/W) emulsions. The emulsion are interrelated to more complex form, and may formed

as the water-in-oil or oil-in-water but due to much agitation or turbulence, it will become multistage as water-in-oil-in-water emulsion when water films enveloped the small volume of the water-in-oil emulsions (Warren, 2007). Figure 2.1 shows the photomicrograph of various types of emulsions.

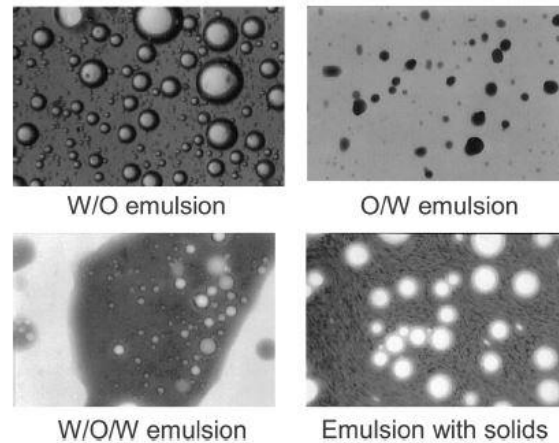


Figure 2.1: Photomicrographs of types of emulsions (Kokal, 2005)

2.2 Emulsion Formation in Oil and Gas Production

According to Warren (2007), the conditions for a crude oil emulsion to form are the liquids subjected to form crude oil emulsion must have the natural characteristic of immiscible liquids and with the assistance of sufficient turbulence and agitation, it will disperse one liquid to another in droplets and chemically bound or stabilized by surface active components as emulsifying agents. Warren (2007) also added that the agitation energy may be supplied from pressure-reducing equipment from subsurface such as bottomhole pump; the flowing of crude oil through tubing, wellhead, subsea manifold and flowlines to surface equipment that may cause pressure drop such as flows through chokes, valves or other surface facilities equipment. Kokal, Al-Ghamdi and Meeranpillai (2007) notified based on their findings that moderate amount of mixing is beneficial and necessary for the emulsification but severe mixing or agitation can lead to tight emulsions and it may re-emulsify after water separation.

Figure 2.2 shows the schematic representation of various processes occurring during emulsion formation. The drops are depicted by thin lines and the surfactant by heavy lines and dots.

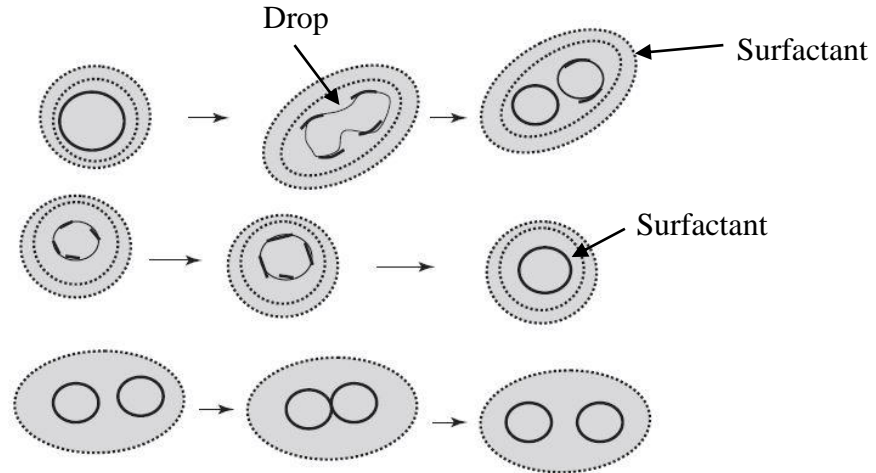


Figure 2.2: Schematic representation of the various processes occurring during emulsion formation. (Tadros, 2013)

Emulsion are stabilized by emulsifiers such as surface active agent and surfactants that tend to bind at the mixture of oil and water interface and eventually form an interfacial film that will reduce the interfacial tension (IFT), thus encourage the dispersion of the water droplets in crude oil (Kokal, 2005). He also added that naturally occurring emulsifier such as asphaltenes and resins, and organic acid and bases that have higher boiling point fractions are the main contributing factor to interfacial film as it form around the dispersed water in crude oil emulsion. Kokal (2005) also indicated that the chemicals that are injected into the reservoir formation may consist of surfactants. The chemicals used are for the purpose of drilling fluids, well stimulation or chemical inhibitor specifically used to inhibit corrosion, wax, scale and asphaltenes. Based on the research paper by Kokal and Wingrove (2000) explained that fine inorganic solid particles are capable in stabilizing crude oil emulsion effectively as organic solids altered the wettability of the inorganic solids, forming rigid structures of the interfacial films that strictly inhibit the dispersed water to coalesce in crude oil emulsion.

2.3 Effects of Emulsion to Production Operations

The typical form of crude oil emulsion under normal oilfield conditions is water droplets disperse in the oil or commonly known as water-in-oil (W/O) emulsion (Kokal & Wingrove, 2000). Kokal et al. (2001) also explained that the largest national oil company, Saudi Aramco has encounter with numerous production operations problems due to formation of crude oil emulsions in oil fields and resulting to an increase in the usage of demulsifier, off-specifications crude oil production and sometimes will cause equipment failing in the gas oil separation plant (GOSP) and even in wet crude handling facilities (WCHF). According to Udonne (2012) through his research on ‘Chemical treatment of emulsion problem in crude oil production’ added that undesirable consequences such as corrosion, leaching of additives and raise conductivity will affect from the presence of dispersed water in crude oil will results in troublesome consequences. As an initiative to reduce crude oil emulsions severity, crude oil emulsions need to be treated to break the dispersion of water droplets along with emulsifier to meet the requirement for transportation, storage and export along the lines to the downstream processing (Kokal & Wingrove, 2000).

2.4 Nature of the Emulsifier in Crude Oil

Udonne (2012) stated that emulsion consists of the three different phases; the internal or known as discontinuous phase consists of fine droplets distribution, the external or known as continuous phase where the medium holds the suspended droplets and the inter-phase where emulsifier or stabilizer that induces the stability of the emulsion. The most effective emulsifiers are non – ionic surfactants that can be used to emulsify water-in-oil emulsion. Surfactants can stabilize the emulsion against flocculation and coalescence (Tadros, 2013). He also explained that the emulsifiers are surfactants and consists hydrophobic and hydrophilic molecules ends. With the presents of two immiscible liquid the emulsifiers will attract to the internal and external phases across the interface, thus layering the round droplets with a protective sheath on the dispersed phase (Udonne, 2012).

According to Abdel-Raouf (2012) stated that the hydrocarbons in crude oil are categorized into four broad classes; aromatics, saturated, resin and asphaltenes. The structures of the saturated, asphaltenes, aromatics and resins are as shown Figure 2.3;

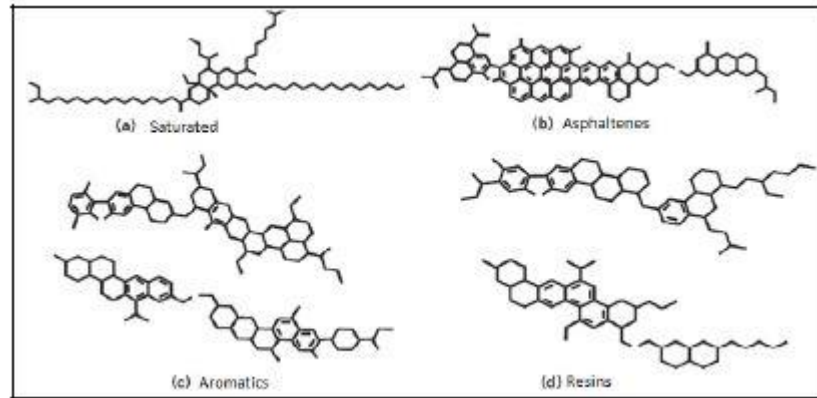


Figure 2.3: Structures represent a) saturated b) asphaltenes c) aromatics and d) resins.
(Bernucci et al., 2006)

Wan Razak et al. (2014) stated that the significant factors that will contribute to the stable formation of crude oil are mixing of two or more immiscible liquids; emulsifying agents including surface active such as resins, organic acids, asphaltenes, and bases and injected surfactant; and finely divided solids such as clay particles, sands, asphaltenes, wax, scale and other. Wan Razak et al., (2014) concluded that natural fines are the major contributing factor to stable emulsion formation based on their studies on the reservoir.

Kokal and Wingrove (2000) discussed that most of produced emulsion are stable, but within specific duration, crude oil emulsion itself equipped with the certain kinetic stability of the emulsions. The degree of kinetic stability are classified into loose emulsions which can be break down within few minutes; medium emulsions which can be breakdown within ten of minutes; and tight emulsion which can be breakdown within hours, days and might extend into weeks.

2.5 Emulsion Stability Mechanism

According to Warren (2007) through his publication entitled ‘Emulsion Treating’ stated that emulsion separation of crude oil and formation water mixture into its respective phase of oil and formation water consists of three degree of separation; coalescence or flocculation, destabilization or coagulation, and gravity selection or sedimentation. Warren’ statement was supported by Tadros (2013) through his publication entitled ‘Emulsion Formation and Stability’” stated that some of separation processes might occur on the vessel or separator influence by the difference in density between the dispersed droplet and the medium and droplet-size distribution in the emulsion. Ostwald ripening can identify the droplet-size distribution and the solubility of dispersed droplets while attractive and repulsive forces magnitude identify flocculation. Coalescence can identify the liquid film stability and the other process is phase inversion. Figure 2.4 shows illustrated the schematic diagram of the various emulsion breakdown processes in emulsions.

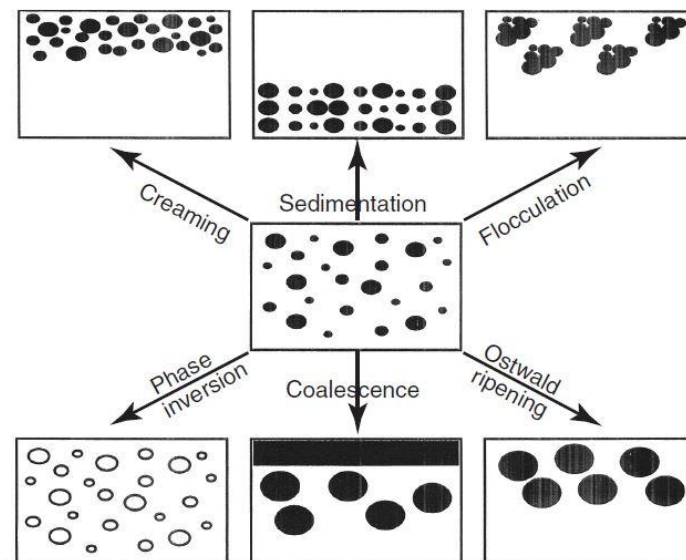


Figure 2.4: Emulsion formation and stability (Tadros, 2013)

Tadros (2013) also explained that physical phenomena occurred in every separation mechanism are complicated and involves extensive analysis of the various surface forces within the emulsions. The separation mechanism might occur at the same time, not in the orderly processes, thus it will intensify the emulsion analysis.

2.6 Emulsion Treating Methods

According to Kokal (2005) through his research ‘Crude Oil Emulsion: A State-of-the-Art Review’ stated that crude oil emulsions can be treated in every phase of oil production, transportation and processing; in the reservoir, wellbore, wellhead, wet crude handling facilities; transportation by pipelines, flowlines; onshore crude storage and petroleum processing. Thro (2007) also added that there is no such as perfect separation, and therefore there will always some dispersed water left in crude oil, which range from less than 1% of water content to more than 20% water content in crude oil by volume. The demulsifier will become ineffective with the watercuts more than 60% and the peak viscosity will be as high as 10000 cp.

Demulsification or emulsion treating is defined as separation of dispersed liquid from another liquid where it is suspended (Udonne, 2012). The purpose of the demulsification is to break down the interfacial film in crude oil emulsion and induce the surfactant to desired phase, either to the oil side or the water side by enhancing the coalescence of the oil particles and sediments (Udonne, 2012). Warren (2007) added that an emulsion treating unit or system might use one or more methods in aiding the destabilization, coalescence and gravity separation. Figure 2.5 shows the methods used to aid the destabilization, coalescence and/or gravity separation.

METHODS TO AID DESTABILIZATION, COALESCENCE. AND/OR GRAVITY SEPERATION		
<u>Destabilization</u>	<u>Coalescence</u>	<u>Gravity Separation</u>
Heating	Heating	Heating
Chemical	Agitation	Gravity settling
Distillation	Coalescing plates	Centrifugation
	Electrostatic field	
	Water washing	
	Filtering	
	Fibrous packing	
	Retention time	
	Centrifugation	

Figure 2.5: Emulsion treating methods (Warren, 2007) NATCO Group Inc.

Design and operational parameters can affect crude oil emulsion separation and must be adjusted and optimized according to its optimum separation process, for example operating temperature and pressure, chemical demulsifier selection, flow rate, chemical demulsifier injection rate, viscosity, separator design and sizing and fluid capacity and levels (Warren, 2007). The idea was supported by Udonne (2012) added that every oil well has different type of emulsion separation, thus usage of chemical treatment as an universal solution has to consider the different of stability, pH, surface active surfactant or natural surfactant in different oil wells.

According to According to Chin (2007), the significant factors that will help in treating the foaming oil is by assistance of agitation or baffling, gravity settling, heating, chemical and centrifugal force. The idea was supported by Soffian and Niven (1993) previously through research paper on Emulsion Treatment Program, oil treating methods can be grouped into four main categories such as chemical, settling time, heat and agitation. Each oil-treating method categories is very unique and must be analyses individually to come up a definite solution. Warren (2007) stated that if demulsification is properly done, using agitation, less heat with least chemical dosage, and gravity settling capacity or time can provide the most cost-effective breakdown of the emulsion.

Agitation or turbulence is needed for crude oil emulsion to form, unless it is controlled, it can help in treating the emulsion (Warren, 2007). He added that through agitation, it can increase the collision of dispersed water in crude oil, thus increase the possibility for the dispersed water to coalesce and settle from the emulsion. Warren warned that excessive agitation or turbulence may result in intense emulsification rather than resolving the emulsion. Warren (2007) through his findings suggested that to achieve a good coalescing conditions, the turbulence have to be keep at average Reynolds numbers range at 50,000 to 100,000. Warren' ideas was supported by Kokal et al. (2007) stated that the gentle agitation are need after the mixture of the demulsifier follows by gravity settlement within specific duration to allow the dispersed water to coalesce by gravity settling.

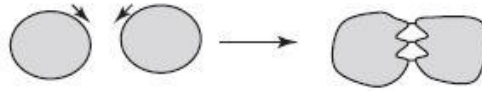


Figure 2.6: Schematic representation of surface fluctuations (Tadros, 2013)

Demulsification can be assisted by placing a properly designed baffles plates by distributing the emulsion evenly in a vessel or separator that will generate gentle agitation that will assist the coalescence of dispersed water droplets by applying energy for dispersed water particles to collide and coalesce to the surface (Warren, 2007). Figure 2.6 shown the schematic representation of surface fluctuations due to gentle agitation applied to the droplets. Warren also warned that excessive baffling would cause too much on the agitation or turbulence and, thus might increase the emulsification and retard the water droplet settling.

2.7 Experimental Approach for Demulsification Activities

Crude oil emulsion must be separated almost completely before the oil can be transported and processed further. The treatment methods for emulsion in crude oil are distinguished into few applications namely as application of heat, electricity, chemicals, polymers and natural treatment (Udonne, 2012). This statement was supported through his findings as five experiment were conducted using sulphuric acid as the demulsifier which varies in mixing speed and resulted in higher percentage of basic sediment of water obtained in higher mixing speed. Kokal and Wingrove (2000) also experimented the mixture of oil and water in an automatic shaker and blender for varying mixing duration which resulted decreases in Emulsion Separation Index (ESI) and produced more stable emulsion.

Similarly, Opawale and Osisanya (2013) conducted series of laboratory test to study the effects of shear energy and emulsifier on the stability of emulsion at various watercuts. It was observed that higher shearing energy and the concentration of emulsifier increases the degree of tightness of the emulsion. The recent studies by Abdurahman, Azhari and Yunus (2013), investigated the effect of mixing speed on the emulsion viscosity and stability for Masilla and Tapis oil-in-water emulsion at mixing speed of 800, 1000, 1200, 1500, 1700, and 1800 rpm. Based on the studies, increase in mixing speed resulted in an increase in viscosity and stability of the emulsion for both crude oil. For the continuation of the studies, Liyana, Abdurahman, Rizauddin, Gimibun and Nurdin (2014) conducted series of experiment to study the influence of mixing speed on the viscosity and stability of oil-in-water emulsions at higher mixing speeds of 10,000, 12,000 and 15,000 rpm. Similarly, the experiment resulted an increased in stability and viscosity of the emulsion and decrease in oil droplets size as higher mixing speed applied.

On the other part, Caubet, Le Guer, Grassl, El Omari and Normandin (2009) studied the droplet size distribution behaviour during the emulsification with different physical and formulation parameter such as stirring time, rotational speed, surfactant type, concentration and salt addition. The diameter of the droplets size decrease with increasing stirring time and rotational speed of the rod. In contrast, the water-in-oil emulsion with different water volume fraction of 0.1, 0.25 and 0.4 were prepared by using

a high shear rotating homogenizer at two distinct rotational speed of 14,000 and 22,000 rpm. The result showed that the emulsion produced stable emulsion and did not show any phase separation with higher rotation speed and water fraction of 22,000 and 0.4 respectively (Karcher, Perrechil and Bannwart, 2015). Lastly, Martinez, Medellin, Papayanopoulos, Sanchez and Lozano (2007) experimented emulsion prepared at different mixing speed to study the droplets size and viscosity of oil-in-water emulsions. The results revealed that the droplet size decreases dramatically when the mixing speed is increased from 1000 to 1500 rpm while the droplet size is maintained very similar when the speed is increased from 1500 to 2500 rpm. Table 2.1 shows the summary of the experimental approach for demulsification activities for the past 20 years.

Table 2.1: Summary of experimental approach for demulsification activities

Literature	Method Description	Results
Udonne (2012)	Five experiment were conducted using sulphuric acid as a demulsifier in 500 and 1000 revolution per minute in the centrifuge. The test tubes were later inserted into the machine for spinning and for separation.	The result shows that using sulphuric acid as a demulsifier in 500 and 1000 revolution per minute in the centrifuge, 1.25 and 2.5% basis sediments of water was obtained.
Kokal and Wingrove (2000)	Three experiments were conducted which the oil and water were shaken in an automatic shaker and blender for varying mixing duration. These prepared emulsions were treated with 100 ppm of demulsifier at 120°F.	ESI decreases with increased shear, which is the emulsion becomes more stable.
Opawale and Osisanya (2013)	Series of laboratory test were carried out to study the effects of shear and emulsifiers on the stability of oilfield emulsion at various watercuts.	It was observed that shearing energy and the concentration of asphaltenes determines the degree of emulsion tightness.
Abdurahman, Azhari and Yunus (2013)	Investigate the influence of mixing speed on the emulsion viscosity and stability, the behavior of Masilla, Tapis and the Masilla and Tapis blend oil-in-water emulsions was studied at mixing speeds of 800, 1000, 1200, 1500, 1700, and 1800 rpm.	Increasing the mixing speed clearly results in an increase in emulsion viscosity. This increase is explained by the decrease in the droplet size of the oil dispersed phase caused by the increased mixing speed, which in turn increases emulsion viscosity.

Caubet, Le Guer, Grassl, El Omari, Normandin (2009)	The droplet size distribution during the emulsification was investigated according to different physical and formulation parameters such as stirring time, rotational speed, surfactant type, concentration, and salt addition.	The diameter of the droplet size decrease with increasing stirring time. The mean droplet radius decreases with increasing rotational speed of the rod.
Liyana, Abdurahman, Rizauddin, Gimbut, Nurdin (2014)	The influence of mixing speed on the viscosity and stability of o/w emulsions has been thoroughly studied. The stability of heavy o/w emulsions have been investigated at different mixing speeds of 10,000, 12,000 and 15,000 rpm	Increasing mixing speed slightly increased the stability and viscosity. The increasing of mixing speed promotes decreasing in the oil droplet size and increasing the viscosity of the emulsion.
Karcher, Perrechil and Bannwart (2015)	W/O emulsions containing different water volume fractions (0.1, 0.25 and 0.4) were prepared in a batch calorimeter by using a high-shear rotating homogenizer at two distinct rotation speeds (14000 and 22000 rpm).	The emulsions produced at different rotation speeds (14000 and 22000 rpm) and water volume fractions (0.1, 0.25 and 0.4) were highly stable and did not show phase separation.
Martinez, Medellin, Papayanopoulos, Sanchez and Lozano (2007)	The viscosity and droplet size of oil in water emulsions prepared at different speed of mixing have been measured.	The results reveal that the droplet size decreases dramatically when the mixing speed is increased from 1000 to 1500 rpm while the droplet size is maintained very similar when the speed is increased from 1500 to 2500 rpm.

CHAPTER 3

METHODOLOGY

This chapter discussed the overview of methodology through project execution flowchart. The Gantt chart for the project, key milestones and experimental design specification will also be discussed thoroughly in this chapter.

3.1 Overview of Methodology

This project is an experimental-based project to study crude oil emulsion stability behaviour towards the agitated turbulence, operating temperature and emulsion treatment time in the laboratory condition. Figure 3.1 shows the project execution flow chart.

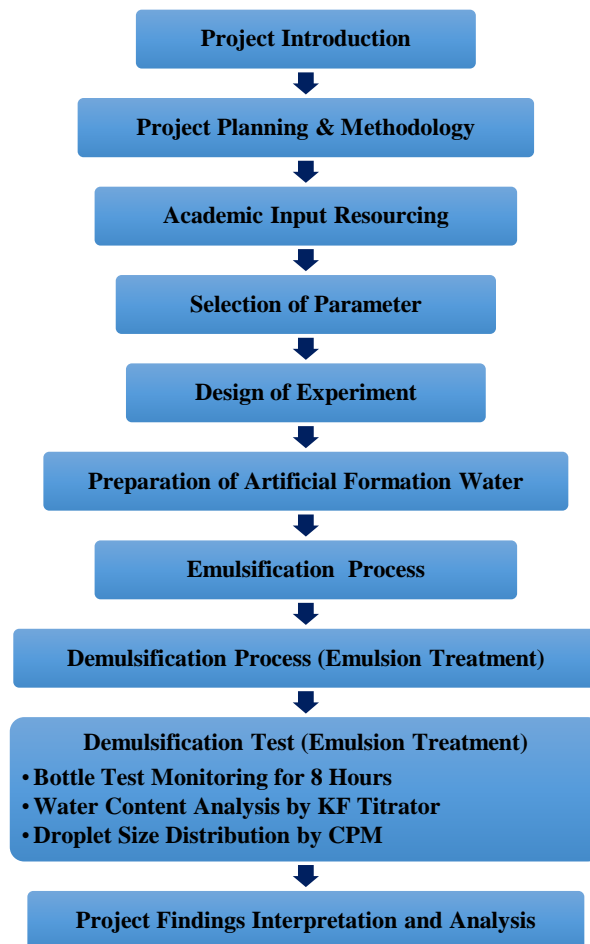


Figure 3.1: Project execution flowchart

According to project planning, there were twelve (12) crude samples that were studied with different parameter applied. Each sample have to undergo the whole process in project execution chart illustrated in Figure 3.1. The design of experiments for each sample are performed using Microsoft Excel spreadsheet to set up experiments conditions with respect to variables to be measured.

The preparation for experiments consisted of few aspects to be taken into accounted; which include the preparation of artificial formation water before mixed up with crude sample to form the emulsion in emulsification process. The artificial formation water is made up of ultra-pure water with several chemical composition explained in the next experimental design specification section. Following the completion of the preparation scopes, the demulsification or emulsion treatment process will be conducted by using in-house demulsification test equipment. Upon completion of demulsification process, the bottle test monitoring will be performed for eight (8) hours to measure the emulsion separation of the emulsion samples. Observation or measurement on the bottle samples was conducted at selected time intervals. After the bottle test completed, the samples was tested with Karl Fischer titrator equipment to determine the water content percentage in emulsion layer for several intervals. Then, the samples have to undergo droplet size distribution test by using cross – polarized microscope (CPM) to determine the water droplet mean size at desired intervals.

The results and outcomes of these tests and experiments were analysed and interpreted for two times; which are findings for the each phases and overall findings for both phases. The findings for each phases involved the data gathering, compilation and immediate result interpretation. While overall findings for both phases involved full analyses which cover the results of all experiments conducted in the project.

In addition, the project was performed as according to international standards including referring to API 12L – Specifications for Vertical and Horizontal Emulsion Treater and API 12J – Specifications for Oil and Gas Separators. This compliance to the standards will provide the reliability of the project experimental results to be acceptable for industrial applications.

3.2 Project Gantt chart and Key Milestones

The project Gantt chart and the key milestones for Final Year Project (FYP) entitled ‘Effect of Agitated Turbulence on Demulsification of Crude Oil Emulsions in Production Separator’ was separated in two sections, FYP I timeline and FYP II timeline presented in Figure 3.2 and Figure 3.3 respectively. The star-shaped indicates the key milestones for any submission required by FYP coordinator or project achievements. The milestones are summarized in Table 3.1.

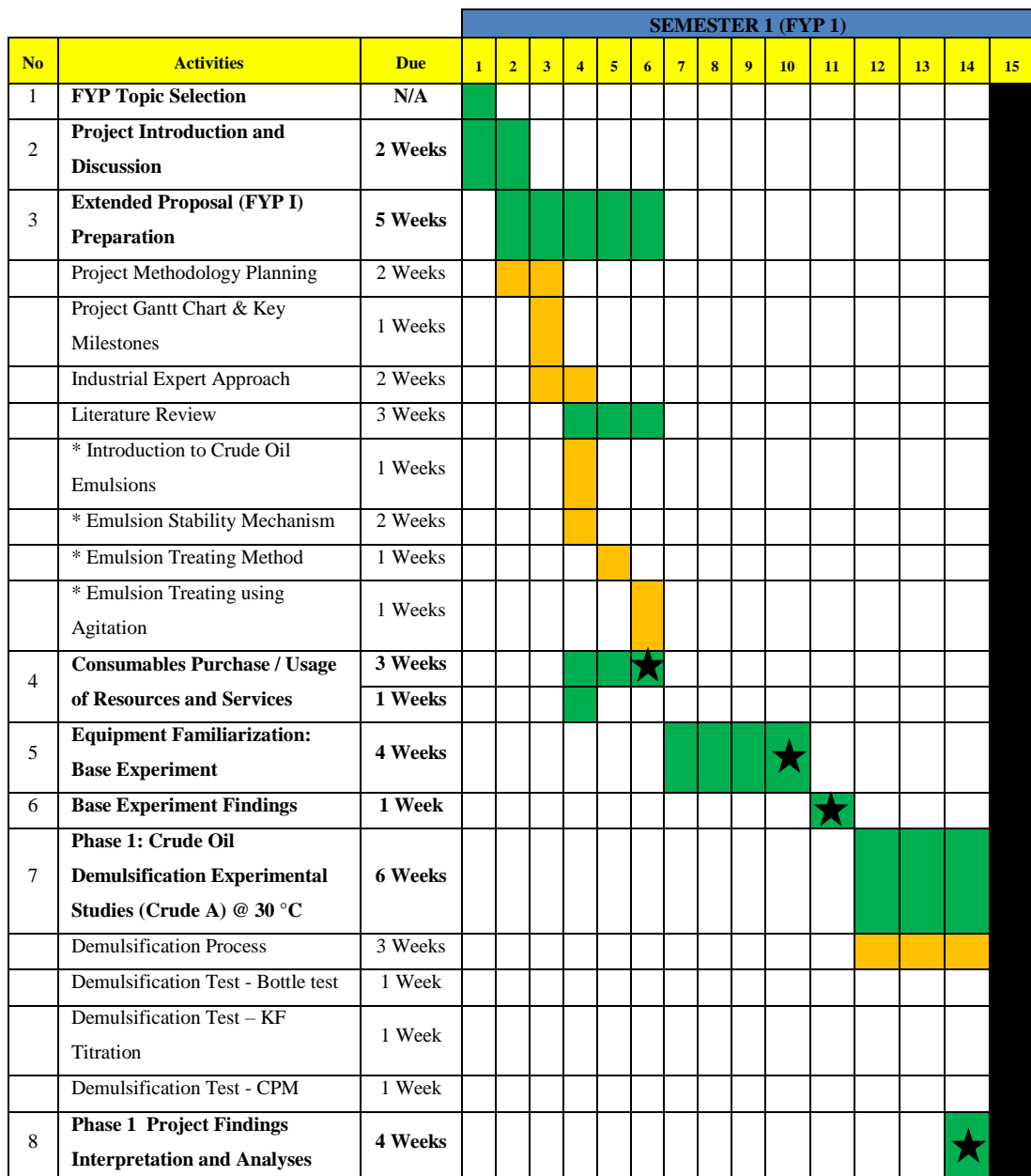


Figure 3.2: FYPI Timeline

		SEMESTER 2 (FYP 2)															
No	Activities	Due	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Phase 1: Crude Oil Demulsification Experimental Studies (Crude A) @ 30 °C (Cont.)	6 Weeks	█	█	█★												
	Demulsification Test – Bottle Test	1 Week	█														
	Demulsification Test – KF Titration	1 Week		█													
	Demulsification Test – CPM	1 Week			█												
2	Phase 2: Crude Oil Demulsification Experimental Studies (Crude A) @ 50 °C	6 Weeks				█	█	█	█	█	█★						
	Demulsification Process	3 Weeks				█	█	█									
	Demulsification Test - Bottle test	1 Week							█								
	Demulsification Test – KF Titration	1 Week								█							
	Demulsification Test - CPM	1 Week									█						
3	Phase 2 Project Findings Interpretation and Analyses	1 Weeks										█★					
4	Overall Project Findings Interpretation and Analyses	3 Weeks										█	█★				

Figure 3.3: FYP II Timeline

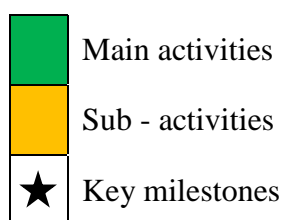


Table 3.1: Key Milestones

Key Milestone	Description
Equipment Familiarization: Base Experiment	Successfully performed the base experiment to acquire the familiarization on the equipment for Phase 1 and Phase 2 studies.
Base Experiment Findings	Successfully analysed the base experiment which performed without the emulsion treatment applied.
Consumable Purchase	Successfully purchased the consumable that were needed for the research studies
Phase 1: Crude Oil Demulsification Experimental Studies on RE-110 Crude at 30°C	Successfully performed the studies on demulsification process of RE-110 crude at 30°C and evaluating the behaviour of the emulsion separation during FYP1
Phase 1 Project Findings Interpretation and Analyses	Successfully analysed interpretation of the project finding from the Phase 1 studies by using several of analytical test.
Phase 2: Crude Oil Demulsification Experimental Studies on RE-110 Crude at 50°C	Successfully performed the studies on demulsification process of RE-110 crude at 50°C and evaluating the behaviour of the emulsion separation during FYP2
Phase 2 Project Findings Interpretation and Analyses	Successfully analysed interpretation of the project finding from the Phase 2 studies by using several of analytical test.
Overall Project Findings Interpretation and Analyses	Successfully analysed overall interpretation of the project finding by using several of analytical test.

3.3 Experimental Design Specifications

3.3.1 Crude Oil Samples

The crude samples used in this research were the RE-110 crude from Terengganu Crude Oil Terminal (TCOT) in Kerteh, Terengganu. The crude samples were provided and supplied by Petronas Carigali Sdn Bhd (PCSB) for researches purpose. The detailed information on the crude samples properties were illustrated in Table 3.2. The details in the crude oil was used to select the parameter on the variables and calculate the agitation speed for the selected temperature which is provided in Appendix II.

Table 3.2: Crude oil specifications

Crude Oil Origin	RE-110 from Terengganu Crude Oil Terminal (TCOT), Kerteh, Malaysia
Wax Appearance Temperature (WAT)	23.43°C
Pour Point Temperature	12°C
Density	808.65 kg/m ³ @ 30°C 793.49 kg/m ³ @ 50°C
API	40.91963 (Very light)
Viscosity	0.003581 Pa.s @ 30°C 0.002327 Pa.s @ 50°C

3.3.2 Preparation of Artificial Formation Water

The artificial formation water was prepared using selected agitation speed at specific operating temperature with selected chemical composition within fixed duration. The artificial formation water was used to mix along with crude oil according to the water-to-oil ratio of 30:70. The speed of the homogenizer is fixed at 400 rotation per minute for duration of 1 hour. The type of water used for the artificial formation water was ultra-pure water along with specific concentration of chemical substances stated in Table 3.3. The ultra-pure water is dispense from the PURELAB flex 4 as shows in the Figure 3.4. The water source entered for purification in the PURELAB flex 4 was directly from feed water. The purified water filtered particles with at least 0.2 μm and having effectively natural pH.

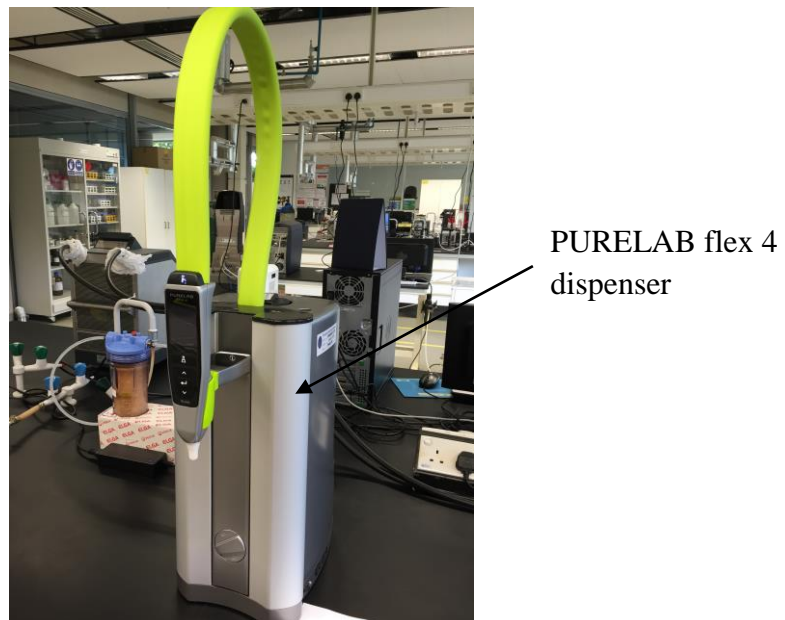


Figure 3.4: PURELAB flex 4

The dispensed purified water was then mixed together with the selected chemical substance shown in the Table 3.3, to replicate the formation water in the oil fields. The concentration of chemical substance used are the most common chemical composition used to replicate the formation water.

Table 3.3: Selected composition of artificial formation water

Substances	Concentration (g/L)
Sodium Bicarbonate, NaHCO_3	5.1260
Potassium Chloride, KCl	0.2646
Sodium Chloride, NaCl	6.0114
Barium Chloride Dihydrate, $\text{BaCl}\cdot 2\text{H}_2\text{O}$	0.0067
Strontium Chloride Hexahydrate, $\text{SrCl}_2\cdot 6\text{H}_2\text{O}$	0.0141
Magnesium Chloride Hexahydrate, $\text{MgCl}\cdot 6\text{H}_2\text{O}$	0.0750
Calcium Chloride Dihydrate, $\text{CaCl}\cdot 2\text{H}_2\text{O}$	0.2344

3.3.3 Emulsification Process

The emulsification process is the mixing process of artificial formation water and crude oil to form crude oil emulsion. The water cut was set at 30%, thus the water-to-oil ratio is 30:70. The water cut was set at 30% because it is the most common water cut production in most oil fields. The total liquid volume per sample was fixed at 50 ml, which is 35 ml for crude oil and 15 ml for artificial formation water. The artificial formation water and crude oil was mixed into the 100 ml beaker for the emulsification process. The duration for the emulsification process was set at 15 minutes and mixing temperature at 60°C to achieve desired stable crude oil emulsion.

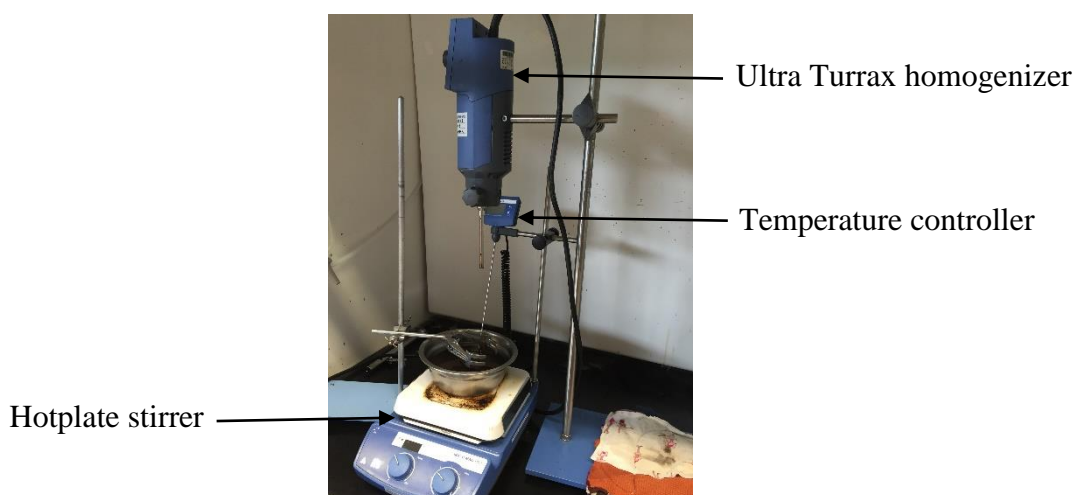


Figure 3.5: Equipment arrangement for emulsification process

The mixing temperature was controlled and maintained by using the IKA ETS – D5 temperature controller. The heating element used to supply the heat was the IKA C-MAG HS 7 hotplate stirrer. The mixing agitation speed was fixed at 12,000 rpm by using the IKA T25 Ultra Turrax Digital Homogenizer. The completed equipment arrangement for emulsification process is shown in the Figure 3.5.

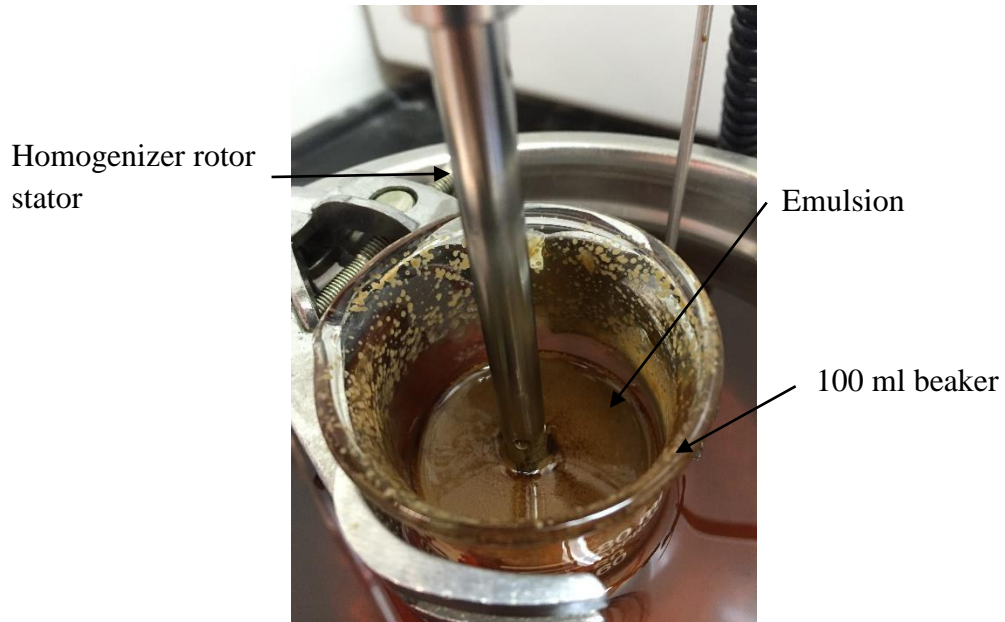


Figure 3.6: Brownish emulsion formation during emulsification process

The emulsion was assumed to be stable as they formed a brownish solution due to the mixing of the artificial formation water and crude oil sample as shown in Figure 3.6. As soon as the 15 minutes duration completed, the emulsion samples were then placed into the bottle test and ready for the emulsion treatment or demulsification process.

3.3.4 Emulsion Treatment or Demulsification Process

After the emulsification process, the emulsion was treated at varying agitation energy under constant temperature within specified duration. Before conducting the demulsification process, there are several calculation that need to be calculated.

As stated before in the literature review, Warren (2007) suggested that keeping the turbulence or agitation energy to its moderate Reynolds number of 50,000 to 100,000 can achieves good coalescing conditions. Thus, Reynold number of 50,000, 75,000 and 100,000 Re were selected for the experimental parameter. In order to convert the Reynolds number to rotational speed, several calculation need to be calculated. According to Kiss et al. (2011), the Reynolds numbers can be converted to velocity of the flow using the Reynolds number, N_{RE} by:

$$N_{RE} = \frac{\rho VD}{\eta} \quad (3.1)$$

where ρ is the density of crude oil, V is the velocity of the flow, D is the diameter of the production separator, and η is the dynamic viscosity of crude oil.

According to Potter and Wiggart (2008), the mass flow rate of the in the separator can be defined by calculating the multiplication of density, velocity of the flow and cross-sectional area. The mass flow rate, \dot{m} equation can be expressed by:

$$\dot{m} = \rho VA \quad (3.2)$$

where ρ is the density of crude oil, V is the velocity of the flow, A is the cross – sectional area of the production separator.

As crude oil moves from another to another, it will experiences an acceleration or deceleration. According to Newton’s second law of motion, the net force acting on crude oil can be expressed by:

$$F = ma = \dot{m}V \quad (3.3)$$

where F is the net force exerted, m is the mass of crude oil, a is the acceleration or deceleration, \dot{m} is the mass flow rate of crude oil, V is the velocity of crude oil flow.

The work, W can be calculated by:

$$\text{Work} = F \times D \quad (3.4)$$

where F is the net force in kgm/s^2 acquired from equation (3.4) and D is the length of the separator in meter.

The agitation speed, N of the rotational device used to quantify the amount of agitation can estimated the kinetic energy produced in the separator by:

$$KE = \frac{1}{2} M_s \left(\frac{2\pi N}{60} \times R_a \right)^2 \quad (3.5)$$

where kinetic energy, KE is assumed to be work, W in kgm^2/s^2 , M_s is the mass of the agitator, N is the rotational speed in rpm and R_a is the radius of arm for the agitator.

Opawale and Osisanya (2013) suggested that the agitation speed to be scale down for application of the agitator in the laboratory due to difference of experience on the real field and the laboratory equipment.



Figure 3.7: IKA KS 260 Basic

The IKA KS 260 Basic as shown in Figure 3.7, was used as the agitator that has the agitation speed range of 0 to 500 rpm, thus the agitation speed calculated have to be scale down to meet the specification of the agitator. The agitation speed are round off to nearest increment of 50 due to agitator speed scale are designated with increment of 50 from 0 to 500 rpm. The agitator was placed in the incubator to supply and maintain the operating temperature of the emulsion treatment. Figure 3.8 shows the Memmert IPP 110 Peltier-cooled Incubator that were used in the emulsion treatment process.



Figure 3.8: Memmert IPP 110 Peltier-cooled incubator

Based on the spreadsheet calculation shown in the Appendix 1, the agitation speed was differed with varying treatment temperature. At treatment temperature of 30°C, the agitation speeds were 50, 200 and 250 and classified as low, medium and high magnitude respectively. At treatment temperature of 50°C, the agitation speeds were 50, 100 and 150 rpm and were categorized as low, medium and high magnitude respectively. The treatment duration used for crude oil samples are at two duration, 10 and 30 minutes. The agitation speed for RE-110 crude at temperature of 30° and 50°C used to conduct the experiment was selected from the Table 3.4 and Table 3.5 respectively. The detailed data of the calculation using Microsoft Excel spreadsheet shown in the Appendix II.

Table 3.4: Agitation speed for RE-110 crude at T = 30°C

Sample No	Agitation Rate, Re	Fluid Density, kg/m³	Fluid Viscosity, Pa. s	Agitation Speed, RPM
1	50000	808.65	0.003581	50
2	75000	808.65	0.003581	200
3	100000	808.65	0.003581	250

Table 3.5: Agitation speed for RE-110 crude at T = 50°C

Sample No	Agitation Rate, Re	Fluid Density, kg/m³	Fluid Viscosity, Pa. s	Agitation Speed, RPM
1	50000	793.49	0.002327	50
2	75000	793.49	0.002327	100
3	100000	793.49	0.002327	150

3.3.5 Bottle Test Monitoring

After the emulsion treatment or demulsification process completed, the bottle test monitoring was conducted immediately to monitor the emulsion separation within 8 consecutive hours. The purpose of the bottle test monitoring was to monitor the emulsion separation under gravity settling for specified duration. The bottle test monitoring monitored the emulsion separation at 5th min, 15th min, 30th min, 1st hour, 2nd hour, 4th hour, 6th hour and 8th hour, which sum up to 8 consecutive hours of emulsion separation. The bottle test was placed into the universal oven to maintain the desired temperature. Figure 3.9 shows the Memmert U55 Universal Oven that was used during the bottle test monitoring to maintain the operating temperature. The bottle test temperature was fixed at 60°C. The temperature 60°C was used because it is the most common operating temperature in the production separator for the emulsion settling process. The bottle test monitoring contributed to the emulsion stability behaviour of crude oil emulsions which will be explained on the next chapter, results and discussion. Stable emulsion contributed to longer emulsion separation time while, less stable emulsion will need shorter emulsion separation time to resolve to its respective phases of crude oil and water. The lower the stability of emulsion, the shorter the emulsion separation time needed, which is the desired results for the emulsion separation.



Figure 3.9: Memmert U55 Universal Oven

The bottle test monitoring is the process of gravity settling for the emulsion. As the demulsification process or emulsion treatment applied, the water droplets started to coalesce with each other into a bigger water droplets. The gravity settling process is the process where the bigger water droplets settled from the emulsion phase through its density tendencies under static conditions.

3.3.6 Water Content Analysis

The determination of the amount of water in crude oil and petroleum products is very important. The water determination used in this research studies was based on the titration of the oil layer and emulsion layer with the Karl Fischer Titrator. The standard that was used during the water content analysis is ASTM D-4928. Figure 3.10 shows the Mettler Toledo V30 Karl Fischer Titrator that was used for the water content analysis. This method has been used for many years to determine the water content in liquid petroleum products (Mohajer, 2015). The Karl Fisher method is four times more accurate than centrifuge and about two times more accurate than distillation. The standard of oil required by the industry should contain water content below 0.5% to be considered as satisfactory.



Figure 3.10: Mettler Toledo V30 Karl Fischer Titrator

The oil and emulsion layer was collected after the treatment and bottle test to study the water content at interval of these processes. These samples were then placed at the tray on the Karl Fisher titrator for water content analysis. One samples needed about 1 hour approximately to complete.

3.3.7 Droplet Size Distribution

The droplet size distribution test was conducted to capture the microscopic image of the of the emulsion layer after treatment applied and after the bottle test monitoring, to measure the size and distribution of water droplets in the emulsion layer by using the cross – polarized microscope (CPM). This test was required to study how the varying parameter affect the water droplet size distribution of emulsion samples. Bigger water droplets size increases the higher possibility for the water droplets to settle from the emulsion layer. Figure 3.11 shows the Olympus BX53-P Cross-Polarized Microscope that was used to conduct the droplet size distribution test.



Figure 3.11: Olympus BX53-P Cross-Polarized Microscope

Figure 3.12 shows the software interface that was used to measure the size of water droplets in the emulsion layer after treatment applied and after 8 hours of bottle test monitoring. The size of water droplets in the emulsion after treatment applied and after 8 hours bottle test monitoring were compared to analyses the differences in water droplets size at the interval of these processes. All water droplets in the emulsion layer microscopic image were measured as much as possible to get more accurate mean size of water droplets in micron meter (μm). Figure 3.12 shows the example of measured water droplets size through the imaging software that was used to acquire cumulative of water droplets mean size. Each droplets were measured in the image captured to acquire the mean size of the water droplets.

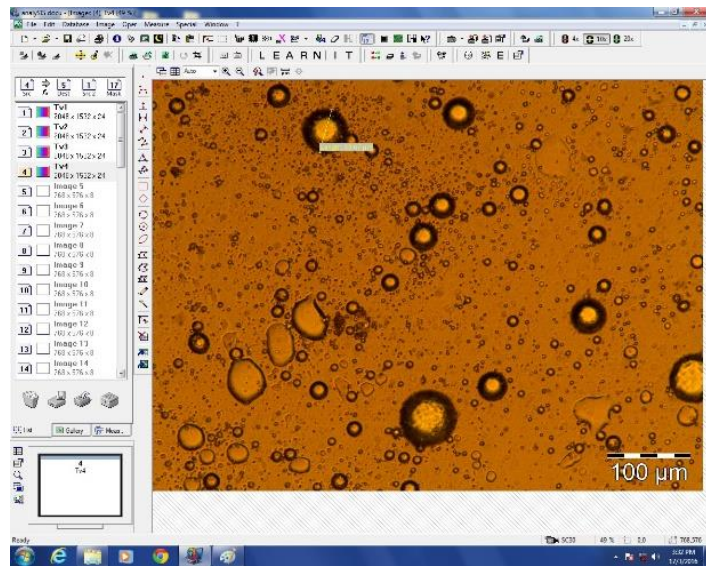


Figure 3.12: Measured water droplet through imaging software

3.3.8 Summary on Experimental Studies

The experimental design specifications for RE-110 Crude was summarized in Table 3.6 on its processes. Each samples needed to undergo every process to acquire completed analysis on the varying parameter applied on the emulsion samples. The emulsion samples were prepared with same parameter until the emulsification process but then tested with differing parameter during the demulsification process.

Table 3.6: Summary on experimentation for RE-110 Crude

Properties	RE-110
Wax Appearance Temperature (WAT)	23.43 °C
Total Liquid Volume per Sample	50 mL
Water-to-Oil Ratio	30:70
RE-110 Crude - Specifications	
Artificial Produced Water	Prepare the artificial produced water with the speed of homogenizer of 400 rpm at absolute temperature using the selected composition within an hour
Emulsification Process	Mix the artificial produced water with the selected crude at 12,000 rpm at 60 °C for 15 minutes
Demulsification Process	Treat the emulsion under varying agitation rates at different temperature for 10 and 30 minutes.
	Agitation rates of 50, 200 and 250 at 30 °C
	Agitation rates of 50, 100 and 150 rpm at 50 °C
Bottle Test Monitoring	Monitor the emulsion separation at 5 th min, 15 th min, 30 th min, 1 st hour, 2 nd hour, 4 th hour, 6 th hour and 8 th hour

CHAPTER 4

RESULTS AND DISCUSSION

In this chapter, the results and discussion of the project findings based on the methodology outlined in the previous chapter are presented. This chapter also includes the results and discussion for RE-110 demulsification studies.

The experiment was started with the emulsification process of Crude A at 60°C. As the emulsification process completed, the treatment for the emulsion was applied by using varying agitation speed in rpm at operating temperature at 30°C and 50°C. The agitation speed was calculated and scale down to appropriate agitation speed in the previous chapter. The emulsion samples were undergone 8 hour of bottle test. In this chapter, the emulsion samples were labeled as R1 until R12 and the detailed information for each emulsions is provided in Appendix I section.

4.1 Emulsion Stability

The stability of emulsions was studied using the conventional bottle test. As mentioned in the previous section of methodology, the bottle test was conducted for 8 consecutive hours with the readings taken for every 5th minutes, 15th minute, 30th minute, 1st hour, 2nd hour, 4th hour, 6th hour and 8th hour. The results acquired from the bottle test shows the tightness of the emulsion. Stable emulsion required a specific duration to completely separate the oil and water phase. The longer the time to separate the emulsion, the higher the kinetic stability of the emulsion.

The experiment conducted focuses on the emulsion separation into respective phases of crude oil and water. The volume of each layer is measured to indicate stability of the emulsion sample.

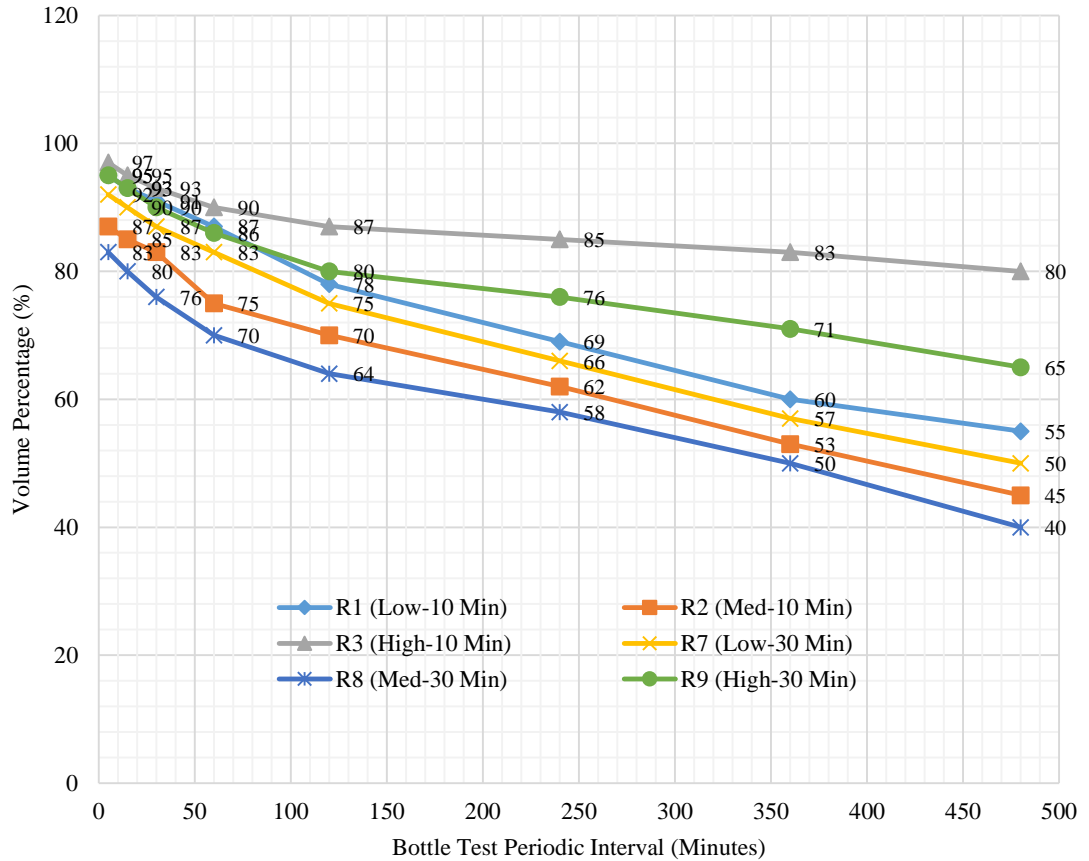


Figure 4.1: Temporal variation of volume percentage of emulsion layer at 30°C

The aim of the stability of the emulsion are to have an unstable emulsion which need shorter time to separate the emulsion to its respective crude oil and water. Stable emulsion needed more time to separate due to smaller dispersed water droplets that not be able to separate from the continuous phase of crude oil through gravity settling phase. From Figure 4.1, the samples treated at 30°C with medium magnitude of agitation speed have higher reduction of emulsion volume percentage compared to high and low magnitude of agitation speed. The low agitation speed might be too weak for the dispersed water droplets to coalesce with each other and inhibit the water droplets from settling by gravity to its respective phases. While, high magnitude of agitation speed might be too strong for the water to coalesce with another water droplets, and might re-emulsify to form smaller water droplets. However, medium magnitude of agitation speed gave a good coalescence medium for the dispersed water droplet to de-emulsify and settle from the emulsion. In emulsion treatment time, emulsion sample R8 which was treated with medium magnitude of agitation speed for 30 minutes, gave slightly higher reduction of emulsion volume percentage compared

to the emulsion sample R2, which was treated with medium magnitude of agitation speed for 10 minutes. Longer emulsion treatment duration contribute to high reduction in emulsion volume percentage as it gave more time for the dispersed water droplets to coalesce with each other and separate to its respective phases.

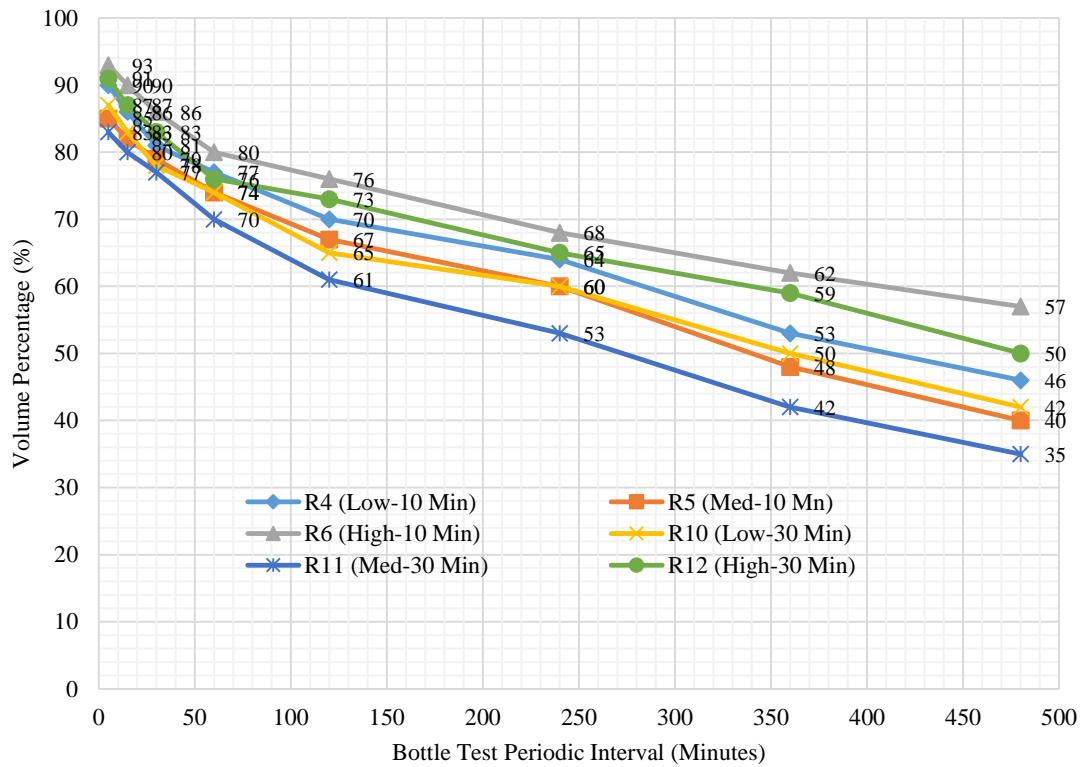


Figure 4.2: Temporal variation of volume percentage of emulsion layer at 50°C

Based on the Figure 4.2, the reduction of volume percentage of emulsion layer at 50°C gave higher reduction up to 65% of volume percentage of emulsion. Overall, emulsion samples treated with higher temperature gave higher reduction in emulsion compared to emulsion samples treated with lower temperature of 30°C as stated in Figure 4.1. Higher temperature promotes the destabilized effects across the interface of the water droplets caused by Brownian motion and mass transfer. The decrease in interfacial viscosity of the internal or discontinuous phase which is the water droplets, thus increase the momentum between the water droplets, induces coalescence between the water droplets and separate the discontinuous phase from the emulsion layer.

From Figure 4.2, the emulsion samples treated with medium agitation speed contribute to higher emulsion separation, range from 60 to 65% reduction of volume percentage of emulsion compared to emulsion samples treated with high and low

agitation speed for 10 and 30 minutes. As mentioned before, medium agitation speed induces good coalescence medium for the dispersed water droplets to coalesce, forming bigger water droplets size and settle from the emulsion layer. Longer emulsion treatment duration also contributes to higher reduction of volume percentage of emulsion as much as 5% compared to emulsion samples that was treated with lower emulsion treatment duration of 10 minutes. This due to longer emulsion treatment duration that gave the emulsion sample more time for the dispersed water droplets to coalesce with each other, thus having higher reduction of volume percentage of emulsion compared to emulsion sample treated for 10 minutes.

From the emulsion stability studies, it was concluded that emulsion sample that was treated with medium agitation speed at higher temperature for longer emulsion treatment duration gave higher reduction of volume percentage of emulsion.

4.2 Water Content Analysis

The Karl Fischer Titrator was used to analyse the water content in the emulsions after the agitation treatment was applied and after the completion of 8 hours bottle test. There are three layers that were taken into account for its water content. The first layer is the emulsion layer after the treatment using the agitation, the second is the oil layer after 8 hours bottle test and lastly is the emulsion layer after 8 hours bottle test.

4.2.1 Emulsion Layer after Treatment

The water content in the emulsion layer was investigated to study the percentage of water trapped in the emulsion layer after the emulsion treatment applied. The water content percentage was used as an indicator to measure the efficiency of the treatment for emulsion separation into its respective phases of oil and water.

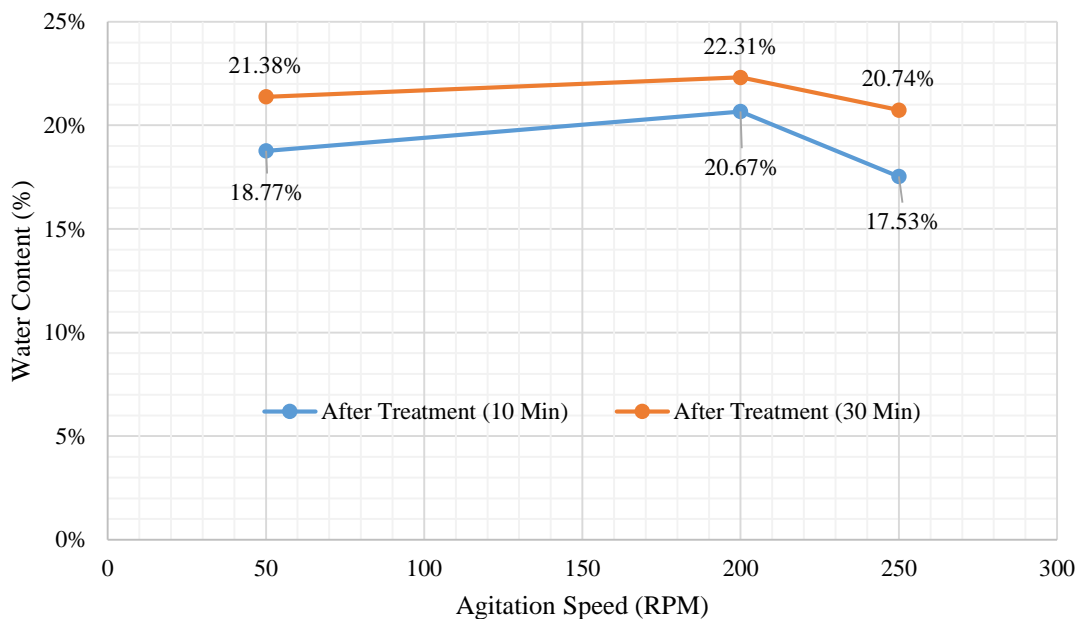


Figure 4.3: Variation of water content percentage in emulsion layer with agitation speed of 50, 200 and 250 RPM after treatment at 30°C

Figure 4.3 shown above is the water content percentage for the emulsion layer collected after the emulsion treatment was applied at low temperature of 30°C. Based on the figure, the water content percentage of emulsion samples treated with medium agitation speed appeared to be slightly higher than the emulsion samples treated with

low and high agitation speed. As neither one of the emulsion samples treated at 30°C formed water layer after the emulsion treatment, the dispersed water droplets were not able to separate from the rag layer to form the water layer, instead it only formed the oil layer. As no water layer formed after emulsion treatment, it caused the water content percentage for emulsion samples treated with medium agitation speed appears to be high, as it have the highest reduction of volume percentage of emulsion layer. The emulsion samples treated for longer emulsion treatment duration have higher water content percentage compared to the emulsion samples treated with shorter emulsion treatment duration.

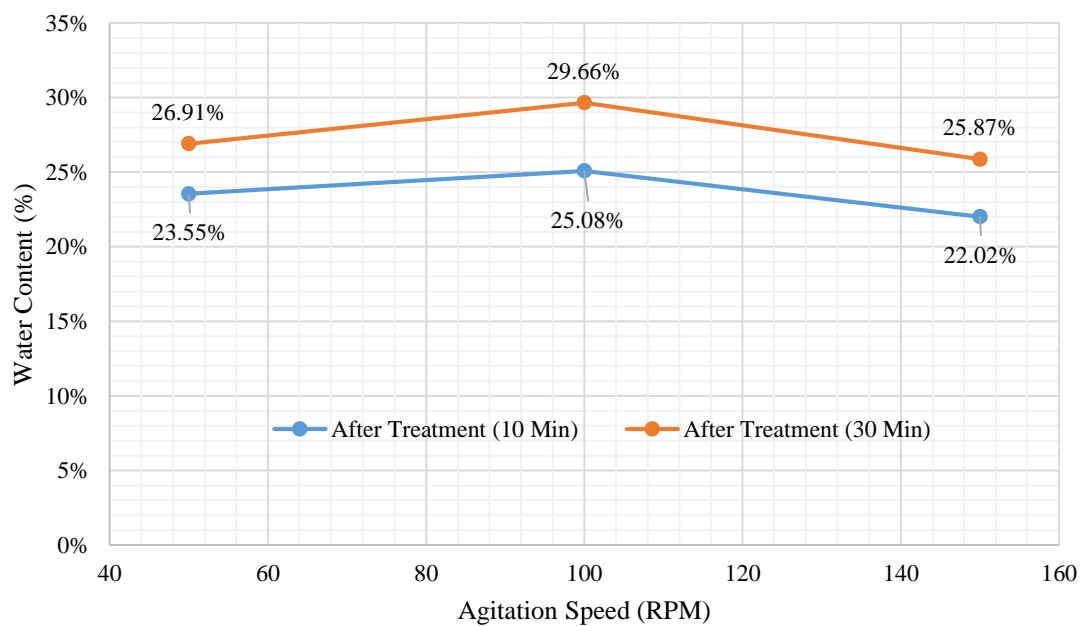


Figure 4.4: Variation of water content percentage in emulsion layer with agitation speed of 50, 100 and 150 RPM after treatment at 50°C

Similarly to Figure 4.3, the water content percentage of emulsion samples treated with medium agitation speed resulted higher water content percentage than the emulsion samples treated with low and high agitation speed. As mentioned before in the emulsion stability section, emulsion samples treated with high temperature of 50°C result in higher reduction of volume percentage of emulsion layer than the emulsion samples treated with low temperature of 30°C. Due to Brownian motion affect and mass transfer, emulsion samples appears to have higher reduction in volume percentage of emulsion layer. As there was no water layer formed after the emulsion treatment was applied, the dispersed water droplets were still in the emulsion layer

and resulted in higher water content percentage in the emulsion layer. Emulsion treatment duration also contribute largely on the water content percentage as longer emulsion treatment duration gave higher water content percentage compared to shorter emulsion treatment duration of 10 minutes. Longer emulsion treatment duration contribute to higher reduction of emulsion volume, however there was no water layer formed at the end of the emulsion treatment, thus resulted in higher water content percentage in the emulsion layer collected after the treatment.

Therefore, higher water content percentage was preferable in the emulsion layer as it can achieve higher reduction of emulsion when there was no water layer formed after the emulsion treatment was applied. Thus, the emulsion samples that having the highest water content percentage appeared to be having medium agitation speed, high temperature and longer emulsion treatment duration during the emulsion treatment.

4.2.2 Oil Layer after 8 Hours Bottle Test

The water content in oil layer after 8 hour bottle test was studied to examine the quality of oil produced after the emulsion separation. The most stable emulsion have higher water content in oil layer while the least stable emulsion have lower water content in oil layer. The standards of crude oil required by the industry should contain water content up to 0.5% to be consider as acceptable.

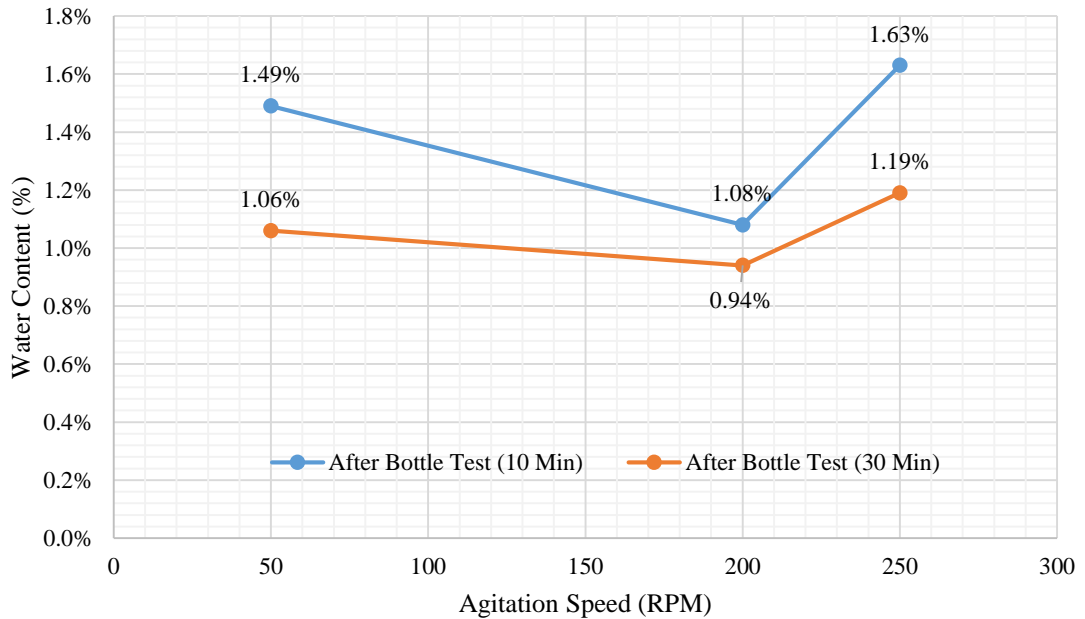


Figure 4.5: Variation of water content percentage in oil layer with agitation speed of 50, 200 and 250 RPM after 8 hours bottle test at 30°C

Based on the Figure 4.5, the water content percentage in oil layer for the emulsion samples treated with medium agitation speed showed lower water content percentage compared to emulsion samples treated with low and high temperature. Unlike to water content percentage in emulsion layer after emulsion treatment was applied, the water content percentage in oil layer showed otherwise. This oil layer that was separated from the emulsion layer only carried less dispersed water droplets during the gravity settling process of 8 hours for medium agitation speed as medium agitation speed gave good coalescence conditions for the dispersed water droplets and able to settle from the oil layer and moves to other phase according to its gravity or density tendencies. However, the emulsion samples treated for longer emulsion treatment duration of 30 minutes contributed much lower water content percentage

than the emulsion samples treated for shorter emulsion treatment duration of 10 minutes. Longer emulsion treatment duration contributed to higher possibility for the water droplets to settle from the oil layer and separated by the density of the phase. Contrarily, shorter emulsion treatment duration provided short time for the water droplets to settle from the oil layer and resulted in higher water content percentage compared to longer emulsion treatment duration applied. Thus, based on the standards required by the industry were not complied as the water content percentage in the oil layer in every emulsion samples exceed the 0.5% as mentioned before.

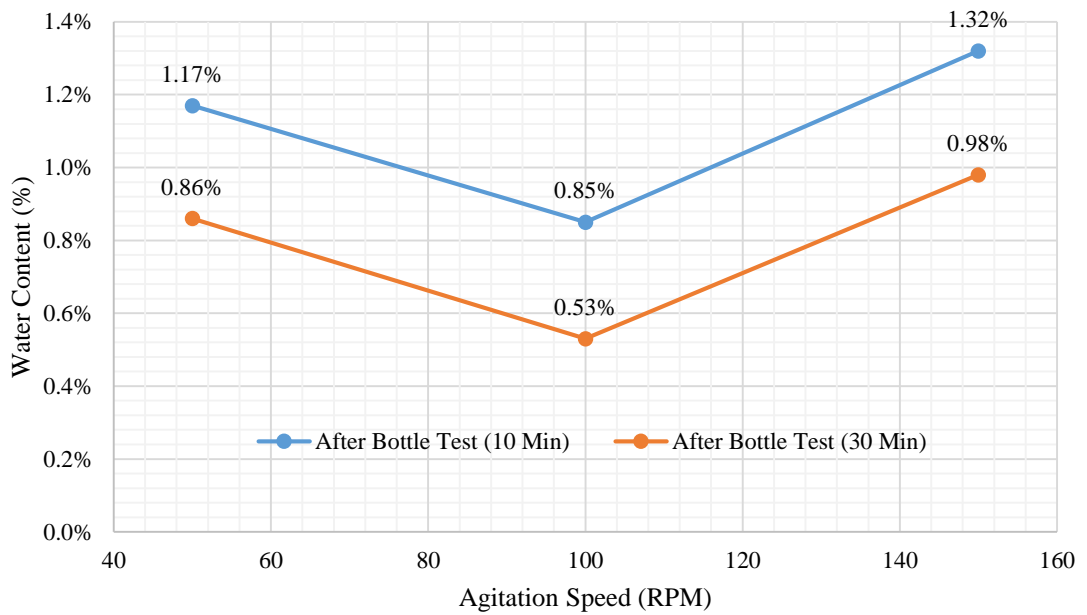


Figure 4.6: Variation of water content percentage in oil layer with agitation speed of 50, 100 and 150 RPM after 8 hours bottle test at 50°C

Figure 4.6 shows the water content percentage in oil layer taken after bottle test monitoring for 8 hours. Based on the figure, the emulsion samples treated with low and high agitation speed showed poor water content percentage as it produced high water content percentage more than the percentage required by the standards. Nevertheless, the emulsion samples treated with medium agitation speed contribute significant decreased in water content percentage and could be considered as good water content percentage as it nearly reached the water content percentage required by the standards. Medium agitation speed provided good coalescence conditions for the water droplets to separate from the oil layer because low agitation speed might be too weak for the water droplets to coalesce while high agitation speed are too strong

for coalescence and might even emulsify the water droplets into much smaller water droplets.

The emulsion samples treated with high temperature of 50°C gave much lower water content percentage when compared with the emulsion samples treated with low temperature of 30°C as illustrated in Figure 4.5. This emulsion behaviour could be explained as the effect of Brownian motion and mass transfer taken place to decrease the interfacial viscosity of the emulsion layer and assisted in separating the water droplets from the emulsion layer according to its gravity tendencies and formed the oil layer after 8 hours bottle test. This could be proved with the reduction of volume percentage of emulsion layer illustrated earlier in Figure 4.1 and Figure 4.2. Longer emulsion treatment also provided more duration for the emulsion samples to settle the emulsion layer to its respective phase of crude oil and water and resulted in much lower water content percentage in oil layer.

As a result for the oil layer taken after 8 hours bottle test, lower water percentage is more desirable as the maximum water content percentage needed is 0.5% from the oil layer. Therefore, the emulsion sample that acquired lower water content percentage was treated with medium agitation speed and high temperature for longer emulsion treatment duration.

4.2.3 Emulsion Layer after 8 Hours Bottle Test

The water content of the emulsion layer after 8 hours bottle test was investigated to analyse the percentage of water trapped in crude oil after the emulsion undergone 8 hour of bottle test. The water droplets need specific duration for the water droplets to settle from crude oil phase. The duration increase the possibility for water droplets to coalesce and settle to its respective phases of crude oil and water. The gravity settling and time can provide the most effective breakdown of the emulsion. The higher percentage in water content shows lower emulsion stability while lower percentage in water content shows higher emulsion stability.

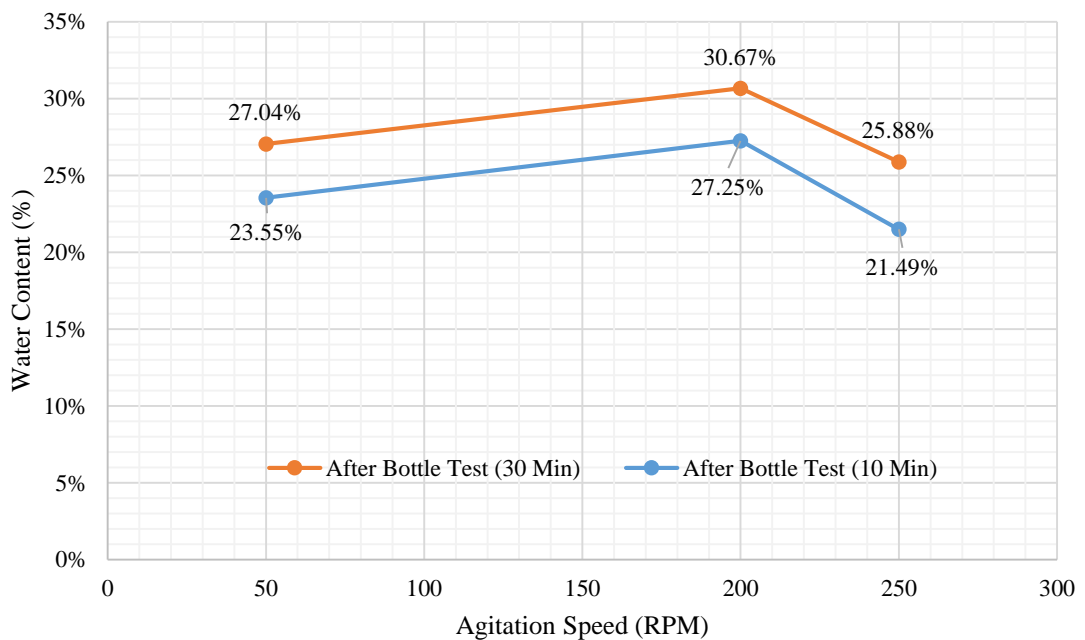


Figure 4.7: Variation of water content percentage in emulsion layer with agitation speed of 50, 200 and 250 RPM after 8 hours bottle test at 30°C

Figure 4.7 shown is the water content percentage in emulsion layer collected after 8 hours bottle test monitoring at low temperature of 30°C. Based on the analysis made from the figure above, the emulsion samples treated with medium agitation speed showed higher water content percentage compared to emulsion samples treated with low and high agitation speed. Medium agitation speed contributes good coalescence medium for the water droplets to coalesce and resulted in higher reduction of volume percentage of emulsions, but since there is no water layer formed after 8 hours bottle test, the water droplets are largely settled in the emulsion layer

that caused the water content percentage to be significantly higher than the emulsion samples treated with low and high agitation speed. In addition, emulsion treatment time also played an important role in water content percentage as emulsion samples treated with longer emulsion treatment duration achieved higher water content percentage than the emulsion samples treated with shorter emulsion treatment duration. The emulsion treatment duration is closely related to the stability of the emulsion, which can be measured with the reduction of volume percentage of emulsion. As longer emulsion treatment duration gave higher reduction of volume percentage of emulsion, thus it acquired higher water content percentage in emulsion layer as there is no water layer formed at the end of 8 hours bottle test.

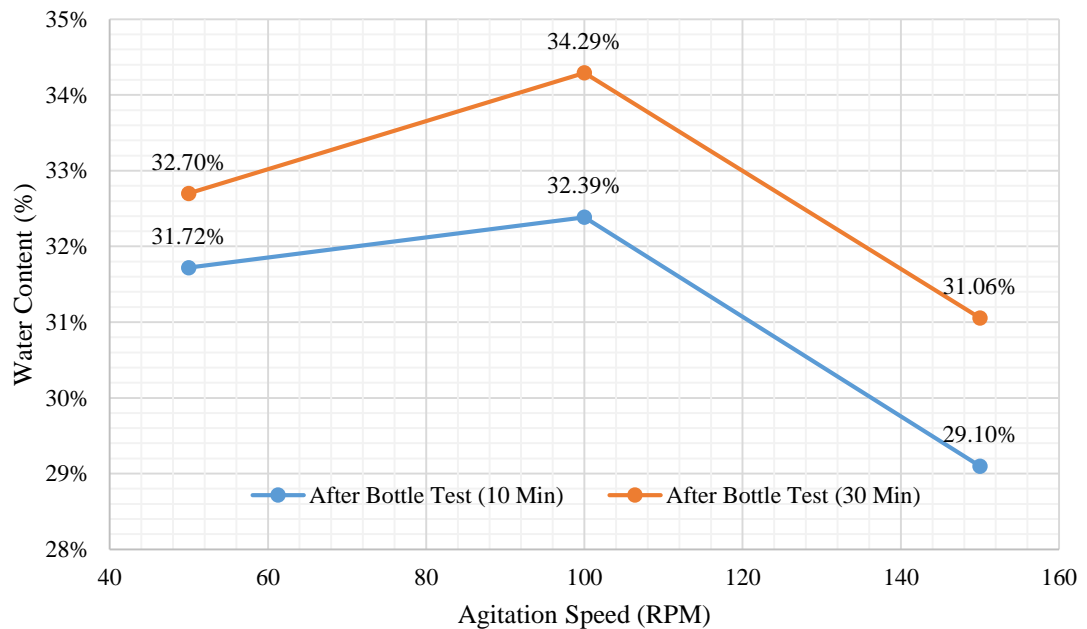


Figure 4.8: Variation of water content percentage in emulsion layer with agitation speed of 50, 100 and 150 RPM after 8 hours bottle test at 50°C

The water content percentage in emulsion layer collected after 8 hours of bottle test at 50°C was shown in Figure 4.8. Based on the figure, the emulsion samples treated with medium agitation speed have noticeably higher water content percentage followed by low agitation speed and high agitation speed having the least water content percentage. Medium agitation energy that was supplied to the emulsion samples that was enough to break the interfacial across the water droplets and assisted in promoting coalescence medium for the water droplets and settled from the emulsion. But, as mentioned before, there was no water layer formed in every

emulsion samples. Without formation of water layer proved that the emulsion layer collected after 8 hours of bottle test to have higher water content percentage as the water droplets could not settled from the emulsion to create the water layer.

On the other part, the temperatures also be significantly affect the water content percentage. In comparison between Figure 4.7 and Figure 4.8, the emulsion samples treated with higher temperature achieved higher water content percentage compared to the emulsion samples treated with lower temperature. Since the emulsion samples treated with higher temperature achieved higher reduction in volume percentage of emulsion and there was no water layer formed, the emulsion layer collected after the 8 hours bottle test should appears to be higher since the water droplets could be separate and form the water layer. As compared the emulsion treatment duration in Figure 4.8, the emulsion treated for longer emulsion treatment duration had higher water content percentage compared to emulsion samples treated for shorter emulsion treatment duration. Longer emulsion treatment duration gave higher reduction in volume percentage of emulsion compared to shorter emulsion treatment duration, thus the water droplets could not settled from the emulsion layer as there was no water layer formed and resulted in higher water content percentage.

In conclusion, for the emulsion layer taken after 8 hours bottle test, higher water content percentage is favorable in the emulsion layer as it can achieve higher reduction of emulsion when there was no water layer formed after the emulsion treatment was applied. Based on Figure 4.8, the emulsion samples treated with medium agitation speed, high temperature and longer emulsion treatment duration during the emulsion treatment have the highest water content percentage.

4.3 Droplet Size Distribution

The tabulation of water droplet size in the emulsion was conducted by acquiring the water droplet size distributions from the photomicrograph images obtained from the Cross-Polarized Microscopy (CPM) equipment. Each water droplet were measured using the software on it diameter to acquire the mean size of the water droplet of the emulsion layer for every after treatment and 8 hour bottle test. Higher water droplet mean size gave higher possibility for the water droplets to settle or separate from the continuous phase of crude oil through gravity settling of bottle test monitoring period of 8 hours.

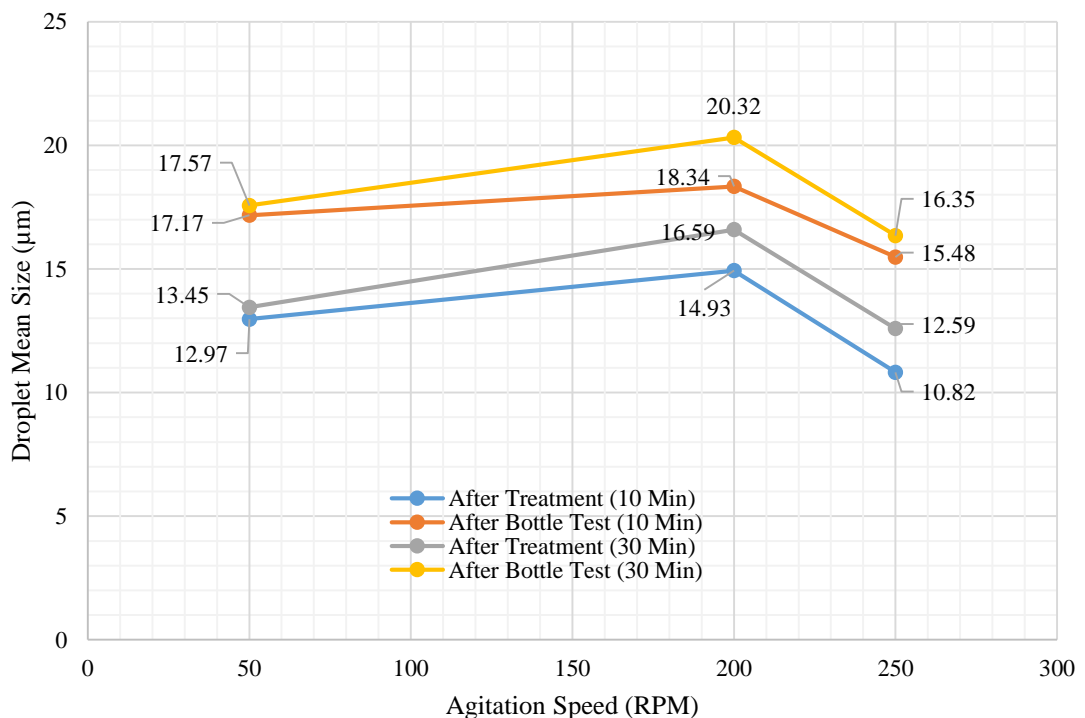


Figure 4.9: Water droplet size distribution at 30°C

Figure 4.9 shows the water droplet size distribution treated at 30°C for emulsion treatment duration of 10 and 30 minutes. From the figure, the emulsion samples treated with medium agitation speed have higher water droplets mean size than the emulsion samples treated with low and high agitation speed. Medium agitation speed contributes provides good coalescence conditions for water droplets to coalesce with each other. The coalescence water droplets create bigger droplet size and increase the possibility for the water droplets to settle from the emulsion to its own phases by density tendencies. The emulsion samples treated for longer emulsion

treatment duration have slightly higher water droplet mean size at intervals of after treatment and after 8 hours bottle test compared to emulsion samples treated for shorter emulsion treatment duration. Longer emulsion treatment duration gave more time for the water droplets to coalesce and create bigger water droplets size as shorter emulsion treatment duration gave less duration for the water droplets to coalesce, thus limit the coalescence between the water droplets.

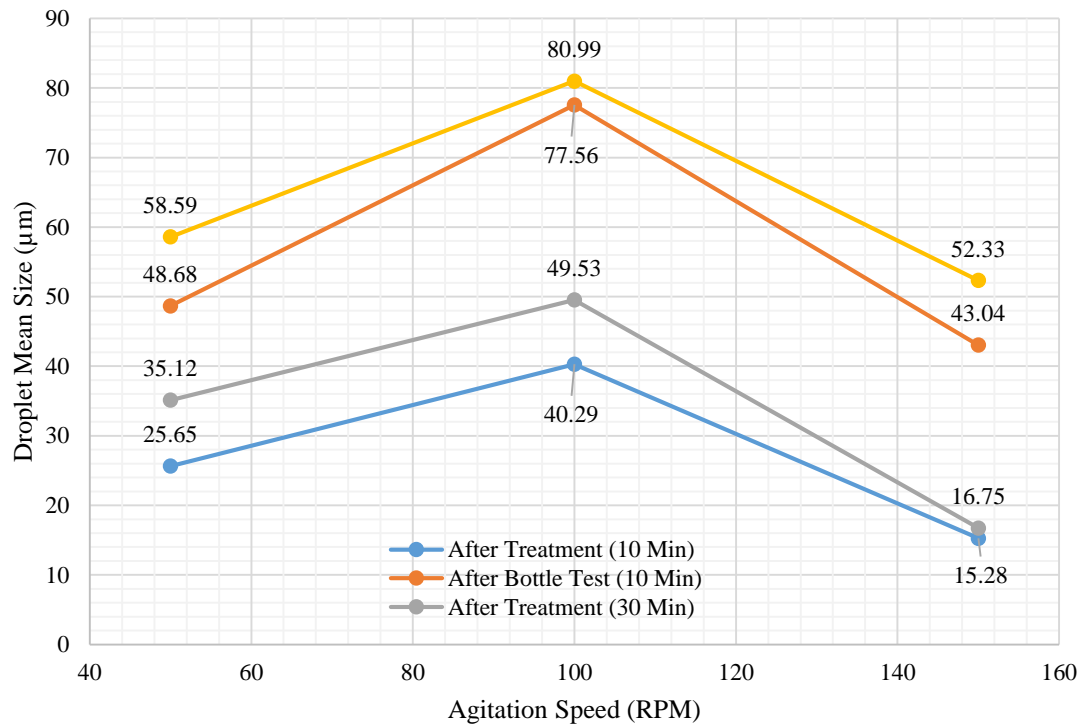


Figure 4.10: Water droplet size distribution at 50°C

Based on Figure 4.10, the water droplets size distribution at 50°C have higher range of droplet mean size which range about 15.28 to as much as 80.99 µm. While, the water droplet size distribution at 30°C contribute to much lower range from 10.82 to 20.32 µm . From the statement above, it can be concluded that higher temperature induces higher water droplet mean size compared to low temperature. As explained in previous section, high temperature applied during emulsion treatment can increases destabilization on the interface of the water droplets due to Brownian motion and mass transfer on the emulsion samples. High temperature reduced the viscosity of the emulsion layer and increases the momentum between the dispersed water droplets and create good coalescence medium. Bigger water droplets size increase the possibility for the dispersed water droplets to settle from the continuous phase of crude oil. Based

on Figure 4.10, the water droplets mean size treated for longer emulsion treatment duration appeared to be higher than emulsion samples treated for shorter emulsion treatment duration. As mentioned before, longer emulsion treatment provided more time for the water droplets to coalesce with each other but shorter emulsion treatment time gave less time for coalescence of water droplets, thus having much lower water droplets mean size.

Based on Figure 4.9 and Figure 4.10, higher water droplets mean size is preferable as higher water droplets size increase possibility for water droplets to settle from the emulsion layer. Thus, the emulsion samples that acquired higher water droplets mean size are treated with medium agitation speed and higher temperature for longer emulsion treatment duration.

CHAPTER 5

CONCLUSION & RECOMMENDATION

As a conclusion, the optimum operating conditions for emulsion stability studies in the laboratory condition were selected based on certain parameters. The parameter includes the maximum oil layer separated from the emulsion, minimum rag layer that could not be separated after 8 hours of bottle test monitoring and minimum water content in the oil layer that must be complied for the standards to consider it to be acceptable to the industry. These parameters can be concluded based on the outcomes of the experiment;

5.1 Emulsion Stability

The stability of emulsions was studied using the conventional bottle test. The volume of each layer is measured to indicate the stability of the emulsion sample. Stable emulsion required a specific duration to completely separate the oil and water phase. The longer the time to separate the emulsion, the higher the kinetic stability of the emulsion. Stable emulsion needed more time to separate due to smaller dispersed water droplets that not be able to separate from the continuous phase of crude oil through gravity settling phase while unstable emulsion need shorter time to separate the emulsion to its respective crude oil and water. Based on the analysis, the emulsion samples treated with medium agitation speed appears to have higher reduction in volume percentage of emulsion up to 65% while emulsion samples treated with low and high only reach maximum of 58% reduction in volume percentage of emulsion. Similarly, the emulsion samples treated with high temperature contributed to higher reduction in volume percentage of emulsion compared to emulsion samples treated with lower temperature. Higher temperature achieved higher range of reduction in volume percentage of emulsion from 43% to 65% while lower temperature resolved only 20% to 60% of volume percentage of emulsion. Longer emulsion treatment duration of 30 minutes contributes to higher reduction of volume percentage of emulsion as much as 5% compared to emulsion samples that was treated with lower emulsion treatment duration of 10 minutes. From the emulsion stability studies, it was

concluded that higher reduction in volume percentage of emulsion is preferable as it resolve more oil layer in the emulsion samples.

5.2 Water Content Analysis

The water content analysis was used to analyses the water content in the oil and emulsions after the agitation treatment was applied and after the completion of 8 hours bottle test. There were three layers that were taken into account for its water content. The first layer was the emulsion layer after the treatment using the agitation, the second was the oil layer after 8 hours bottle test and lastly was the emulsion layer after 8 hours bottle test.

For the emulsion layer after the emulsion treatment, the emulsion samples treated with medium agitation speed achieved higher water content percentage as much as 3.79% than the emulsion samples treated with low and high agitation speed. Emulsion samples treated with high temperature of 50°C result in water content percentage in emulsion layer than the emulsion samples treated with low temperature of 30°C. Emulsion treatment duration also contributed largely on the water content percentage as longer emulsion treatment duration gave higher water content percentage up to 4.58% compared to shorter emulsion treatment duration of 10 minutes.

However, the water content percentage in oil layer acted differently compared to emulsion layer. The emulsion samples treated with medium agitation speed contributed significant decrease at 0.53% in water content percentage and could be considered as good water content percentage as it nearly reached the water content percentage required by the standards, which is 0.5% water content percentage. The emulsion samples treated with high temperature of 50°C gave much lower water content percentage as much as 0.41% when compared with the emulsion samples treated with low temperature of 30°C. Longer emulsion treatment also provided more time for the emulsion samples to settle the emulsion layer to its respective phase of crude oil and water and resulted in much lower water content percentage in oil layer.

Similarly, the water content percentage in the emulsion layer taken after 8 hour bottle test was expected to have higher water content percentage as there was no water layer formed after the bottle test. However, the variables on agitation speed,

temperature and emulsion treatment duration affect the water content percentage for each samples. The emulsion samples treated with medium agitation speed have noticeably higher water content percentage followed by low agitation speed and high agitation speed having the least water content percentage. The emulsion samples treated with higher temperature achieved higher water content percentage compared to the emulsion samples treated with lower temperature. The emulsion treated for longer emulsion treatment duration had higher water content percentage compared to emulsion samples treated for shorter emulsion treatment duration. In conclusion for the emulsion layer taken after 8 hours bottle test, higher water content percentage is favorable in the emulsion layer as it can achieve higher reduction of emulsion when there is no water layer formed after the emulsion treatment was applied.

5.3 Water Droplet Size Distribution

The water droplet size in the emulsion was conducted by acquiring the water droplet size distributions from the photomicrograph images obtained from the cross – polarized microscopy. The mean size of the water droplet of the emulsion layer for every emulsion samples after treatment and 8 hour bottle test. Higher water droplet mean size gave higher possibility for the water droplets to settle or separate from the continuous phase of crude oil through gravity settling of bottle test monitoring of 8 hours period. Generally, the emulsion samples treated with medium agitation speed have higher water droplets mean size than the emulsion samples treated with low and high agitation speed. In contrast to the temperature, the water droplets size distribution at 50°C have higher range of droplet mean size which range about 15.28 to as much as 80.99 μm . While, the water droplet size distribution at 30°C contribute to much lower range from 10.82 to 20.32 μm . From the statement above, it can be concluded that higher temperature significantly induces higher water droplet mean size compared to low temperature. The water droplets mean size treated for longer emulsion treatment duration appears to be higher than emulsion samples treated for shorter emulsion treatment duration. As conclusion, higher water droplets mean size is preferable as higher water droplets size increase possibility for water droplets to settle from the emulsion layer.

Therefore, based on the studies conducted on the stability of emulsion, water content percentage and water droplet size distribution, the emulsion samples that have

higher reduction in volume percentage of emulsion, higher water content in emulsion layer and lower water content in oil layer, and higher water droplet size distribution is the most optimum operating conditions as it have maximum oil layer separated from the emulsion, minimum rag layer that could not be separated after 8 hours of bottle test monitoring and minimum water content in the oil layer that must be complied for the standards to consider it to be acceptable to the industry. Thus, it is concluded that the emulsion treated with medium agitation speed of 100 rpm at higher temperature of 50°C for longer emulsion treatment duration of 30 minutes is the best demulsification parameter for crude oil emulsion.

This research has been focused on the effect of agitated turbulence with limited parameter on operating temperature and emulsion treatment duration. Having said so, a more thorough research can be conducted on the effect of medium agitated turbulence on higher temperature more than 50°C and longer emulsion treatment duration more than 30 minutes. The characteristic of the water-in-oil emulsion can then be further analysed for a more comprehensive understanding of the relationship between high operating temperature and longer emulsion treatment duration on emulsion stability, water content percentage on emulsion and oil layer and water droplet size in the emulsion layer.

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Appendix I Details of crude Oil Emulsion Experimental Parameter

Sample Name	Crude Type	Temperature (°C)	Duration (minute)	Agitation Speed (RPM)	Magnitude
R1	RE-110	30°C	10	50	Low
R2	RE-110	30°C	10	200	Medium
R3	RE-110	30°C	10	250	High
R4	RE-110	50°C	10	50	Low
R5	RE-110	50°C	10	100	Medium
R6	RE-110	50°C	10	150	High
R7	RE-110	30°C	30	50	Low
R8	RE-110	30°C	30	200	Medium
R9	RE-110	30°C	30	250	High
R10	RE-110	50°C	30	50	Low
R11	RE-110	50°C	30	100	Medium
R12	RE-110	50°C	30	150	High

Appendix II Calculation of Agitation Speed of RE-110 Crude

Separator Data

Length of the separator, L (m)	3.048
Separator inner diameter (m)	9.144
Separator inner radius (m)	4.572
Cross-sectional area of separator (m ²)	65.80322

Agitation Data

Mass of Agitation (kg)	2.5
Radius of arm, Ra (m)	0.005

RE-110

Sample Name/ No.	Temp , C°	Duration , minute	Agitation rates, Re	Fluid Density, kg/m ³	Fluid Viscosity, Ns/m ² (Pa.s)	Fluid Speed, m/s	Mass Flow Rate, kg/s	Force, kgm/s ²	Work, kgm ² /s ²	Mass of sample (kg)	Agitation Speed, RPM	Scale down speed, RPM (1:230)	Speed, increment of 50
R1	30	10	50000	808.65	0.003581	0.0242	1,288.50	31.20	95.10	0.0404325	16,533.76	71.89	50
R2	30	10	75000	808.65	0.003581	0.0363	1,932.75	70.20	641.92	0.0404325	42,955.97	186.77	200
R3	30	10	100000	808.65	0.003581	0.0484	2,577.01	124.80	1,141.19	0.0404325	57,274.63	249.02	250
R4	50	10	50000	793.49	0.002327	0.0160	837.29	13.43	122.77	0.0396745	18,788.79	81.69	50
R5	50	10	75000	793.49	0.002327	0.0241	1,255.94	30.21	276.24	0.0396745	28,183.18	122.54	100
R6	50	10	100000	793.49	0.002327	0.0321	1,674.59	53.71	491.09	0.0396745	37,577.57	163.38	150

Sample No.	Temp, C°	Duration, minute	Agitation rates, Re	Fluid Density, kg/m ³	Fluid Viscosity, Ns/m ² (Pa.s)	Fluid Speed, m/s	Mass Flow Rate, kg/s	Force, kgm/s ²	Work, kgm ² /s ²	Mass of sample (kg)	Agitation Speed, RPM	Scale down speed, RPM (1:230)	Speed, increment of 50
R7	30	30	50000	808.65	0.003581	0.0242	1,288.50	31.20	95.10	0.0404325	16,533.76	71.89	50
R8	30	30	75000	808.65	0.003581	0.0363	1,932.75	70.20	641.92	0.0404325	42,955.97	186.77	200
R9	30	30	100000	808.65	0.003581	0.0484	2,577.01	124.80	1,141.19	0.0404325	57,274.63	249.02	250
R10	50	30	50000	793.49	0.002327	0.0160	837.29	13.43	122.77	0.0396745	18,788.79	81.69	50
R11	50	30	75000	793.49	0.002327	0.0241	1,255.94	30.21	276.24	0.0396745	28,183.18	122.54	100
R12	50	30	100000	793.49	0.002327	0.0321	1,674.59	53.71	491.09	0.0396745	37,577.57	163.38	150

Appendix III Emulsion Stability

Sample	Temp	Duration	Agitation Speed	Mag.	5	15	30	60	120	240	360	480
R1 (Low)	30	10	50	Low	95	93	91	87	78	69	60	55
R2 (Med.)	30	10	200	Medium	87	85	83	75	70	62	53	45
R3 (High)	30	10	250	High	97	95	93	90	87	85	83	80
R7 (Low)	30	30	50	Low	92	90	87	83	75	66	57	50
R8 (Med.)	30	30	200	Medium	83	80	76	70	64	58	50	40
R9 (High)	30	30	250	High	95	93	90	86	80	76	71	65

Sample	Temp	Duration	Agitation Speed	Mag.	5	15	30	60	120	240	360	480
R4 (Low)	50	10	50	Low	90	86	81	77	70	64	53	46
R5 (Med)	50	10	100	Medium	85	82	79	74	67	60	48	40
R6 (High)	50	10	150	High	93	90	86	80	76	68	62	57
R10 (Low)	50	30	50	Low	87	83	78	74	65	60	50	42
R11 (Med)	50	30	100	Medium	83	80	77	70	61	53	42	35
R12 (High)	50	30	150	High	91	87	83	76	73	65	59	50

Appendix IV Water Content Analysis

Sample Name	Temp	Duration	Agitation Speed	Mag.	Water Content (%)		
					Emulsion Layer	Oil Layer	Emulsion Layer
					After Treatment (10 Min)	After Bottle Test (10 Min)	After Bottle Test (10 Min)
R1	30	10	50	Low	18.77%	1.49%	23.55%
R2	30	10	200	Medium	20.67%	1.08%	27.25%
R3	30	10	250	High	17.53%	1.63%	21.49%
Sample Name	Temp	Duration	Agitation Speed	Mag.	Water Content (%)		
					Emulsion Layer	Oil Layer	Emulsion Layer
					After Treatment (30 Min)	After Bottle Test (30 Min)	After Bottle Test (30 Min)
R7	30	30	50	Low	21.38%	1.06%	27.04%
R8	30	30	200	Medium	22.31%	0.94%	30.67%
R9	30	30	250	High	20.74%	1.19%	25.88%

Sample Name	Temp	Duration	Agitation Speed	Mag.	Water Content (%)		
					Emulsion Layer	Oil Layer	Emulsion Layer
					After Treatment (10 Min)	After Bottle Test (10 Min)	After Bottle Test (10 Min)
R4	50	10	50	Low	23.55%	1.17%	31.72%
R5	50	10	100	Medium	25.08%	0.85%	32.39%
R6	50	10	150	High	22.02%	1.32%	29.10%
Sample Name	Temp	Duration	Agitation Speed	Mag.	Water Content (%)		
					Emulsion Layer	Oil Layer	Emulsion Layer
					After Treatment (30 Min)	After Bottle Test (30 Min)	After Bottle Test (30 Min)
R10	50	30	50	Low	26.91%	0.86%	32.70%
R11	50	30	100	Medium	29.66%	0.53%	34.29%
R12	50	30	150	High	25.87%	0.98%	31.06%

Appendix V Droplet Size Distribution

Sample Name	Temp	Duration	Agitation Speed	Mag.	Droplet Mean Size (μm)	
					After Treatment (10 Min)	After Bottle Test (10 Min)
R1	30	10	50	Low	12.97	17.17
R2	30	10	200	Medium	14.93	18.34
R3	30	10	250	High	10.82	15.48
Sample Name	Temp	Duration	Agitation Speed	Mag.	Droplet Mean Size (μm)	
					After Treatment (30 Min)	After Bottle Test (30 Min)
R7	30	30	50	Low	13.45	17.57
R8	30	30	200	Medium	16.59	20.32
R9	30	30	250	High	12.59	16.35

Sample Name	Temp	Duration	Agitation Speed	Mag.	Droplet Mean Size (μm)	
					After Treatment (10 Min)	After Bottle Test (10 Min)
R4	50	10	50	Low	25.65	48.68
R5	50	10	100	Medium	40.29	77.56
R6	50	10	150	High	15.28	43.04
Sample Name	Temp	Duration	Agitation Speed	Mag.	Droplet Mean Size (μm)	
					After Treatment (30 Min)	After Bottle Test (30 Min)
R10	50	30	50	Low	35.12	58.59
R11	50	30	100	Medium	49.53	80.99
R12	50	30	150	High	16.75	52.33