Waxy Crude Oil Demulsification Study

by

Muhammad Arief Fikry bin Zainalabidin

13666

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

May 2014

Universiti Teknologi PETRONAS Bandar Seri Iskandar 31750 Tronoh Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

Waxy Crude Oil Demulsification Study

by

Muhammad Arief Fikry bin Zainalabidin 13666

A project dissertation submitted to the

Mechanical Engineering Programme

Universiti Teknologi PETRONAS

In partial fulfilment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(MECHANICAL)

Approved by,

(Dr Azuraien binti Jaaper @ Jaafar)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2014

CERTIFICATION OF ORIGINALITY

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

MUHAMMAD ARIEF FIKRY BIN ZAINALABIDIN

ABSTRACT

Upstream petroleum production is one of the most critical phase in a petroleum lifecycle activities where the stage require performance consistency to ensure maximum profit can be made upon huge investment that was made during field development stage. As one of the most critical challenges in flow assurance, emulsion formation lead to inconsistency in production performance in means of crude unloading from well to surface, crude transportation through flowlines and crude separation process at the topside facilities. References on previous academic studies and researches towards emulsion treatment or demulsification have proven various methods to encounter the problem which include through heating method and chemical demulsifier injection method. However, these methods are observed to be experimented individually. As provided by one of the academic journal, the best demulsification solution is involving two or more combination of solution available. In this study, combinations of demulsification solution approaches are tested simultaneously by using a specified laboratory device. The combinations cover the approaches of mechanical heating, chemical demulsifier injection and gas aeration. The effects of the combinations are studied and analyzed before the best demulsification solution approaches are identified through optimization analysis. The combination of demulsification approaches, experimental analysis and optimization analysis are performed using Design Expert 6 software while supporting data input obtained through market survey and Aspen Hysys for cost analysis purpose. Seven approaches are presented as results for optimization analysis and the best solution with highest desirability is selected to be the primary operating conditions to encounter emulsion problem for particular crude. Technical insights on in-progress emulsion treatment for two additional crudes are also provided as a way forward to the research.

ACKNOWLEDGEMENT

First and foremost, I would like to express my gratefulness and thankful to The Almighty that with His Blessings I am finally capable to complete my Final Year Project (FYP) which ran for two consecutive semesters with period span of 8-months and for granting me opportunity conducting the project in best conditions that I can ever ask for. I will try my hardest to benefit these privileges and produced desirable results with outstanding performance.

I also would thank Universiti Teknologi PETRONAS (UTP), for providing the course for me, which I believe is essential to provide great foundation of practices for theoretical knowledge that have been harvested after 4-years of studies. This project will give opportunity for me to equip self with technical and soft skills before heading to professional world upon graduation.

Millions of gratitude also goes to my final year project supervisor, Dr.Azuraien bt. Jaafar for facilitating me with useful advices on the project, providing technical guidelines, sharing knowledge and facilities as my project media. Not to be forgotten, I would thank Mr.Petrus Tri Bhaskoro for assisting me performing the project in detail, from the planning step until execution phase.

Not to be forgotten, I would also like to give credits to my mentor, Mr.Khor Siak Foo whom is the team lead flow assurance engineer in Murphy Oil Corporation for his advice and contribution in providing industrial input for my project. Credits of appreciation also goes to Petronas Carigali Sdn. Bhd. (PCSB) for providing crude oil as the main resources and supplies for the projects conducted.

Millions of love and appreciation for my parents, Mr. Zainalabidin bin Jurani and Madam Nor Azizah bt Brahim for their love, care and inspiration. Token of appreciation also goes to my friends, Muhammad Nasrullah bin Annuar, Mohamad Faiz bin Mohd Nor, Azlan bin Azahar, Mohamad Shukor bin Sahroni and Nik Hariz bin Nik Zurin for their continuous supports. Thank you to Nadhira binti Mohd Khairuddin, for her support of love and motivation. Lastly, I would express thousands of gratitude to those who has directly and indirectly contributed to the project success.

TABLE OF CONTENTS

CERTIFICATI	ION OF APPROVAL	i
CERTIFICATI	ION OF ORIGINALITY	ii
ABSTRACT		iii
ACKNOWLED	OGEMENT	iv
TABLE OF CC	DNTENT	V
LIST OF FIGU	URES	vii
LIST OF TABI	LES	ix
ABBREVIATI	ON AND NOMENCLATURES	X
CHAPTER 1:	INTRODUCTION	
	1.1 Background of Studies	1
	1.2 Problem Statement	1
	1.3 Project Objectives	2
	1.4 Scope of Studies	2
CHAPTER 2:	LITERATURE REVIEW	
	2.1 Introduction to Emulsion	4
	2.2 Contributing Factors of Emulsion Formation	4
	2.3 Effects of Emulsion to Production Operations	6
	2.4 Methods to Encounter Emulsion Formation	7
	2.5 Optimization Chemical Demulsifier	8
	2.6 Demulsifier Application to Resolve Emulsion	8

	2.7 Experimental Approach for Demulsification Activities	10
CHAPTER 3:	METHODOLOGY	
	3.1 Project Execution Flow Chart	11
	3.2 Experimental Specifications	15
	3.3 Project Gantt Chart and Key Milestone	17
CHAPTER 4:	RESULTS AND DISCUSSION	
	4.1 Chapter Introduction	18
	4.2 Demulsifcation: Engineering Principle	18
	4.2 Malaysian Sample I – MiriCrude Evaluation	19
	4.3 Malaysian Sample II – Sepat-7 Emulsion Blend	32
	4.4 Malaysian Sample III – TCOT Emulsion Blend	37
	Evaluation	57
	4.5 Miri Crude Demulsification Optimization Design	42
	4.6 Miri Crude Demulsification Operational Feasibility	47

RECOMMENDATION	53
CONCLUSION	54
REFERENCES	55
APPENDIX	57

LIST OF FIGURES

NO	FIGURE TITLE	PAGE
1	Figure 3.1: Project Execution Flow Chart	11
2	Figure 3.2: Demulsification Test Rig Device13	
3	Figure 3.3: Gas Bubble Emulsion Unit (Demulsification Test Rig) 13	
	Process Schematic Diagram	
4	Figure 4.1: Emulsion Phase Separation Quality (%) Profile at	20
	Operating Temperature 35 °C and 200 PPM Demulsifier	
	Concentration	
5	Figure 4.2: Emulsion Phase Separation Quality (%) Profile at	22
	Operating Temperature 35 °C and 400 PPM Demulsifier	
	Concentration	
6	Figure 4.3: Emulsion Phase Separation Quality (%) Profile at	23
	Operating Temperature 35 °C and 600 PPM Demulsifier	
	Concentration	
7	Figure 4.4: Emulsion Phase Separation Quality (%) Profile at	24
	Operating Temperature 57.5 °C and 200 PPM Demulsifier	
	Concentration	
8	Figure 4.5: Emulsion Phase Separation Quality (%) Profile at	26
	Operating Temperature 57.5 °C and 400 PPM Demulsifier	
	Concentration	
9	Figure 4.6: Emulsion Phase Separation Quality (%) Profile at	27
	Operating Temperature 57.5 °C and 600 PPM Demulsifier	
	Concentration	
10	Figure 4.7Emulsion Phase Separation Quality (%) Profile at	29
	Operating Temperature 80 °C and 200 PPM Demulsifier	
	Concentration	
11	Figure 4.8: Emulsion Phase Separation Quality (%) Profile at	30
	Operating Temperature 80 °C and 400 PPM Demulsifier	
	Concentration	
12	Figure 4.9: Emulsion Phase Separation Quality (%) Profile at	31
	Operating Temperature 80 °C and 600 PPM	

	DemulsifierConcentration	
13	Figure 4.10: Sepat-7 Emulsion at 0 th minute after treatment (left)	34
	and demulsification reactor conditions upon treatment of Sepat-7	
	(right)	
14	Figure 4.11: Sepat-7 Emulsion at 50 th day after first treatment	36
15	Figure 4.12: Comparative Chart of Sample Quality at Water Cuts	38
	50:50 and 70:30 at Unit Volume (ml) and Unit Fraction (%)	
16	Figure 4.13: Comparative Chart of Sample Emulsion Quality at	40
	Water Cuts 50:50 and 70:30 at Unit Volume (ml) ad Unit Fraction	
	(ml)	
17	Figure 4.14: Comparative Chart of Sample Synthetic Produced	42
	Water Quality at Water Cuts 50:50 and 70:30 at Unit Volume (ml)	
	and Unit Fraction (%)	
18	Figure 4.15: Total Associated Operating Cost Breakdown	43
19	Figure 4.16: Separator Simulation Layout at Temperature 35 °C in	45
	Aspen HYSYS	
21	Figure 4.17: Figure 4.16: Separator Simulation Layout at	46
	Temperature 57.5 °C in Aspen HYSYS	
22	Figure 4.18: Figure 4.16: Separator Simulation Layout at	46
	Temperature 80 °C in Aspen HYSYS	
23	Figure 4.19: Miri CrudeDemulsificaion Design Summary	47
24	Figure 4.20: Mir CrudeDemulsificationConstraints Setting	48
25	Figure 4.21 :Miri Crue Demulsification Solutions	51
	DesirabilityChart	
26	Figure 4.22 : Miri Crude Demulsification Solutions Desirability	52
	Breakdown Histogram	

NO	TABLE TITLE	PAGE		
1	Table 3.1: Miri Emulsion Blend Activities			
2	Table 3.2: Sepat-7 and TCOT Blend Activities	16		
3	Table 3.3: Project Key Milestone	17		
4	Table 4.1: API 12J Design Criteria of Three Phase Separators	18		
5	Table 4.2: Sepat-7 Run 8 Operating Conditions	32		
6	Table 4.3: Experiment Event Sequence on Sepat-7 Sample	33		
	Demulsification Process			
7	Table 4.4: Operating Temperature Elevation for Sepat-7 Fluid	34		
	Flowing Measurement			
8	Table 4.5: Phase Separation Qualities for Sepat-7 Run 8 at			
	Elevated Heating Temperature 75 °C			
9	Table 4.6: Comparative Table of Sample Oil Quality at Water Cuts			
	50:50 and 70:30			
10	Table 4.7: Comparative Table of Sample Emulsion Quality at	40		
	Water Cuts 50:50 and 70:30			
11	Table 4.8: Comparative Table of Sample Synthetic Produced4			
	Water Quality at Water Cuts 50:50 and 70:30			
12	Table 4.9: Chemical Demulsifier Cost Analysis	44		
13	Table 4.10: Chemical Demulsifier Cost at Separator Size	45		
14	Table 4.11: Heating Power and Corresponding Cost Analysis	46		
15	Table 4.12: Miri CrudeDemulsificationProposed Solutions	50		

LIST OF TABLES

ABBREVIATION AND NOMENCLATURES

Demulsification – Process of resolving emulsion or emulsion breakdown through various methods, including methods of heating temperature, chemical demulsifier and gas aeration.

Demulsifier - A synthetic chemical designated to break emulsion formation through chemical reaction.

Watercuts – *The fraction of water volume over total amount of hydrocarbon produced at the surface. For example; 70:30 watercuts representing 70% oil and 30% water composition in crude.*

Produced Water – Water (or brine) produced alongside with oil at the surface facilities from well and reservoir.

Production Choke Valve -A type of valve used to control the opening of the well which corresponds to flow rate of crude produced from particular well.

Design of Experiment -A computational approach to design or construct planning for a set of experiment comprised of multiple variables.

CHAPTER 1 INTRODUCTION

1.0 INTRODUCTION

1.1 Background of Studies

The Final Year Project (FYP) entitled 'Waxy Crude Oil Demulsification Study' is one of the crucial case study projects in Oil & Gas Industry, commonly associated within the production operations engineering scope during oil and gas production phase at the oilfield. The term of demulsification is rooted from word emulsion which is a mixture of two immiscible liquids. In oil and gas production, demulsification is a process referred to separation of emulsion consisted of oil and water, which commingle together during the multiphase fluid flow from the oil and gas reservoir in the subsurface to the surface production facilities. In order to design effective emulsion treatment, the emulsion behaviour shall be highly considered. The emulsion behaviour is normally depending on the rate of 'exposure' of the liquid to the emulsion formation contributing factors while unloading the hydrocarbon from the well and transporting it to the surface.

1.2 ProblemStatement

During production lifecycle of an oil and gas field, the hydrocarbon which composed of oil, gas and brine (produced water) will be produced together from the well and flowing in commingle way in the pipeline before reaching the surface production facilities. This multiphase commingled production is exposed to shear across the reservoir into the well (in-flow) as well as across the production choke valve (PCV) which will eventually contribute to emulsion formation. The increasing emulsion formation rate will result in increasing viscosity of the mixture which leads to higher pressure loss along the flowlines during production. In typical surface production platform, once arrived at the facilities, the flowing hydrocarbon is subjected to separation process first before being transported for further hydrocarbon process. Multiphase separation of oil, water and gas is usually to be performed in the multiphase separators. Nevertheless, complex emulsion formed in the flowing fluid may result in ineffective separation. The well-mixed viscous emulsion cannot be separated easily using common separation technique like gravity settling method. Thus, following of these issues, proactive measures of engineering practices are to be performed to ensure proper separation of oil and water take place accordingly. This will require emulsion breakdown process or commonly known as demulsification.

1.3 Objectives

The main objective of the project is *to study the performance of combined demulsification methods*. These variables includetemperature, demulsifier injection rate, water-oil ratio (WOR) and effect of gas aeration during separation process. The secondary objective of the project is *to determine optimum demulsification operating conditions by imitating as real as possible the production conditions in the field*. Optimization of the crude oil demulsification process will consider two aspects, as follow:

- Engineering aspect; which measures the effectiveness of demulsification due to few factors including separation settling time, demulsifier dosage and aeration rate.
- Economical aspect; which is considering cost associated with related variables including consumption of demulsifier chemical, heating power and aeration systems.

1.4 Scope of Studies

Generally, the project is part of upstream flow assurance project at UniversitiTeknologi PETRONAS (UTP). Extensive research studies are performed to resolve the emulsion problem during hydrocarbon production. Under this project, scopes of studies to be covered include:

- a. Reproduction of emulsion with synthetic formation water and oil sampled from selected Malaysian oilfields.
- b. Water-in-Oil (W/O) emulsions behaviour at few parameters including specific liquid temperature, defined mixing energy or shear rates and water volumetric fraction in the liquid.
- c. The demulsification performance of the emulsions at (b) at different parameters setting including operating heating temperature, demulsifier chemical dosage and gas aeration rate on the emulsions.

CHAPTER 2 LITERATURE REVIEW

2.0 LITERATURE REVIEW

2.1 Introduction to Emulsion

As according to Udonne (2012), emulsion can be technically defined as dispersion of droplets of a liquid in another liquid which is incompletely immiscible. Oliveira and Goncalves (2005) also added that water-in-oil (w/o) emulsions are normal and commonly occur in petroleum industry; especially in the upstream operations. Emmanuel and Emmanuel (2013) stated that emulsion formed during flow through pumps, chokes and valves and are stable as the crude oil contains natural surfactants.

Kokal and Wingrove (2000) also defined emulsion as an unstable system and then classified emulsions into few types, according to degree of kinetic stability of the mixture. The classes are looses emulsions which will separate in matter of few minutes, medium emulsions which will separate in matter of tens of minutes and tight emulsions which will fully or partially separated in hours, days or weeks (Kokal&Wingrove, 2000). Sefton and Sinton (2010) have explained emulsion classification based on viscosities which are viscosity dependence (non-Newtonian properties) and viscosity independence (Newtonian properties). Water-in-oil emulsion is experiencing viscosity dependence at lower temperature, while sufficiently high temperature promotes the emulsion to be in viscosity independence state and behave as Newtonian fluid (Sefton& Sinton, 2010).

2.2 Contributing Factors of Emulsion Formation

Emulsion formation is a natural occurrence which exists due to several reasons. They are formed in natural way during oil and gas production with water cuts that can reach at most 60% by volume (Oliveira &Goncalves, 2005). A field case study finding conducted by Kokal and Wingrove (2000) in one of the largest oilfield has supported the fact,

which high percentages of water for about 80 - 95% of water cut resulting in very tight and complex emulsions. The viscosity of water-in-oil emulsion is also greatly increased by increasing the water cut and reduction in temperature (Oliveira &Goncalves, 2005). As added in their finding, emulsion viscosity increases almost by linear to water volume fraction values of 20%.

Sefton and Sinton (2010) findings result in similar trend, which the viscosity at lower water cuts gradually increase as water cuts reach 30%, by using various models including Hatschek model, Sibree model and Eiler model. Nevertheless, Kokal and Al-Juraid (1999) stated that emulsions become less tight as water cut become higher, which is easily to be separated. The further increase in water concentration will caused decrease in viscosity due to dilution effect (Abdulkasim Omer, 2009). Another factor that leads to emulsion formation is shear condition. Lab observations conducted by Oliveira and Goncalves (2005) have notified that increment in shear rate has caused the decrement of the size of internal phase droplets which eventually influence the emulsion viscosity.

The graph showed that high shear induces higher apparent viscosity compared to low shear during emulsion formation. The figure of "The effect of shear condition applied during the emulsion's generation on the apparent viscosity of a typical Brazilian heavy crude oil emulsion" is presented as follows.



Note. From "Emulsion Rheology – Theory vs Field Observation" by R.C.G. Oliveira and M.A.L. Goncalves, 2005, 2005 Offshore Technology Conference.

Oliveira and Goncalves' statement has been supported by Kokal and Al-Juraid (1999) through their findings in tests conducted on effect of shear to emulsion, which resulting shear does increasing emulsion stability. From the tests that were conducted, emulsion which was applied with high shear rate unable to complete the separation (only partial separation observed) while emulsion applied with medium and low shear undergone complete separation after 20 minutes and 10 minutes, respectively. Thus it is concluded that increase in shear results in tighter emulsion (Kokal& Al-Juraid, 1999). In addition, Kokal and Al-Juraid (1999) have added asphaltene as contributing factor which cause emulsion problems and also acting as emulsion stabilizers.

2.3 Effects of Emulsion to Production Operations

As part of flow assurance concern, emulsion formation has indeed cause multiple problems to upstream production process. Kokal and Al-Juraid (1999) through their publication 'Quantification of Various Factors Affecting Emulsion Stability: Watercut, Temperature, Shear, Asphaltene Content, Demulsifier Dosage and Mixing Different Crudes' has listed few operational problems which include tripping of equipment in the separation facilities as well as high pressure drops in flowlines. These emulsions cause increment in demulsifier usage, specifications nonconformance crude production and even cause shutdown of the processing equipments at the downstream side. In technical perspective, Oliveira and Goncalves (2005) have highlighted the importance of emulsion rheology on multiphase flow. They also indicated that numerical flow simulation will allow the study of possible flow assurance issue in wells and flowlines. For this, Oliveira and Goncalves (2005) have presented their analyses on pressure drop behavior which related to emulsion water cut. Increasing produced water has led to increase in emulsion viscosity. As consequence, both authors conclude that pressure drop through the production system is increasing as well. Nevertheless, Oliveira and Goncalves (2005) have reminded on theory of single-phase flow which state that for high Reynold's numbers (turbulent pattern), the viscosity will give low effect to pressure drop of most production system.

2.4 Methods to Encounter Emulsion Formation

Emulsion breaking or de-emulsification is the separation of dispersed liquid from the liquid in which it is suspended (Udonne, 2012). Udonne's research also has stated the objective of this demulsification is to eliminate the interfacial film and deliver surfactant to either side of oil and water. In addition, demulsification can be enhanced by decreasing water phase viscosity or increasing oil viscosity. The treatment methods for emulsion in crude oil are distinguished into few applications namely as application of heat, application of electricity, application of chemicals, polymers and natural treatment (Udonne, 2012). The idea was supported by Emmanuel and Emmanuel (2013) through their research 'Application of Physico-Technological Principles in Demulsification of Water-In-Crude Oil System' which destabilization of emulsion can be conducted through four methods namely as mechanical, thermal, chemical and electrical. The application of heat assisted demulsification process by decreasing the viscosity of the oil and thus enhancing gravity settling due to density difference between oil and water. Applications of electricity and chemicaldemulsifier help to promote coalescence of water droplets in emulsion treatment. For polymers and natural treatment, Udonne (2012) state that they are used in surfactants to counteract the effect of asphaltenes in demulsification as well as by means of storage in tanks and pits, respectively. Kokal and Al-Juraid (1999) through their thesis has stressed out that temperature by itself does not resolve emulsions although at extreme temperature, and this high temperature is only effective as demulsifier is added. Thus, demulsifier and heat application combination provides the best demulsification (Kokal& Al-Juraid, 1999). By using Ronningsen model, Sefton and Sinto (2010) also managed to prove decreasing viscosity profile with increasing temperature, at varying water cuts 10 - 40%. On the other hand, Kokal and Wingrove (2005) suggested minimizing tight emulsion formation by reducing shear induced on crude oil by minimizing excessive choking and turbulence occurrence.

2.5 Optimization Chemical Demulsifier

Kokal (2008) through defined demulsifier as chemical designated to neutralize the stabilizing effect of emulsifying agents. Emmanuel and Emmanuel (2013) stress on that chemical demulsification was widely applied to treat emulsion and involves the use of chemical additives to increase the rate of emulsion separation process. Demulsifier added into emulsion will weaken the rigid film of oil and water interface and enhance water droplet coalescence (Kokal, 2008). Kokal (2008) also added that demulsifier is comprised of few components which are solvents, surfaceactive ingredients and flocculants and it have to make close contact, thoroughly mix with the emulsion for the demulsification process takes place effectively. It is important for the petroleum industry to find best and efficient way of testing the chemicals in the laboratories before applying them in the field (Emmanuel & Emmanuel, 2013). Thus, Kokal and Wingrove (2005) recommended bottle test and field test conducts on new demulsifiers for every one to two years to find most cost-effective demulsifier.

2.6 Demulsifier Application to Resolve Emulsion

With high water cut and resultant tight emulsions, installation of demulsifier skid which is to inject demulsifier at rated dosage in offshore facilities is recommended as part of the solution (Kokal&Wingrove, 2000). Dosage is an important factor to be considered as small dosage of demulsifier will cause the emulsion unresolved while too much of demulsifier will cause adverse effect which can lead to produce very stable emulsions (Kokal, 2008). For that, Kokal and Wingrove (2000) have conducted series of oil-water separation tests for demulsifier screening. Numbers of type of demulsifiers at different concentrations 50, 100, 150, 200 and 1000 ppm are used during the tests which are operating at similar temperature as in the field. Kokal and Wingrove (2000) also added that the best demulsifier is then selected for field trials purpose.

Abdulkadir (2010) has conducted series of bottle tests to study the effect of demulsifier in resolving emulsions at few variables including temperature and concentration. Through the bottle test, the smallest amount of chemical (demulsifier) to separate emulsion completely can be determined (Abdulkadir, 2010). It is shown that at higher temperature, the separation percentage is increasing which the occurrence is due to crude viscosity reduction thus induces density difference between oil and water (Abdulkadir, 2010). Abdulkadir (2010) also highlights on effect of retention time which can resulting in separation over certain period but is also possible to cause re-emulsification, in negative way. Thus, he suggests that optimum retention time shall be observed, to allow proper demulsification take place accordingly. Abdulkadir (2010) also stated that performance of demulsifier is affected by API gravity of the crude oil. In treating crude with lower API (heavy oil), the degree of water drop or separation may be lower compared to treating crude with high API. Udonne (2012) stated that demulsifier is not necessarily to be injected into downhole or oil well as emulsion is not formed in the well when the oil is produced. Injecting the chemical in the field provide great advantages as it can reduce the pressure drop in pipelines and promote emulsion separation (Emmanuel & Emmanuel, 2013).

Literature	Method Description	Results
Udonne (2012)	Two types of experiments are conducted which one is performed with emulsion breaker and another one is without the emulsion breaker. Different numbers of drops of emulsion breaker are added to each sample. Samples are spun in a centrifuge machine for separation.	Base Sediment and Water (BS&W) of water fraction increases as drops of emulsion breaker increases. Example: at 500 rpm rotation, the BS&W have difference of 2.5% while the 1000 rpm rotation produces difference of 10%.
Kokal and Wingrove (2000)	Demulsifiers at different concentrations of 50, 100, 150, 200 and 1000 ppm are used during demulsifier screening with similar field operating temperature of 90°F.	The Emulsion Separation Index (ESI) which shown the water separation quality increases with increasing demulsifier concentration. Approximately 18% ESI recorded at highest concentration of 1000 ppm.
Abdulkadir (2010)	Evaluating demulsification at different temperature 40 °C and 60 °C and type of demulsifier. The demulsifier concentration is kept at constant 50 ppm.	Separated water percentage (ESI) increase at higher temperature 60 °C. Four different demulsifier exhibits different ESI.
Emmanuel and Emmanuel (2013)	Blending the crude oil samples with gasoline (diluents) at different ratios and demulsifier. 2 ppm demulsifier added, samples manually shaken, centrifuged for 10 minutes at 3000 rpm. Six samples are set for each.	API gravity increase with increasing diluents percentage. Increasing diluents percentage ratio resulting in higher percentage of volume water separation (with and without demulsifier).

2.7 Experimental Approach for Demulsification Activities

CHAPTER 3 METHODOLOGY

3.0 METHODOLOGY

3.1 Project Execution Flow Chart

The 'Waxy Crude Oil Demulsification Study' is an experimental-based project to study the emulsion behaviour as in the real production field.



Figure 3.1: Project Execution Flow Chart

Flow Chart Breakdown

1. Project Definition

Defining the project based on the problem statement, background of studies, scope of studies and objectives to be achieved at the end of the project.

2. Project Planning & Methodology

Discuss and construct the project planning by designing the gantt chart and anticipated key milestone of the project.

3. Project Input Resourcing (Academic)

Gathering project information and background studies on related topic from various academic sources including thesis and journals.

4. Project Input Resourcing (Industrial)

Gathering input on related topics from industrial personnel which due to industrial experience in performing project of similar field of studies. Input gathered include the process description, demulsification techniques and advice on technical analysis.

5. Design of Experiment (DOEs): Samples of Different Crude

Preparing design of experiment (DOE) by using Design Expert software which include three different demulsification variables to be measured namely as heating temperature application, demulsifier concentration and gas aeration. Three level factorial design model is used for the project, considering presence of three variables. Resulting 32 experiments with various combinations of these three factors are established. See Appendix for details.

6. Preparation for Experiment: Demulsification Test Rig Familiarization

Having familiarization with equipments to be used through hands-on application in the working area.Equipments include demulsification test rig, bottle test equipments and equipments used prior demulsification treatment which is the preparation process. Preparation for experiments comprised of few aspects; which include the preparation of produced water/formation water before mixed up with the crude sample and forming emulsion. The produced water is made up of solution of de-ionized water with few chemical compositions.

The main equipment used for the project experiment is Demulsification Test Rig, which is observed as follows:



Figure 3.2: Demulsification Test Rig Device



Figure 3.3: Gas Bubble Emulsion Unit (Demulsification Test Rig) Process Schematic Diagram

The demulsification test rig is capable to operate demulsification activities under combined variables which include heating, demulsifier injection and gas aeration, simultaneously. During crude demulsification treatment, the gas (air) will be injected through tube from bottom of reactor cylinder while the heating elements surround the cylinder will heat up the crude in the reactor. The chemical demulsifier will be injected by batch, although continuous injection is also applicable.

7. Demulsification Treatment: Variables of Heating Temperature, Demulsifier Concentration and Gas Aeration Rate

Following the completion of the preparation scopes, the demulsification experiments will be conducted by using an in-house demulsification test rig.

8. Bottle Test Monitoring

Upon demulsification, bottle tests will be performed to measure the separation quality of the samples. Observation or measurement on the bottle samples will be conducted at selected time intervals (5th min, 15th min, 30th min, 1st hour, 2nd hour and 4th hour).

9. Demulsification Experiment Analysis

Analyzing experimental results produced based on various demulsification factors combinations in 32 experiments. The results are compared with the findings from the academic literature and journals as well as initial hypothesis made.

10. Cost Analysis and Simulation

Performing cost analysis which covers the operational cost of the demulsification factors combination. The cost inputs include through process simulation with Aspen Hysys and market cost of particular resources.

11. Demulsification Optimization Analysis

Performing demulsification optimization analysis by using Design Expert Analysis and Optimization Tool. Measured data include three measuring factors (heating, demulsifier injection and gas aeration) as well as selected responses including multiphase separation qualities in BS&W percentage and associated operating costs.

12. Project Compilation

Compiling project report and technical report for assessment and publication purpose.

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

3.2 Experimental Specifications

3.2.1 Activity 1: MIRI Emulsion Blend

The experimental specifications for Miri emulsion blend activity are attached as follows.

MIRI EMULSION BLEND – ACTIVITIES		
DemulsificationProcess	Apply following demulsification operating variables:	
	 Three heating temperature ranging 10 °C > WAT until 80 °C. Three demulsifier concentration ranging 200 - 600 ppm. Gas aeration into the liquid 30-100 cc/min 	
	 The experiment session: Demulsification with heating, demulsifier injection and gas aeration. Treatment Period of 30 minutes Monitoring Period of 4 hours 	
Bottle Test	Monitoring emulsion separation at 5th min, 15th min, 30th min, 1st hour, 2nd hour and 4th hour.	

Table 3.1: Miri Emulsion Blend Activities

The experimental procedure is attached in Appendix section. Based on the initial study, Miri crude is originally composed of water and oil with approximated 70:30 composition respectively.

3.2.2 Activity 2: SEPAT-7 & TCOOT Crudes

The experimental specifications for Sepat-7 and TCOT crude activities are detailed as follows. The experiments for the crudes are currently in progress with few findings that will be presented in the result section.

Table 3.2: Sepat-7 and TCOT Emulsion Blend Activities

PROPERTIES		SEPAT-7 CRUDE	TCOT CRUDE
Wax AppearanceTemperature (WAT)		39.4 °C	22 °C
Total Liquid Volume	per Sample	300 mL	
10 : 90		30 mLcrude + 270 mLbrine	
50 : 50		150 mLcrude + 150 mLbrine	
90:10		270 mLcrude + 30 mLbrine	
SEPAT-7 & TCOOT C		RUDES - ACTIVIT	FIES
Emulsification Mixing the pro- and 10 °C above 5 minutes.		oduced water with c ve WAT at 10:90, 5	crude at 8000 rpm 0:50 and 90:10 for
Demulsification	 Apply following demulsification operating variables: 1. Three heating temperature ranging 10 °C > WAT until 80 °C 2. Three demulsifier concentrations ranging 0 - 600 ppm. 3. Gas aeration into the liquid 0-100 cc/min 		

	The experiment session:
	 Demulsification with heating only. Demulsification with heating and demulsifier injection. Treatment Period of 30 minutes Monitoring Period of 4 hours
Bottle Test	Monitoring emulsion separation at 5th min, 15th min, 30th min, 1st hour, 2nd hour and 4th hour.

3.3 Project Gantt Chart and Key Milestone

The completed Gantt Chart of final year project entitled 'Waxy Crude Oil Demulsification Study' is presented in Appendix Section: Appendix A.

Table 3.3: Project Key Milestone

KEY MILESTONE	DESCRIPTION
Extended Proposal	Define project scopes, objectives and
Preparation	methodology. Resourcing input from
	industries and academic publications.
Proposal Defence	Improvement section of the project through
	feedback from students and lecturers.
Miri Emulsion Evaluation	Performing demulsification for Miri
	emulsion blend.
Preliminary Sepat-7	Evaluating the phenomenon occurring to
Emulsion Phenomenon	Sepat-7 crude sample during
Evaluation	demulsification process.
TCOT Emulsion Separation	Evaluating the natural separation of TCOT
Experiment Based on	emulsion based on water cuts to select the
Water Cuts	more stable emulsion blend.
Result Analyses	Perform complete interpretation of project
	findings using various analytical methods
	including cost, simulation and optimization
	analysis.
Final Report Submission	Present written report on final project
	outcomes.

CHAPTER 4 RESULTS AND DISCUSSION

4.0 RESULTS AND DISCUSSION

4.1 Chapter Introduction

This section will present the project findings based on the methodology highlighted in the previous section. The results and discussion will cover following aspects according to the project experiments chronological sequence.

- a) Miri Emulsion Blend Evaluation
- b) Sepat-7 Crude Evaluation
- c) TCOT Crude Evaluation

For the Final Year Project scope, analysis will be highly focusing on the Miri Emulsion Blend Evaluation; however insights of evaluation activities on Sepat-7 and TCOT Crudes will be highlighted as well. At the end of the experiments, demulsification optimization will be performed to analyze the best operating conditions for demulsification to take place accordingly.

4.2 Demulsification: Engineering Principle

As reference to international petroleum standards of API 12J: Specifications for Oil and Gas Separators and API 12L: Specifications for Vertical and Horizontal Emulsion Treaters, the design of the three phase separators shall in compliance with following basic design criteria for liquid retention time.

Minutes (Typical)
3 to 5
5 to 10

Table 4.1: API 12J Design Criteria of Three Phase Separators

80+° F	10 to 20
60+° F	20 to 30

Based on the standards requirements above, the maximum retention time for separation to take place in the designated experiments are 30 minutes. For the demulsification treatment time in the emulsion treater, the specifications in API 12L is referred which allows the residence time in the oil settling zone typically in range of 30 to 100 minutes. As the project aims for the best operating condition of demulsification, thus minimum residence time is selected which is 30 minutes.

The retention time factor is affected by (i) oil settling time to allow adequate water removal from oil and (b) water settling time to allow adequate oil removal from water.Based on the literature review, the factor (a) which is water settling time to allow adequate water removal from oil is taken as the main measurement method. The formula for Base Sediment and Water (BS&W) is presented as follows:

$$BS\&W = \frac{Fraction of a phase presence in crude (ml)}{Total volume of crude (ml)}$$

A phase as defined in formula above can be either oil, water or emulsion. For the experiment, BS&W for emulsion is mainly used. Nevertheless, for additional data which showcase the separated oil and emulsion quality (in percentage BS&W) are provided as well to observe the deviations during the experiments.

4.3 Part A: Malaysian Sample I – MiriCrude Evaluation

Miri crude is one of the three crude samples provided by PetronasCarigaliSdnBhd (PCSB) from one of its field for the purpose of demulsification study in UTP. Miri crude is also the main crude under studies for this final year project. For demulsification, the stability of emulsion in the project is measured by the Base Sediment & Water (BS&W) qualities which are represented in term of percentages. The emulsion stability is measured through the demulsification qualities of emulsion at combined factors or variables. The data will eventually be compared with respect to effect of heating, demulsifier concentration and gas aeration to the demulsification process. Full results can be viewed at Appendix E.

4.3.1 DemulsificationComparative Studies at Heating Temperature 35 °C at Different Variables Combinations

Before the experiments were conducted, few hypothetical statements were constructed and assumed which are presented as follows:

- 1. Increasing demulsifier concentration will lead to higher rate of demulsification between oil and water.
- Increasing gas aeration will induce well-mixed water-oil and demulsifier mixture thus providing higher rate of demulsification.
- Assuming that the mixture is dispersed thoroughly upon mixed up in demulsification test rig, the volumetric percentage (%) is taken as a reliable measuring parameter.





Based on graph above, the expected decreasing trend is observed on the percentage of emulsion produced with respect to time, at 200 PPM demulsifier concentration. The variable parameter in the graph is gas aeration which use air as the type of gas. Thus it can be generally deduced that gas aeration assist the demulsification rate, by mixing up the demulsifier thoroughly in the mixture in the demulsification test rig. As expected, Run 1 with 30 cc/min has the highest emulsion fraction in the five minutes, and the trend is continuously observed until the 4th hour of bottle test observation. This hypothetically indicate that the demulsifier is less mixed up or disperse in the water-oil emulsion. However, the emulsion fraction for all three runs started to chart closely upon 15th minute of the experiments. At 200 PPM, approximately 10% emulsion left at the end of 4th hour of all three experiments.



Figure 4.2: Emulsion Phase Separation Quality (%) Profile at Operating Temperature 35 °C and 400 PPM Demulsifier Concentration

At 400 PPM demulsifier concentration, different trend is collected compared to the trend exhibited in previous graph for 200 PPM. At higher rate of gas aeration, the emulsion fraction is increasing as well at initial stage. The initial hypothesis for this might due to too high concentration of demulsifier or unstabilize emulsion condition. Nevertheless, the trend is slowly approaching expected outcome at the end of the 4th hour bottle test observation. Besides, Run 1 indicates an increment in the emulsion percentage at 4th hour of observation, from 15% to 23%. This may due to re-emulsification of the water-oil emulsion as the resulting of heat loss (decreasing temperature) and decreasing or degradation of effectiveness of the chemical demulsifier. There is also uncertain trend observed at Run 3, where the trend is fluctuating, however by considering the rate of change is less than 5% tolerance, thus the trend change is considerably minor. Minimum of 7% emulsion fraction is observed at the end of the 4th hour of the bottle test observation.





At highest demulsifier concentration applied which is 600 PPM, three normal decreasing emulsion fraction trends are observed in the graph above. Nevertheless the demulsification rate at lower gas aeration rate will result in lower demulsification rate, which is significantly observed at the first five minutes of the bottle test. 56% of emulsion percentage is detected at sample with 30 cc/min gas aeration compared to lower 27% and 36% gas emulsion fraction percentages at 65 and 100 cc/min gas aeration rates, respectively. Nevertheless, the trend for Run 2 and Run 3, almost charted at similar values of emulsion fractions produced at sequencing minutes and hours. Thus the deduction made is that, no significant variance in emulsion fraction is observed between at operating conditions of 65 to 100 cc/min gas aeration rate, thus they are approximated to be the optimum level of gas aeration rate for

the experiments. At 600 PPM, minimum of 10% emulsion fraction is observed at the end of the 4th hour of the experiments.

For the experiments performed at 35 °C, the demulsification qualities are observed to obey the hypothetical statements as presented. At higher gas aeration rate, higher demulsification quality (lower emulsion quality) is produced. Based on general observation, higher demulsifierconcentration tend to contribute to lower emulsion quality. Over observation period, the lowest emulsion produced is lower than 10% which is resulting at operating conditions of 35°C, 400 PPM and 100 cc/min.

4.3.2 Demulsification Comparative Studies at Heating Temperature 57.5 °C at Different Variables Combinations



Figure 4.4: Emulsion Phase Separation Quality (%) Profile at Operating Temperature 57.5 °C and 200 PPM Demulsifier Concentration

Above data and graph is constructed at the conditions of operating temperature 57.5 °C, 200 PPM and at three different gas aeration rates. The results of experiments at 35 °C as presented in the previous report are provided the appendix section. In comparison to the demulsification at 35 °C which is also conducted at 200 PPM demulsifier concentration and three similar gas aeration rates, the demulsification results at temperature of 57.5 °C are exhibiting better separation qualities. 60% of emulsion is observed during demulsification at 57.5 °C compared to the nearly 90% emulsion fraction at 35 °C temperature in 5 minutes bottle test observation. The demulsification occurred to take place at higher rate as the gas aeration rate is increased. The findings combined with the results of demulsification at 35 °C as mentioned above has supported the application of gas aeration in demulsification treatment. The produced air bubbles from the aeration assisted to mix up the demulsifier to be thoroughly dispersed throughout the sample fluids. Based on the concept of Compact Flotation Unit (CFU), the flotation of gas will induce the formation of bubbles which eventually tend to attach to the crude particles. This attachment will cause the decrease of oil droplet specific gravity and will drive the droplets to the surface at faster rate. Thus, this higher difference of density between oil droplets and produced water droplets will cause emulsion breakdown and reforming additional two layers; which are oil and water layers simultaneously.


Figure 4.5: Emulsion Phase Separation Quality (%) Profile at Operating Temperature 57.5 °C and 400 PPM Demulsifier Concentration

Above data and graph is constructed at the conditions of operating temperature 57.5 °C, 400 PPM and at three different gas aeration rates. The findings are also compared with the previous experiments conducts which operates at 35 °C. The finds that effective comparison analysis also more demulsification occurred at higher temperature compared to lower temperature at initial stage. However, it is indicated that at the particular demulsifier concentration, higher rate of demulsification occurred at sample with temperature of 35 °C compared to sample with temperature of 57.5 °C. Similar patterns can be observed at the demulsification treatment at 600 PPM which to be discussed in next part.

At highest gas aeration rate 100 cc/min, the emulsion quality for temperature 35 °C is observed at less than 10% upon 4^{th} hour of observation. This amount of emulsion left is much

lower compared to the amount of the same mixture at temperature 57.5 °C which is approximately 20%. This finding is contradicting with theoretical concept of heating which to lowering the viscosity of the emulsion, thus introducing higher difference of density between water and oil droplets. Nevertheless for this situation, optimization to select the best demulsification operating conditions will be performed later. As for gas aeration factor, increasing rate of gas in-flow increase the separation quality of emulsion as observed in other profiles.



Figure 4.6: Emulsion Phase Separation Quality (%) Profile at Operating Temperature 57.5 °C and 600 PPM Demulsifier Concentration

Above data and graph is constructed at the conditions of operating temperature 57.5 °C, 600 PPM and at three different gas aeration rates. The temperature comparison analysis for 600 PPM demulsifier concentration at 35 °C and 57.5 °C heating

temperature are observed to result similarly behave as in 400 PPM demulsifier condition, in perspective that the emulsion quality is observed to be at lower percentage in overall at lower temperature (35 °C) compared to at high temperature (57.5 °C). However, at the initial stage, higher temperature condition still produces more effective demulsification compared to lower temperature condition. While for the gas aeration variable, high rate of gas in-flow will induce high separation quality of the emulsion as per theory explained previously.

For the experiments performed at 57.5 °C, generally at higher gas aeration rate, lower emulsion quality will be observed at all demulsifier concentration. Nevertheless, demulsifier as concentration increases, the demulsification quality is observed to drop, notable at 400 PPM and 600 PPM. This can be justify with to inappropriate concentration of chemical demulsifier (too high concentration) which lead to re-emulsification of emulsion upon treatment. Thus suggested demulsifier concentration is 200 PPM which produce more stable result. The lowest emulsion quality produced is lower than 10% which operating at 57.5 °C, 200 PPM and 30 cc/min.

4.3.3 Demulsification Comparative Studies at Heating Temperature 80 °C at Different Variables Combinations



Figure 4.7: Emulsion Phase Separation Quality (%) Profile at Operating Temperature 80 °C and 200 PPM Demulsifier Concentration

At the highest temperature measured which is 80°C, inverse reaction is resulted during the experiments. In comparison between all three runs performed, the rate of demulsification is reduced as the gas aeration rate increases. Upon 30-minutes demulsification treatment, all three runs started up at range of 50% - 60% emulsion, which is considerably higher compared to previous experiments performed at 57.5 °C. Upon 30 minutes of bottle observation, the demulsification quality at 30 cc/min is decreasing rapidly compared to at 65 and 100 cc/min and the trend continuously similar until at the 4thhour. Thus, based on above graph, run at 30 cc/min produced better demulsification quality (lower emulsion percentage) compared to results produced at 65 and 100 cc/min. The lowest emulsion quality observed is approximately 20% at 30 cc/min.



Figure 4.8: Emulsion Phase Separation Quality (%) Profile at Operating Temperature 80 °C and 400 PPM Demulsifier Concentration

Above graph of bottle test observation for demulsification at 80 °C and 400 PPM is showing the trend that concludes demulsification qualities reduction at increasing gas aeration rate. Similar trend is observed in previous graph discussed. The emulsion qualities upon demulsification treatment at 65 and 100 cc/min are charted high at start-up of bottle test observation. Nevertheless, the emulsion quality at 30 cc/min gas aeration is distinctly lower than two of its counterparts, which is relevantly lower only at 40% emulsion quality. Thus the run with 30 cc/min gas aeration rate produced better results compared to the other two runs at higher gas aeration rate. The lowest emulsion quality observed is approximately at 25% at 30 cc/min.





Similarly trended with two experiments performed at 80°C, above run also proved that lower gas aeration rate will produce better result on demulsification quality. Emulsion quality at 30 cc/min is significantly lower compared to emulsion quality at 65 and 100 cc/min almost at similar percentage difference along the observation period. Generally, emulsion qualities are observed to chart higher at 600 PPM compared to at 400 PPM. The lowest emulsion quality observed is 30% at 35 cc/min.

These results have deduced that very high temperature would disrupt the qualities of demulsification treatment. The justification behind the phenomenon includes performance degradation of chemical demulsifier due to very high temperature. This supporting evidence is deduced as the ideal demulsification will take place by heating and demulsifer application, based on previous academic studies. Furthermore, for this particular temperature only, inverse results are observed for all three experiments which lower gas aeration rates will produce higher demulsification qualities. This finding is theoretically oppose the previous findings which the gas aeration rate will increase the density difference between oil and water thus assisting the separation process in the emulsion. Thus it is observed that the temperature is the dominant factor in the experiments, and will affect other variables performance. The best result observed is charted at run performed at 80 °C, 400 PPM and 35 cc/min with relevantly lowest emulsion quality produced.

4.4 Part B: Malaysian Sample II – Sepat-7 Crude Evaluation

For Sepat-7 crude, initial experiment for demulsification using the demulsification test rig has been performed with the similar format as Miri emulsion blend experiments. However, an unexpected phenomenon has occurred during the demulsification process which will be elaborated in details as follows.

A 300 ml waxy crude oil-produced water emulsion of Sepat-7 samplewith 90:10 water cut has been taken into demulsification process by which the operating conditions are set up as follow:

Sepat-7 Run 8 Operating Conditions				
Operating Variable	Variable Setup			
Heating Operating Temperature	50 °C (10 °C above WAT)			
Demulsifier Concentration	Not available			
Gas Aeration	Not available			
Reactor Treatment Period	30 minutes			

Table 4.2: Sepat-7 Run 8 Operating Conditions

In the first experiment conducted on Sepat-7, only heating application was present from three measuring variables. The water-oil emulsion of Sepat-7 was heated up in the reactor for 30 minutes as for treatment process. The result of demulsification is presented in the following table:

Sequence of Event	Details/Observation/Results	Time/Peri od
Experiment Run 8 commencement	300 ml emulsion sample is placed into the reactor of the demulsification test rig on 15 th May 2014.	3.00 pm
Completing sample demulsification treatment	The sample extraction process is conducted however no flowing liquid is observed at the outlet upon opening the valve.	3.30 pm
Consultation on next step required to be taken	The encountered problem led to immediate consultation with superior in-charge. Upon discussion, agreement is made to increase the operating temperature of the sample until the temperature where the sample started to flow (Wax Disappearance Temperature/WDT).	4.00 pm
Increasing operating temperature by periodic interval of 3°C in every 10 minutes.	The rig operator increased the temperature of the test rig and checked of any flowing fluid from the outlet with valve open. If no flow presence, the temperature will be elevated at respective interval. <i>(Temperature elevation can be observed in following table)</i>	5.00 pm – 5.50 pm
Reaching rig operating temperature of 75 °C	The liquid sample in the rig reactor started to flow through outlet valve at 75°C.	5.50 pm
Collecting sample for further studies	42.5 ml Sepat-7 crude is taken from total 300 ml sample volume and was kept in the centrifuge tube. Bottle test observation is conducted to measure	6.10 pm

Table 4.3: Experiment Event Sequence on Sepat-7 SampleDemulsification Process

the separation quality of the sample.



Figure 4.10: Sepat -7 Emulsion at 0th minute after treatment (left) and demulsification reactor conditions upon treatment of Sepat-7 (right)

Images above exhibited a sample of emulsion blend of Run 8 at 0th minute upon demulsification treatment with demulsification test rig. 42.5 ml sample is extracted for bottle test studies which the results are presented in following analysis. The left image is showing the condition of rig reactor upon demulsification treatment. Deformation of rubber connector to bottom outlet valve is observed. During the treatment, the surface of the sample which is positioned at the third level outlet valve is observed to be in liquid form, while the bottom part is suspected to be gelled up due to no-flow condition occurred during sample extraction and collection.

 Table 4.4: Operating Temperature Elevation for Sepat-7 Fluid Flowing

 Measurement

Actual Temperature Supplied	Rated Temperature Supplied	Remarks
63°C	73°C	Fluid not flowing
66°C	76°C	Fluid not flowing

69°C	79°C	Fluid not flowing
72°C	82°C	Fluid not flowing
75°C	85°C	Fluid is flowing

From the sequence of event occurred during the experiment, following deductions are made and to be further justified:

- The sample of emulsion is gelled up in the demulsification test rig during/upon treatment. The variable of the demulsification only include heating application only, without gas aeration and demulsifier injection added.
- The oil at the top of the liquid accumulated in the reactor is in liquid form and did not gelled up, thus only the fluid in the middle and bottom part of the reactor is gelled up.
- The sample of emulsion is flowing down from the reactor at relatively high temperature which is approximately 75°C. This is in contrast with the initial wax appearance temperature (WAT) of the Sepat-7 crude which is tested at 39.4 °C. Thus an insight of increasing WAT of the new emulsion formation from 39.4°C to 75°C is suggested due to the phenomenon occurred.
- The sample taken has been taken into bottle test for phase separation quality measurement within an hour observation period. The results are observed as follow:

Table 4.5: Phase Separation Qualities for Sepat-7 Run 8 at ElevatedHeating Temperature 75 °C

Time Interval of Observation	Oil Phase Quality (ml/%)	Emulsion Phase Quality (ml/%)	Produced Water Phase Quality (ml/%)	Phase Type
0 th minute	0/0	42.5/100	0/0	Liquid
5 th minute	17.5/41	25/59	0/0	Liquid
15 th minute	17.5/41	25/59	0/0	Liquid

30 th minute	17.5/41	25/59	0/0	Liquid
1 st hour	17.5/41	25/59	0/0	Liquid

From the result above, it is indicated that there is no significant change of phase qualities within an hour period of observation, except during the first 5 minutes of observation. Approximately 50 days upon the experiment is conducted, the sample is reviewed again for separation quality measurement. The measurement observed is as follows:



Based on the image shown, about 19.5 ml (or 46% fraction) of oil layer is formed, and 23 ml (or 54% fraction) is still in milky brownish emulsion form with no clear produced water observed. This has proven that the heating application only is not capable to resolve the emulsion for very long time, especially due to waxy properties of the crude oil. The crude oil will be gelled up at room temperature which will stop the separation process due to gravity settling. Thus continuous heating should be applied to determine the maximum separation level at that particular temperature.

Figure 4.11: Sepat -7 Emulsion at 50th day after first treatment

4.5 Part C: Malaysian Sample III – TCOT Crude Evaluation

For the TCOT crude, evaluations based on water cuts have been performed to select the more stable emulsion between two highly steady emulsions at different water cuts. The evaluation process is required to be conducted due to amount of crude supply constraint for the experiment to be carried out. Note that no supporting demulsification methodologies are added into the sample; either bydemulsifier injection, heating application nor gas aeration supply. Thus, the separation is only affected by the natural cause without any external factors. The results obtained are presented as follow:

Table below is showing the natural separation and demulsification evaluation for TCOT crude at two different water cuts which are 50:50 (representing 50% crude oil and 50% synthetic-produced water) and 70:30 (representing 70% crude oil and 30% synthetic-produced water). The sample observations are performed in selected time interval as noted below, with the separation quality of three-phase substances present which are oil, emulsion and water respectively.

4.5.1 TCOT Natural Demulsification Analysis at Different Water Cuts: Oil Separation Quality

The following analysis is showing the synthetic produced water separation quality during the TCOT natural water in oil demulsification using the bottle test. The observation as tabulated in the data is presented in term of unit millilitre and BS&W percentage for water cuts of 50:50 (50% oil and 50% water) and 70:30 (70% oil and 30% water) in 4 hours observation period.

Table 4.6: Comparative Table of Sample Oil Quality at Water Cuts50:50 and 70:30

Time Interval	Observation 50:50		Observation 70:30	
Time interval	Unit (ml)	Unit (%)	Unit (ml)	Unit (%)

0th min	0	0	0	0
5th min	2	13.3	0	0
15th min	2	13.3	0.5	3.3
30th min	2	13.3	0.5	3.3
1st hour	2	13.3	1	6.6
2nd hour	2	13.3	2	13.3
3rd hour	2.5	16.6	3	20
4th hour	2.5	16.6	3	20



Figure 4.12: Comparative Chart of Sample Oil Quality at Water Cuts 50:50 and 70:30 at Unit Volume (ml) and Unit Fraction (%)

Data above are showing the profile of pure crude oil quality against time period during demulsification process which is achieved during natural separation of the emulsion sample. Theoretically, the demulsification of crude oil emulsion will lead to increasing volume of crude oil as the time factor will cause the accumulation and coalescence of the oil particles or droplets in the mixture. The theoretical trend can be observed as in the graph, however the profile is not linearly produced. Thus, it is deduced that the crude of reformation rate is a nonconstant variable and is varying at water cut to another water cut.

Based on the graph, both samples are indicating 100% emulsion formation throughout the centrifuge tube. However, the initial phase separation performance of sample with 50:50 water cut fraction is exhibiting rapid oil phase separation on which approximately 13% of emulsion is completely turn into free oil phase just after 5 minutes of crude oil-produced water mixing period. This amount of free oil phase is observed to be constant until the 2^{nd} hour of the experiment.

On the other hand, TCOT oil phase quality for sample with 70:30 oil-produced water concentrations is showing slower demulsification rate compared to sample of 50:50 water cut ratio. 100% emulsion formation is observed until at the 5th minute after the crude oil-produced water mixing period. Nevertheless, the almost linear demulsification rate profile is observed at the middle of the observation. The TCOT oil quality in 70:30 sample is observed to exceed the 50:50 water cut ratio sample after the 2^{nd} hour of the observation.

4.5.2 TCOT Natural Demulsification Analysis at Different Water Cuts: Emulsion Separation Quality

The following analysis is showing the synthetic produced water separation quality during the TCOT natural water in oil demulsification using the bottle test. The observation as tabulated in the data is presented in term of unit millilitre and BS&W percentage for water cuts of 50:50 (50% oil and 50% water) and 70:30 (70% oil and 30% water) in 4 hours observation period.

Time Interval	Observatio	Observation 50:50		ion 70:30
Time interval	Unit (ml)	Unit (%)	Unit (ml)	Unit (%)
0th min	15	100	15	100
5th min	13	86.6	15	100
15th min	13	86.6	14.5	96.6
30th min	13	86.6	12.5	83.3
1st hour	13	86.6	11	73.3
2nd hour	4.5	30	9	60
3rd hour	3.5	23.3	7	46.6
4th hour	2.5	16.6	7	46.6

Table 4.7: Comparative Table of Sample Emulsion Quality at WaterCuts 50:50 and 70:30



Figure 4.13: Comparative Chart of Sample Emulsion Quality at Water Cuts 50:50 and 70:30 at Unit Volume (ml) and Unit Fraction (%)

The emulsion trending for the phase quality against time is observed to be decreasing within the period of 4 hours. The profile is expected during demulsification due to the separation between oil and water droplets in the emulsion over period of time. Nevertheless, the demulsification rate of the emulsion layer is observed to be non-linear, and differ from one water cut to another.The emulsion layers of the TCOT samples are observed from 0th minute to 4th hour of observation period. Both samples contained 100% emulsion at the 0th minute, which is immediately after the mixing of crude oil and the synthetic produced water.In the first 15minutes, the demulsification rate is higher in the sample with50:50 water cut ratio, compared to another sample with 70:30 water cut ratio. However, higher slope of demulsification rate is observed in 70:30 sample upon 30th minute until the first hour.After 4th hour, approximately only 20% emulsion content is observed in 50:50 sample compared to the 45% emulsion content in another sample.

4.5.3 TCOT Natural Demulsification Analysis at Different Water Cuts: Synthetic Produced Water Separation Quality

The following analysis is showing the synthetic produced water separation quality during the TCOT natural water in oil demulsification using the bottle test. The observation as tabulated in the data is presented in term of unit millilitre and BS&W percentage for water cuts of 50:50 (50% oil and 50% water) and 70:30 (70% oil and 30% water) in 4 hours observation period.

Time Interval	Observation 50:50		Observation 70:30	
	Unit (ml)	Unit (%)	Unit (ml)	Unit (%)
0th min	0	0	0	0
5th min	0	0	0	0
15th min	0	0	0	0
30th min	0	0	2	13.3
1st hour	0	0	3	20

Table 4.8: Comparative Table of Sample Synthetic Produced WaterQuality at Water Cuts 50:50 and 70:30

2nd hour	8.5	56.6	4	26.6
3rd hour	9	60	5	33.3
4th hour	10	66.6	5	33.3

As observed, the volumes of separated synthetic produced water quality at both water cuts are increasing along the time period. Based on the chart, there is no water separated in the first 30 minutes due to although the volume qualities of oil and emulsion in similar period are differentiating.



Figure 4.14: Comparative Chart of Sample Synthetic Produced Water Quality at Water Cuts 50:50 and 70:30 at Unit Volume (ml) and Unit Fraction (%)

4.6 Miri Crude Demulsification Optimization Design

The demulsification optimization is a method used to determine the best demulsification operating conditions which can produce the highest separation qualities of oil and water. The demulsification optimization can be performed by using Design Expert 6 software, which is the similar software used to generate the experimental design for Miri crude. For this project, the demulsification optimization method will only be applied for the Miri crude, which all experiments results have been obtained successfully.

In similar with the experiment design through Design Expert 6, the demulsification optimization also work on three demulsification factors which are heating temperature, demulsifier injection concentration and gas aeration. In addition to the demulsification optimization, five types of responses will be measured to determine the most effective demulsification operating conditions. The measured responses are:

- i. Oil Fraction Separation Quality (BS&W %) at 15th minute.
- ii. Oil Fraction Separation Quality (BS&W %) at 30th minute.
- iii. Rag Layer Quality (BS&W %) at 15th minute.
- iv. Rag Layer Quality (BS&W %) at 30th minute.
- v. Total Associated Operating Cost (RM)

The tabulated data for all five responses input are observed in the Appendix 1. The values obtained for the top four responses are gathered based on the experimental results which are the BS&W qualities upon bottle test. However, the associated operating cost will need further breakdown analyses which are as follows:



Figure 4.15: Total Associated Operating Cost Breakdown

There are three possible sources of operating costs possible in determining demulsification optimization in the project. However, only chemical

demulsifier cost and heating cost are feasible for calculation due to lack of available data required to calculate the gas aeration cost.

The chemical demulsifier cost analysis is mainly represented by unit cost per litre of crude oil. Thus, the demulsifier cost is closely affected by the concentration factor of the demulsifier. Based on the demulsifier concentration assigned, the corresponding volume ratio of chemical demulsifier to crude oil can be determined.

Demulsifier	Unit (ml/litre	Unit (ml/0.3	Cost/tonne	Cost/0.3 litre
Concentration	of sample)	litre of crude)		sample
200 PPM	0.2	0.06	RM 6400	RM 0.40
400 PPM	0.4	0.12	RM 6400	RM 0.80
600 PPM	0.6	0.18	RM 6400	RM 1.20

Table 4.9: Chemical Demulsifier Cost Analysis

The final input obtained from table above for demulsifier cost analysis is the cost of demulsifier for every 0.3 litre sample volume, which is the sample volume for each experiment sample in the project. The price of demulsifierHowever, as the heating cost analysis is performed by simulated separator-sized sample volume using Aspen Hysys, thus the demulsifier cost analysis is also rated at separator-sized volume to uniform the calculation. In the simulation conducted by Aspen Hysys, the separator volume is assumed to sustain approximately 7670 barrels/day of crude oil. As conversion factor of 1 fluid barrel to litre:

1 fluid barrel = 119.24 litre

Thus 7670 barrel/day is equivalent to 1.22×10^6 *litre per day*. By considering that the retention time of liquid in the separator is maximum 30 minutes, thus the volume of crude to be contained in the separator at

one particular period is approximately 25417 litre/30 minutes. Thus, upon detailed calculation, the demulsifier cost for each demulsifier concentration is tabulated as follows:

Demulsifier ConcentrationOperating Cost at Separator Size200 PPMRM 3050.50400 PPMRM 6100.80600 PPMRM 9151.20

 Table 4.10: Chemical Demulsifier Cost at Separator Size

For heating, simulations with Aspen Hysys have been performed in a horizontal separator model with original composition of Miri crude which is 70% of oil and 30% of water. The simulation layout is presented below:



Figure 4.16: Separator Simulation Layout at Temperature 35°C in Aspen Hysys



Figure 4.17: Separator Simulation Layout at Temperature 57.5°C in Aspen Hysys



Figure 4.18: Separator Simulation Layout at Temperature 80°C in Aspen Hysys

The heating power requirement and corresponding heating cost for crude heating process at pre-determined temperature are tabulated in following table:

Temperature	Heating Power Required	Heating Power	Heating Cost
(°C)	(kW)	Rating (kWh)	(RM)
35	233	116.5	8
57.5	900	450	31
80	1585	792.5	54

Table 4.11: Heating Power and Corresponding Cost Analysis

The corresponding heating power required is cross-matched with the fuel gas price in the market. In offshore facilities operations, fuel gas is commonly used as the main source of energy to operate the electric generator thus the cost of the fuel is mainly considered for the separator heating cost calculation. On average, the price of fuel gas is taken at approximately RM 20 per Million British Thermal Unit (MMBTU). Note that 1 MMBTU is equivalent to 293 kWh. Thus based on this conversion, the heating costs are determined and tabulated in the previous table.

4.7 Miri Crude Demulsification Operational Feasibility

By using all the data available in Miri Crude Demulsification Optimization Design which include demulsification factors or variables and measuring responses, the best solution for demulsification operating condition is to be proposed.

Study Type	Response Su	rface	Experiments	32			
Initial Design	3 Level Facto	rial	Blocks	No Blocks			
Design Mode	Quadratic						
Response	Name	Units	Obs	Minimum	Maximum	Trans	Model
Y 1	Oil Fraction S	€%	32	13.00	67.33	None	2FI
Y2	Oil Fraction S	€%	32	13.00	67.67	None	2FI
Y3	Rag Layer (1	5%	32	16.67	87.00	None	2FI
Y4	Rag Layer (30	0%	32	14.33	87.00	None	Linear
Y 5	Total Cost	RM	32	3058.00	9205.20	None	2FI
Factor	Name	Units	Туре	Low Actual	High Actual	Low Coded	High Coded
A	Heating Appli	c Celcius	Numeric	35.00	80.00	-1.000	1.000
В	Demulsifier In	j ppm	Numeric	200.00	600.00	-1.000	1.000
С	Gas Aeration	cc/min	Numeric	30.00	100.00	-1.000	1.000

Figure 4.19: Miri Crude Demulsification Design Summary

The design summary is presented above which indicate the study type, initial design and design model. All the data setup above is selected by default as in Design Expert software. All factors or variables and responses are considering 32 experiments performed for Miri crude demulsification treatment.

Based on the design above from 32 experiments, oil fraction separation quality measured at 15th minute ranging from 13% to 67.33%. Expected increment is observed as the maximum value increases to 67.67% at 30th minute. For the rag layer minimum rag layer observed is 316.67% at 15th minute of observation. The value is expected to decrease over period of time thus at 30th minute, 14.33% of rag/emulsion layer is observed. Minimum associated cost calculated is rated at RM3058 and the most expensive cost is calculated at RM9205.20, based on previous calculations shown.

To determine the best solutions based on design summary, design constraints shall be establish to lower the scope and set up the objective or desirability based on results obtained. The design constraints are presented in figure below. The goals of constraints for factors are all data must be in range of lower and upper limits which represent minimum and maximum values respectively. For oil fraction separation qualities at both 15th and 30th minutes, the maximum values are anticipated as the maximum separation of oil from emulsion is targeted to achieve efficient crude production. On the other side, minimum rag layer is targeted at 15th and 30th minutes as to reduce the emulsion as much as possible during separation process. Finally, the least expensive associated cost is to be achieved to reduce the operational cost for the demulsification treatment in real field operations.

		Lower	Upper	Lower	Upper	
Name	Goal	Limit	Limit	Weight	Weight	Importance
Heating Applic	is in range	35	80	1	1	3
Demulsifier Inj	is in range	200	600	1	1	3
Gas Aeration	is in range	30	100	1	1	3
Oil Fraction Se	maximize	13	67.33	1	1	3
Oil Fraction Se	maximize	13	67.67	1	1	3
Rag Layer (15	minimize	16.67	87	1	1	3
Rag Layer (30	minimize	14.33	87	1	1	3
Total Cost	minimize	3058	9205.2	1	1	3

For the experimental purpose, the importance rating for all factors and responses are set up at 3 which is the intermediate importance rating. Minimum importance rating is 1 while the maximum importance rating is 5. All data is rated similarly to balance the need of each measuring parameter. After selecting the constraints, computational analysis by Design Expert software has proposed seven (7) different solutions or approaches to resolve the Miri crude emulsion. Each solution or approach proposed is providing different set of values for factors and their respective responses.

The seven approaches proposed by Design Expert will be ranked from topmost to bottom based on the desirability which is measure of efficiency to be based on combination of goals set up in constraints earlier. The highest efficiency is 1.0. The higher the value is indicating more efficient proposal. Table below is showing the seven approaches determined based on computational analysis by Design Expert. The details of the approaches include the values for operating factors, expected results of selected responses and the desirability.

	Heating	Demulsifier	Gas	Oil Fraction	Oil Fraction	Rag Layer	Rag Layer	Associated	
No	Application	Concentration	Aeration	at 15 th min	at 30 th min	at 15 th min	at 30 th min	Operating Cost	Desirability
	(°C)	(ppm)	(cc/min)	(%)	(%)	(%)	(%)	(RM)	
1	35.00	200.01	100.00	57.4685	63.0259	23.4518	18.434	3068.94	0.914
2	35.00	202.24	99.92	57.485	63.0153	23.4405	18.4664	3102.67	0.913
3	35.00	200.00	99.06	57.3113	62.881	23.6675	18.3335	3087.09	0.912
4	35.00	251.36	100.00	58.156	63.068	22.7701	19.3726	3808.67	0.893
5	35.00	265.95	100.00	58.3513	63.0798	22.5765	19.6394	4018.89	0.887
6	35.02	200.01	81.41	54.3608	60.1583	27.7143	16.4694	3428.75	0.872
7	46.23	200.00	100.00	51.1687	56.0364	31.9048	27.523	2995.71	0.813

Table 4.12: Miri Crude Demulsification Proposed Solutions

Approach 1 which has the highest desirability of 0.914 is to be selected as the primary approach to resolve the Miri emulsion. By operating the separator or emulsion treater at temperature of 35 °C, demulsifier injection at 200 PPM concentration and gas aeration injection at rate of 100 cc/min, approximately 63% oil fraction can be recovered during separation in 30 minutes. On the other hand, approximately the emulsion can be reduced down to 18% within similar period. This approach can be achieved with minimal cost of RM 3068.94 which is relevantly low in the cost range identified earlier.



Figure 4.21: Miri Crude Demulsification Solutions Desirability Chart

Based on the desirability chart above, the proposed demulsification solution has the highest desirability which to operate at 200 PPM chemical demulsifier concentration, 100 cc/min gas aeration and 35°C heating temperature. As the demulsifier injection and temperature increases, the desirability value decreases as the corresponding responses are deviated further from targeted goals.



Desirability

Figure 4.22: Miri Crude Demulsification Solutions Desirability Breakdown Histogram

The recoverable oil from the emulsion within 30 minutes of bottle test observation can be calculated as:

Separation Efficiency =
$$\frac{Separated \ Oil \ at \ 30th \ minute \ (\%)}{Original \ oil \ composition \ of \ crude \ (\%)}$$

As stated earlier, the original composition of the Miri crude is approximated at 70% of oil and 30% of water. Provided that proposal 1 is expected to recover 63% of oil, thus it is relatively considered as high achievement for recoverable amount from emulsion. The efficiency is then calculated as:

Separation Efficiency =
$$\frac{63\%}{70\%} = 0.90$$

Thus, the overall demulsification process efficiency is rated at 90%, provided that Proposal 1 is selected as the primary operating conditions to encounter emulsion issue.

5.0 RECOMMENDATION

Few recommendations that can be made for improvement are listed as follows:

- 1. Performing few complementary tests to validate the process and data gathering. Tests such as density test and conductivity test will verify the demulsification quality of the crude rheologically which is more detailed and accurate.
- 2. Improvement of the demulsification test rig device. The demulsification test rig device is still under testing process and thus further evaluation on the equipment have to be conducted with series of pilot test experiments.
- In-depth study to measure cost required for application of gas aeration for emulsion separation process. For example, field study on Compact Floatation Unit (CFU) which utilizes gas bubble injection principle can be a benchmark for cost analysis study on gas aeration practicability.

6.0 CONCLUSION

In conclusion, the findings of the experiments have successfully provided insights on the behaviour of the waxy crude oil towards the demulsification based on three different measuring parameters which are heating temperature, demulsifier concentration effect as well as gas aeration effect. In overall, the increment in temperature from low to medium temperature has caused increasing in separation quality of the emulsion at 200 PPM demulsifier concentration, nevertheless adverse effect which decreasing emulsion separation quality are observed at 400 PPM and 600 PPM demulsifier concentration. This has supported previous researches which claim no exact demulsifier concentration formula is universal for all petroleum fields. At 80 °C, the findings deduced that very high temperature would disrupt the qualities of demulsification treatment. The justification behind the phenomenon includes performance degradation of chemical demulsifier due to very high temperature. This supporting evidence is deduced as the ideal demulsification will take place by heating and demulsifer application, based on previous academic studies. Thus it is observed that the temperature is the dominant factor in the experiments, and will affect other variables performance. The demulsification optimization analysis provides the method of selection for the best demulsification approach based on the measured variables and responses. Pre-defined goals and constraints contribute to assist the user in selecting solution with cost effective and operationally feasible criteria. Based on Miri crude demulsification optimization analysis, the selected approach as the best solution to encounter emulsion issue is operating at 35 °C heating temperature, 200 PPM demulsifier concentration by batch injection and 100 cc/min gas aeration. As the objective of the project is to study the separation behaviour of waxy crude oil under different demulsification variables and to establish optimum operating condition to resolve emulsion for crude-in-study, thus the objectives are achieved.

REFERENCES

- Abdulkadir, M. (2010). Comparative Analysis of The Effect of Demulsifiers in The Treatment of Crude Oil Emulsion. *ARPN Journal of Engineering and Applied Sciences*, 5(6), 67-73. Retrieved from http://www.arpnjournals.com/jeas/research_papers/rp_2010/jeas_0610_350.pdf
- Abulkasim Omer, A. O. (2009). Pipeline Flow Behavior in Water-in-Oil Emulsions. (Doctoral Dissertation, University of Waterloo, 2009). Retrieved from https://uwspace.uwaterloo.ca/bitstream/handle/10012/4890/Thesis%203.pdf?sequ ence=1
- Emmanuel, J.A. & Emmanuel, J.E. (2013, January). Application of Physico-Technological Principles in Demulsification of Water-in-Crude Oil System. *Indian Journal of Science and Technology*, 6(1), 60-64. Retrieved from http://www.indjst.org/index.php/indjst/article/viewFile/30561/26480
- Kokal, S. (2008). Chapter 12 Crude Oil Emulsions. In Fanchi, J.R. & Lake, L.W. (Ed.), *Petroleum Engineering Handbook Volume I: General Engineering*. Richardson, TX, USA.
- Kokal, S. & Al-Juraid, J. (1999, October 3-6). *Quantification of Various Factors Affecting Emulsion Stability: Watercut, Temperature, Shear, Asphaltene Content, Demulsifier Dosage and Mixing Different Crudes.* Paper presented at the 1999 SPE Annual Technical Conference and Exhibition, Houston, Texas. Society of Petroleum Engineers, Inc.
- Kokal, S. &Wingrove, M. (2000, October). *Emulsion Separation Index: From Laboratory to Field Case Studies*. Paper presented at the 2000 SPE Annual Technical Conference and Exhibition in Dallas, Texas. Society of Petroleum Engineers, Inc.

- Oliveira, R.C.G. &Goncalves, M.A.L. (2005, May 2-5). *Emulsion Rheology Thesis vs Field Observation*. Paper presented at the 2005 Offshore Technology Conference, Houston, T.X., U.S.A. Offshore Technology Conference.
- Sefton, E. & Sinton, D. (2010). Evaluation of Selected Viscosity Prediction Models for Water in Bitumen Emulsions. *Journal of Petroleum Science and Engineering*, 72, 128-133. Retrieved from http://www.sciencedirect.com/science/article/pii/S0920410510000550
- Udonne, J.D. (2012, December). Chemical Treatment of Emulsion Problem in Crude Oil Production. *Journal of Petroleum and Gas Engineering*, 3(7), 135-141. Doi: 10.5897/JPGE11.065.

<u>APPENDIX</u>

NO	APPENDIX
1	Appendix A: Gantt Chart Final Year Project I (FYP I)
2	Appendix B: Gantt Chart Final Year Project II (FYP II)
3	Appendix C: Miri Crude Demulsification Experimental Procedure
4	Appendix D: Design of Experiment (DOE) Miri Crude Demulsification
5	Appendix E: Miri Crude Demulsification Results

Appendix A: Gantt Chart Final Year Project I (FYP I)

			SEMESTER 1 (FYP I)														
NO	SUBJECT	ALLOCATION	N 1 2 3 4 5 6 7 8 9 10 11 12 13 14												14	15	
1	FYP Topic Selection	N/A															
2	Project Introduction	23/1/2014															
3	Extended Proposal Preparation	29/1/2014															
	Project Methodology Planning	3 Weeks															
	Project Gantt Chart & Milestone Preparation	3 Weeks															
	Industrial Information Sourcing	1 Weeks															
	Literature Reviews	3 Weeks															
4	Consumables Purchasing	3 Weeks															
5	Submission of Extended Proposal	23/2/2014															
6	Proposal Defense Preparation	2 Weeks															
7	Preparation of Experiments	3 Weeks															
	Produced-Water Preparation	1 Week															
8	Submission of Proposal Defense	3/3/2014 - 16/3/2014															
9	Miri CrudeDemulsification Evaluation &Experiments	7 Weeks															
	Demulsification Test Rig (DTR) Familiarization	1 Week															
	Demulsification Test (Blend) - DTR Heating &Demulsifier Injection (Using DOE software to combine all three processes)	3 Weeks															

	Demulsification Test (Blend) - Bottle Test Monitoring	4 Hours / Sample						
10	Preliminary Data Analysis for Familiarization Experiments	1 Week						
11	Sepat Crude Demulsification Evaluation	2 Weeks						
	Demulsification Test 1 - DTR Heating (Heating Temperature range 10°C above WAT to 80°C)	2 Weeks						
	Demulsification Test (Blend) - Bottle Test Monitoring	4 Hours / Sample						
12	Submission of Interim Report	20/4/2014						

Appendix B: Gantt Chart Final Year Project II (FYP II)

			SEMESTER 2 (FYP II)														
NO	SUBJECT	ALLOCATION	N 1 2 3 4 5 6 7 8 9 10 11 12 13 14 1													15	
1	Preliminary Data Analysis for Phase I Experiments	2 Weeks															
2	TCOT Crude Demulsification Evaluation	3 Weeks															
	Demulsification Comparison between Water Cuts 70:30 and 50:50 Oil-Water Ratio	2 Weeks															
	Demulsification Test (Blend) - Bottle Test Monitoring	4 Hours / Sample															
3	Preliminary Data Analysis for Phase II Experiments	2 Weeks															
4	Preparation of Progress Report	2 Weeks															
5	Submission of Progress Report	1 Week															
6	Project Findings Interpretation & Analyses	6 Weeks															
	Comparative Analyses on the Settling Period for Complete Emulsion Separations	1 Week															
	Comparative Analyses on the Separated Water/Oil Volume	1 Week															
	Establishment of Recommended Operating Conditions for Effective Stable Emulsion Separation	1 Week															
	Cost Engineering Analyses	1 Week															
	Compilation of Project Findings Interpretation & Analyses	1 Week															
7	PRE-SEDEX	1 Week															
8	Preparation of Draft Report & Technical Paper	4 Weeks															
9	Submission of Draft Report	1 Week															
10	Submission of Technical Paper	1 Week															

11	Submission of Dissertation (Soft Bound)	1 Week							
12	Oral Presentation / Viva	1 Week							
13	Submission of Project Dissertation (Hard Bound)	1 Week							
GANTT CHART COLOUR LEGENDS									
--									
Period Span for Sub-Activities									
Period Span for Main Activities (A Set of Experiments or Analyses Period									
Period Span for Project Milestone									

Appendix C: Miri Crude Demulsification Experimental Procedure

1. 5 litres of emulsion Miri crude oils were stirred by using S25N-25G stir rod for 15 minutes at 12000rpm.

2. The oil bath was heated up 15 degree Celsius above the expecting temperature while waiting for the emulsions to completely mix up.

3. 300 ml of the sample was measured and been taken out and poured into 400 ml glass bottle.

4. Glass bottle was immersed into the oil bath.

5. Once the sample reached expecting temperature, the sample will be stirred using S25N-25G stir rod for 15 minutes at 12000rpm and expecting volume of demulsifier were added into the sample.

6. The demulsification rig temperature was been set up to it expecting temperature before the sample is been poured into the rig.

7. The sample was stirred for 5 minutes and poured into the demulsification rig.

8. The demulsification rig was run for 30 minutes and the sample was collected and observed.

9. The bottle was been observe over several time ranges, 5 minutes, 15 minutes, 30minutes, 1 hour, 2 hours, 3 hours, and 4 hours.

Std	Run	Block	Factor 1 A:Heating Application Celcius	Factor 2 B:Demulsifier Injection ppm	Factor 3 C:Gas Aeration cc/min	Response 1 Oil Fraction Separated (15th minute) %	Response 2 Oil Fraction Separated (30th minute) %	Response 3 Rag Layer (15th minute) %	Response 4 Rag Layer (30th minute) %	Response 5 Total Cost RM
13	1	Block 1	35.00	400.00	65.00	48.93	55.71	34.82	23.75	6109
24	2	Block 1	80.00	400.00	100.00	22.94	22.94	69.72	66.1	6154
14	3	Block 1	57.50	400.00	65.00	67.33	67.67	32.67	31.67	6132
21	4	Block 1	80.00	200.00	100.00	35.67	36.67	59.83	56.67	3104.4
7	5	Block 1	35.00	600.00	30.00	48	52.55	26.94	22.18	9159
29	6	Block 1	57.50	400.00	65.00	49.67	56.7	35.5	26.67	6132
32	7	Block 1	57.50	400.00	65.00	43.33	42.67	43.33	41.83	6132
15	8	Block 1	80.00	400.00	65.00	28.87	31.69	70.42	66.55	6154.8
1	9	Block 1	35.00	200.00	30.00	48	53.04	35.3	27.13	6109
17	10	Block 1	57.50	600.00	65.00	44	45.33	33.33	30.17	9182
28	11	Block 1	57.50	400.00	65.00	29.48	29.83	69	67.59	6132
31	12	Block 1	57.50	400.00	65.00	39.83	51.83	40	26.67	6132
26	13	Block 1	57.50	600.00	100.00	53.33	53.67	30	28.17	9182
5	14	Block 1	57.50	400.00	30.00	43.33	44.83	53.33	48.33	6132
2	15	Block 1	57.50	200.00	30.00	53.05	52.2	31.69	23.05	3081
16	16	Block 1	35.00	600.00	65.00	51.48	51.85	16.67	14.63	9159
12	17	Block 1	80.00	200.00	65.00	43	45.17	50	46.7	3104.4
6	18	Block 1	80.00	400.00	30.00	48	45.67	35.17	33	6154.8
18	19	Block 1	80.00	600.00	65.00	24	24	76	76	9205.2
30	20	Block 1	57.50	400.00	65.00	26.94	53.51	45.05	25.41	6132
19	21	Block 1	35.00	200.00	100.00	51.83	55.83	26.17	20.83	3058
9	22	Block 1	80.00	600.00	30.00	35	36.57	49.5	46.67	9205.2
23	23	Block 1	57.50	400.00	100.00	54.83	51.5	33.33	30.17	6132
20	24	Block 1	57.50	200.00	100.00	44.67	46.67	25.17	20	3081
22	25	Block 1	35.00	400.00	100.00	61.5	60.28	38.5	39.72	6109
25	26	Block 1	35.00	600.00	100.00	63.13	63.3	17.04	14.33	9159
3	27	Block 1	80.00	200.00	30.00	33.33	36	53.33	49	3104.2
27	28	Block 1	80.00	600.00	100.00	13	13	87	87	9205.2
10	29	Block 1	35.00	200.00	65.00	52.08	60.83	31.25	14.58	3058
4	30	Block 1	35.00	400.00	30.00	51.4	50.84	28.03	22.8	6109
11	31	Block 1	57.50	200.00	65.00	44.56	52.51	36.32	19.12	3081
8	32	Block 1	57.50	600.00	30.00	53.22	52.37	26.44	23.73	9182

Appendix D: Design of Experiment (DOE) Miri Crude Demulsification

Appendix E: Miri CrudeDemulsificationResults

Demulsification : Crude Oil Quality at 35°C

Temperature 35 C, Demulsifier 200 PPM

						Demul	sification P	Periodic Inte	erval Obsei	vation	
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th	30th	1st hour	2nd	3rd	4th
		•				mın	min		hour	hour	hour
13	Series 1	35	200	30	0	48	53.04	55.13	56.17	57.04	57.04
10	Series 2	35	200	65	43.1	52.08	60.83	62.03	60.83	61.46	61.25
14	Series 3	35	200	100	33.83	51.83	55.83	57.17	54.33	55	56.17

Temperature 35 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation 15th 30th 2nd 3rd 4th							
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour	
9	Series 1	35	400	30	51.6	51.4	50.84	57.38	58.13	58.13	28.6	
30	Series 2	35	400	65	0	48.93	55.71	55.36	58.57	60.83	58.4	
3	Series 3	35	400	100	0	61.5	60.28	61.1	60.71	60	31.24	

Temperature 35 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation 5th min 15th 30th 2nd 3rd 4th min min 1st hour hour hour hour						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
15	Series 1	35	600	30	28.91	48	52.55	53.64	53.64	55.27	54.55
26	Series 2	35	600	65	55.93	51.48	61.85	62.6	63.52	63.52	64.26
17	Series 3	35	600	100	8.87	63.13	63.3	62.95	63.16	63.16	63.58

Demulsification :Emulsion Quality at 35°C

Temperature 35 C, Demulsifier 200 PPM

					Demulsification Periodic Interval Observation 15th 30th 2nd 3rd 4th						
Run		Temperature Demulsifier (p	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
13	Series 1	35	200	30	88	35.3	27.13	22.6	19.13	15.3	13.91
10	Series 2	35	200	65	45.6	31.25	14.58	12.5	11.46	10.42	10
14	Series 3	35	200	100	50.33	26.17	20.83	17.33	15.67	13.67	12.5

Temperature 35 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation) 5th min 15th 30th 1st hour 2nd 3rd 4th						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
9	Series 1	35	400	30	28.6	28.03	22.8	16.82	14.95	14.95	22.92
30	Series 2	35	400	65	90.36	34.82	23.75	21.1	16.25	14.64	14.46
3	Series 3	35	400	100	100	38.5	39.72	8.06	8.45	8.45	7.27

Temperature 35 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
15	Series 1	35	600	30	56.36	29.64	22.18	19.1	19.1	14.36	13.64
26	Series 2	35	600	65	26.48	16.67	14.63	13.33	10.74	10.74	9.63
17	Series 3	35	600	100	36.87	17.04	14.33	12.84	11.58	11.58	10.74

Demulsification :ProducedWater Quality at 35°C

Temperature 35 C, Demulsifier 200 PPM

					Demulsification Periodic Interval Observation 15th 30th 2nd 3rd 4th						
Dup		Tomporaturo	Domulaifiar (nnm)	Agration (cc/min)	Eth min	15th	30th	1 ct hour	2nd	3rd	4th
KUN		remperature	Demuisitier (ppm)	Aeration (cc/min.)	Sun min	min	min	ISCHOUL	hour	hour	hour
13	Series 1	35	200	30	12	16.7	19.83	22.27	24.7	27.65	29.04
10	Series 2	35	200	65	11.3	16.67	24.85	25.2	27.7	28.12	28.75
14	Series 3	35	200	100	15.84	22	23.3	25.5	30	31.33	31.33

Temperature 35 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation) 5th min 15th 30th 2nd 3rd 4th min min 1st hour hour hour hour hour						
Run Temperature Demulsifier (ppm) Aeration (cc/min.) 5th							30th min	1st hour	2nd hour	3rd hour	4th hour
9	Series 1	35	400	30	19.8	20.6	26.36	25.8	26.92	26.92	50.09
30	Series 2	35	400	65	9.64	16.25	20.54	23.57	25.17	25.18	27.14
3	Series 3	35	400	100	0	0	0	30.84	30.84	31.55	61.49

Temperature 35 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation)5th min15th30th min2nd3rd4th hour)5th minmin1st hourhourhour						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
15	Series 1	35	600	30	14.73	22.4	25.3	27.3	27.3	30.26	31.82
26	Series 2	35	600	65	13.59	21.85	23.52	24.1	25.74	25.74	26.11
17	Series 3	35	600	100	14.26	19.83	22.37	24.21	25.26	25.26	25.68

Demulsification : Crude Oil Quality at 57.5°C

Temperature 57.5 C, Demulsifier 200 PPM

						Demul	sification F	Periodic Inte	erval Obse	rvation	
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
23	Series 1	57.5	200	30	49.15	53.05	52.2	54.55	58.31	62.03	62.03
12	Series 2	57.5	200	65	23.33	44.56	52.1	52.11	53.15	53.33	63.86
20	Series 3	57.5	200	100	49.5	44.67	46.67	53.67	55.83	56	56

Temperature 57.5 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
27	Series 1	57.5	400	30	43.33	43.33	44.83	48.83	50	50	51.33
AVERAGE	Series 2	57.5	400	65	42.40	42.76	50.37	53.59	53.30	55.09	55.03
25	Series 3	57.5	400	100	49.83	54.83	51.5	49	51.17	52.5	52.33

Temperature 57.5 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation						
Run Series 1		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
2	Series 1	57.5	600	30	39.83	53.22	52.37	49.83	52.71	51.53	52.71
32	Series 2	57.5	600	65	36.5	44	45.33	44.83	48	46.67	48
16	Series 3	57.5	600	100	48.17	53.33	53.67	54.33	56.83	56.67	56

Demulsification :Emulsion Quality at 57.5°C

Temperature 57.5 C, Demulsifier 200 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
23	Series 1	57.5	200	30	40.68	31.69	23.05	14.92	10.85	6.78	6.1
12	Series 2	57.5	200	65	62.81	36.32	19.12	19.12	15.26	14.04	14.04
20	Series 3	57.5	200	100	28.5	25.17	20	10.5	10.5	10	10

Temperature 57.5 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
27	Series 1	57.5	400	30	56.67	53.33	48.33	41.83	40	40	34.5
AVERAGE	Series 2	57.5	400	65	50.72	31.74	33.67	28.04	25.51	14.15	21.67
25	Series 3	57.5	400	100	40.17	33.33	30.17	28.83	23.33	20.83	20.67

Temperature 57.5 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
2	Series 1	57.5	600	30	44.75	26.44	23.73	22.71	18.74	16.78	11.02
32	Series 2	57.5	600	65	41.67	33.33	30.17	28.83	23.83	23.33	21
16	Series 3	57.5	600	100	35	30	28.17	25.5	17.5	17	17.33

Demulsification :ProducedWater Quality at 57.5°C

Temperature 57.5 C, Demulsifier 200 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
23	Series 1	57.5	200	30	10.17	15.25	24.75	30.51	30.85	31.19	31.86
12	Series 2	57.5	200	65	13.86	19.12	28.77	28.77	28.77	36.63	32.63
20	Series 3	57.5	200	100	22	30.17	33.33	35.83	33.67	34	34

Temperature 57.5 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (nnm)	Aeration	5th min	15th min	30th min	1st hour	2nd	3rd	4th bour
27	Series 1	57 5	400	30	0	3 33	6.83	9 33	10	10	14 17
AVERAGE	Series 2	57.5	400	65	7.25	12.99	15.96	18.37	21.24	22.34	23.22
25	Series 3	57.5	400	100	10	11.83	18.33	22	25.5	26.67	30

Temperature 57.5 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
2	Series 1	57.5	600	30	15.42	20.33	23.9	27.46	28.5	31.7	36.27
32	Series 2	57.5	600	65	21.83	22.67	24.5	26.33	28.17	30	31
16	Series 3	57.5	600	100	10.17	16.67	18.17	20.17	25.67	26.33	26.67

Demulsification : Crude Oil Quality at 80°C

Temperature 80 C, Demulsifier 200 PPM

					Demulsification Periodic Interval Observation							
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour	
29	Series 1	80	200	30	34.33	33.33	36	40.67	48.33	48.33	49.5	
19	Series 2	80	200	65	42.67	43	45.17	50	46.5	47.33	47.67	
21	Series 3	80	200	100	36.67	35.67	36.67	37.17	40.5	44	43.17	

Temperature 80 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation							
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th	1st hour	2nd	3rd	4th	
									noui	noui	noui	
1	Series 1	80	400	30	44.67	48	45.67	45.67	44.5	49	50	
4	Series 2	80	400	65	16.55	28.87	31.69	36.44	36.67	39.79	42.78	
5	Series 3	80	400	100	25.5	22.94	22.94	30.64	30.28	44.04	48.62	

Temperature 80 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation							
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour	
7	Series 1	80	600	30	39.67	35	36.67	33.17	41	41.67	44	
6	Series 2	80	600	65	24	24	24	24.33	25	26	24	
8	Series 3	80	600	100	13	13	13	39	39	39.33	39.33	

Demulsification :Emulsion Quality at 80°C

Temperature 80 C, Demulsifier 200 PPM

					Demulsification Periodic Interval Observation							
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	(cc/min.) 5th min 15th 30th 1st hour					3rd hour	4th hour	
29	Series 1	80	200	30	55	53.33	49	33.5	23.67	23.33	21.67	
19	Series 2	80	200	65	51.67	50	46.7	40	39.83	38.33	37.67	
21	Series 3	80	200	100	60	59.83	56.67	53.33	49.5	43.33	40.5	

Temperature 80 C, Demulsifier 400 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
1	Series 1	80	400	30	39.5	35.17	33.33	32.67	32.33	27.67	23.33
4	Series 2	80	400	65	83.5	70.42	66.55	60.04	52.82	43.33	41.37
5	Series 3	80	400	100	70.82	69.72	66.1	56.51	55.04	38.53	33.03

Temperature 80 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
7	Series 1	80	600	30	50	49.5	46.67	45	35.17	33.33	30
6	Series 2	80	600	65	76	76	76	65.67	54.83	53.33	52.67
8	Series 3	80	600	100	87	87	87	61	61	60	59.67

Demulsification :Produced Water Quality at 80°C

Temperature 80 C, Demulsifier 200 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	min.) 5th min 15th min 10.37 13.33	30th	1st hour	2nd	3rd	4th	
						mın	min		hour	hour	hour
29	Series 1	80	200	30	10.37	13.33	15	25.83	28	28.33	28.83
19	Series 2	80	200	65	5.67	6.67	8.33	10	13.67	14.33	14.67
21	Series 3	80	200	100	3.33	4.5	6.67	9.5	10.17	12.64	16.33

Temperature 80 C, Demulsifier 400 PPM

_					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
1	Series 1	80	400	30	15.83	16.83	21	21.67	23.17	23.33	26.67
4	Series 2	80	400	65	0	0.7	1.76	3.5	10.21	14.44	15.85
5	Series 3	80	400	100	3.67	7.34	11.01	12.84	14.68	17.43	18.34

Temperature 80 C, Demulsifier 600 PPM

					Demulsification Periodic Interval Observation						
Run		Temperature	Demulsifier (ppm)	Aeration (cc/min.)	5th min	15th min	30th min	1st hour	2nd hour	3rd hour	4th hour
7	Series 1	80	600	30	13.33	15.5	16.67	21.83	23.83	25	26
6	Series 2	80	600	65	0	0	0.33	10	20	20.67	23.33
8	Series 3	80	600	100	0	0	0	0	0	0.67	1