Minimum Miscibility Pressure Investigation for Carbon Dioxide Injection with Different Malaysian Light Oil Samples

by

Saw Li Juan

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Petroleum Engineering Programme
Universiti Teknologi PETRONAS
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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS
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May 2012

CERTIFICATION OF ORIGINALITY

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

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SAW LI JUAN

ABSTRACT

As the global demand for oil continues to rise, relying on primary and secondary oil recovery methods alone are just inadequate. In Malaysia, there is approximately one billion barrels of oil that can still be produced through enhanced oil recovery (EOR) techniques. One of the tertiary oil recovery techniques that are implemented is the carbon dioxide (CO_2) flooding. Carbon dioxide flooding has been applied globally as miscible, near miscible or immiscible flooding, depending on the reservoir characteristics and oil composition. By measuring the minimum miscibility pressure (MMP), the mode of the displacement can be determined if it is miscible or immiscible. The purpose of this project is to investigate the CO_2 MMP of different Malaysian light oil samples by using the Vanishing Interfacial Tension (VIT). Published correlations were then referred to and compared with the experiments' results.

This study has found that the CO_2 MMP for Angsi and Dulang are 3077 psia and 2957 psia respectively. In comparison the experimental results with several published correlations, (Cronquist, 1977) correlation gave the best predicted MMP for both reservoir oil. The effects of each parameters that affects CO_2 MMP for instance, reservoir temperature and oil composition were discussed in this paper. This study has also demonstrated the reliability of the VIT technique in predicting MMP by pendant drop method experiments in comparison with the other published correlations.

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INTRODUCTION

1.1 Background of Study

Existing primary and secondary oil recovery drives are currently inadequate to meet the rising worldwide oil demand. A substantial amount of hydrocarbons are estimated to be still trapped underground even after primary and secondary recovery. According to Samsudin *et al*., (2005) the estimated oil-in-place from the producing fields in Malaysia stands at approximately 17.0 BSTB, as of January 2005. With the estimated ultimate recovery (EUR) of 5.62 BSTB, this translates into an average recovery factor of 33 percent for the producing fields in Malaysia.

A screening study was completed in year 2000 on seventy-two (72) reservoirs in Malaysia and have identified that approximately a billion barrels of additional reserves can still be gained through tertiary recovery (Hamdan et al., 2005). At present, several tertiary oil recovery projects are in their design or implementation stage to tap this remaining oil-in-place and at the same time, cope with the increasing global oil demand.

One of the major tertiary methods used globally is the injection of CO_2 into the reservoir. CO_2 injection has received substantial attention in the oil and gas industry due to its high displacement efficiency and relatively low cost (Yellig & Metcalfe, 1980). Carbon dioxide flooding is the second most applied enhanced oil recovery method after steam flooding. Carbon dioxide flooding has been practiced either as miscible, near miscible or immiscible displacement (Shedid *et al*., 2008).

1.2 Problem Statement

For CO_2 flooding projects, the CO_2 MMP is an important parameter to be considered for screening and selecting reservoirs. The CO_2 MMP is defined as the pressure at which the injected gas and the contacted oil in place become miscible with each other resulting in a very efficient displacement process. Miscible CO_2 displacement can only be achieved when the CO_2 is injected at a pressure higher than the MMP, in which the MMP must also be lower than the reservoir pressure. Besides that, every reservoir oil sample has its own unique MMP with CO_2 as each oil sample has its own distinctive oil composition. Thus it is critical to rapidly determine the MMP for each reservoir oil sample when screening for CO_2 flooding projects.

1.3 Objectives and Scope of Study

The objectives of this project are:

- To rapidly determine the CO_2 MMP of different Malaysian light oil samples at reservoir temperature and varying pressures.
- To evaluate and compare the results with suitable published correlations used for minimum miscibility pressure predictions.

Firstly, this project aims to determine the minimum miscibility pressure (MMP) of CO_2 injection with different light oil samples from different Malaysian fields. By using the vanishing interfacial tension technique, the interfacial tension (IFT) between crude oil and CO_2 can be determined at reservoir temperature and varied pressures. A function of pressure and IFT is plotted and then extrapolated to zero IFT, to determine the MMP. The results are then compared with published correlations.

1.4 Significance of the Project

Through this experimental study, the minimum miscibility pressure (MMP) of the light oil samples will be measured and determined whether miscible CO_2 flooding is viable or not in the respective reservoirs. The accuracy of rapid determination of MMP through VIT method is also evaluated.

1.5 Relevancy of the Project

CO_2 injection is one of the popular Enhanced Oil Recovery methods due to its high displacement efficiency and relatively low cost. In the oil and gas industry, CO_2 MMP is an important parameter in screening and selecting reservoirs that are suitable for CO_2 injection. For higher oil recovery, it is vital that the reservoir has an average reservoir pressure greater than the CO_2 MMP.

1.6 Feasibility of the Project within the Scope and Time Frame

The early phase of the project was mostly done on reading SPE papers, technical papers, journal papers and books to gain a better understanding of the research project. The slim tube and interfacial tension apparatus is readily available in the university. However, due to the limited time frame of the project, the number of samples will be restricted. Slim-tube test is the most common and standard technique of determining MMP, but this method is time consuming and expensive. On the other hand, the VIT technique is express and requires approximately 3 hours in determining the interfacial tension at different pressures. After that the MMP can be determined by extrapolating the IFT vs Pressure to zero. Gas Chromatograph (GC) is done for the oil samples to identify the oil composition, and MMP prediction is calculated using published correlations. MMP from published correlation and experimental study is compared. With the help and guidance from the dedicated supervisor and technicians, this project was completed within the time frame given.

CHAPTER 2

LITERATURE REVIEW

2.1 CO_2 Flooding

CO_2 flooding is the second most practiced EOR method after steam flooding (Shedid *et al*., 2008). CO_2 flooding has been proven to be effective in prolonging production life of light oil fields nearing depletion that were under waterflooding by 15 to 20 years. Furthermore, CO_2 injection is also capable of recovering 15% to 25% of the original oil in place. Some of the main factors contributing to the oil recovery in CO_2 flooding are low IFT, viscosity reduction, oil swelling, formation permeability improvement, solution gas flooding and density change of oil and water (Yongmao *et al*., 2004).

Carbon dioxide injection is preferred compared to the other gases like ethane, propane and nitrogen because it is cheaper, higher density and provides environmental benefits in CO_2 storage of the reservoir (Dong *et al.,* 1999). Besides that, by using CO_2 as the injection gas, the miscibility of CO_2 and oil can be achieved at a lower pressure compared with hydrocarbon gases and nitrogen (Ghedan, 2009; Rathmell *et al*., 1971; Yellig, 1985).

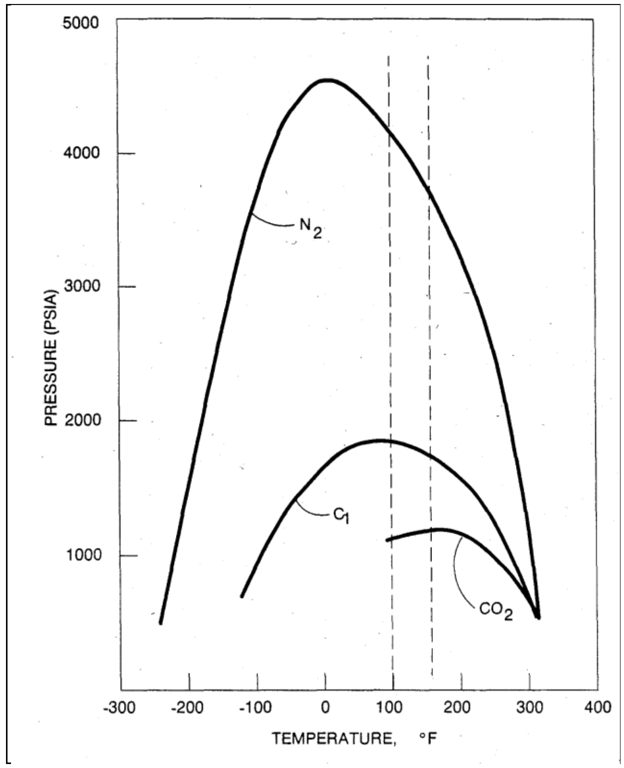


Figure 1: MMP curves of three fluids under reservoir conditions of pressure and temperature

Reference: Injection Of Steam and Solvent for Improved Oil Recovery (Yellig, 1985)

2.2 Miscibility Mechanisms

Miscibility is defined as the ability of two or more fluid substances (gases or liquids) to form a single homogeneous phase when mixed in all proportions. When two fluid phases are formed after some proportion of one fluid is added, those fluids are immiscible (Holm, 1986).

There are two types of miscibility mechanisms, first contact and multiple contacts. First contact miscible solvents will mix directly with the reservoir oil at any ratio, forming a single phase. However, first contact miscibility is only achievable for highly rich gases or at high pressures for lean systems which are usually too costly for continuous injection. Multiple contact miscibility consists of two types of mechanisms, vaporizing gas drive and condensing gas drive.

In vaporizing gas drive, a lean gas is injected into the reservoir and vaporizes methane to liquid petroleum gas (LPG) components from the reservoir oil as it travels through the reservoir and becomes miscible with the virgin reservoir fluid when the gas has vaporized sufficient hydrocarbons. On the other hand, in condensing gas drive, the enriched gas is injected into the reservoir and condenses heavier components to the oil, thus enriching the oil, making it miscible with the freshly injected enriched gas (Holm, 1986).

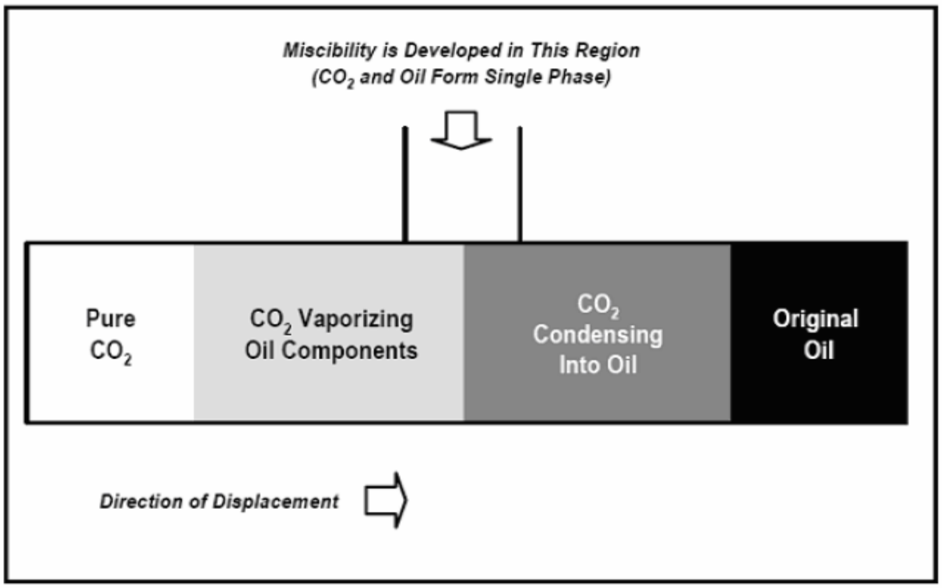


Figure 2: Multiple Contact Miscibility Mechanisms

Reference: Experimental Investigation of CO_2 – Miscible Oil Recovery at Different Conditions (Suleman, 2008)

2.3 Minimum Miscibility Pressure (MMP)

Martin and Taber (1992) stated that in a miscible CO_2 injection, the CO_2 phase and oil phase will combine and flow together, thus achieving higher oil recovery rate compared to immiscible CO_2 injection. The miscible oil recovery of a reservoir can be achieved by CO_2 displacement at a pressure level greater than a certain minimum. This minimum pressure is defined as the CO_2 MMP (Yellig & Metcalfe, 1980).

Many researchers have conducted investigations to determine the factors that could influence the CO_2 MMP for a reservoir fluid. According to Dong (1999), the CO_2 MMP for a reservoir fluid can be affected by the reservoir temperature, oil composition and purity of the injected CO_2. As the reservoir temperature increases, so does the CO_2 MMP.

Yellig (1985) discovered that the CO_2 MMP increases with temperature until it reaches a maximum pressure, then decreases with increasing temperature. He found that there were two temperatures at which a multiple-contact-miscibility gas can be miscible with oil at reservoir pressure.

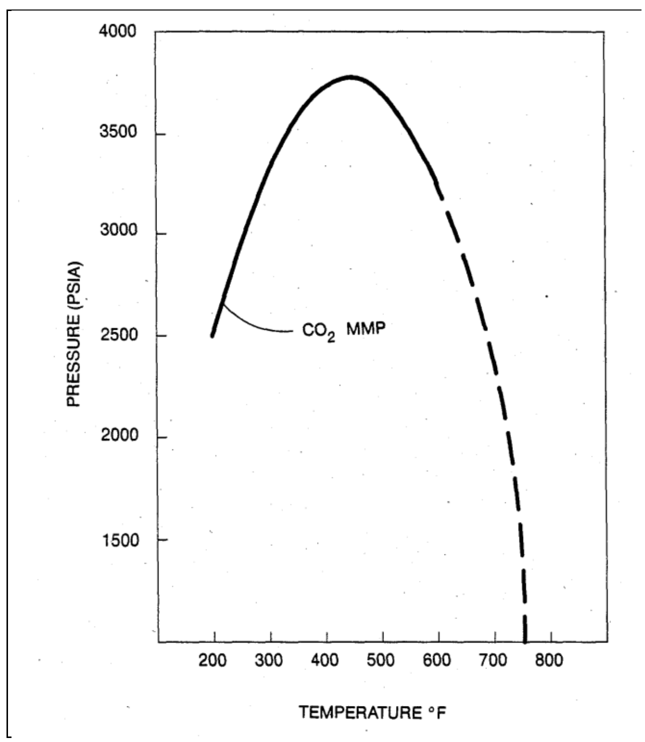
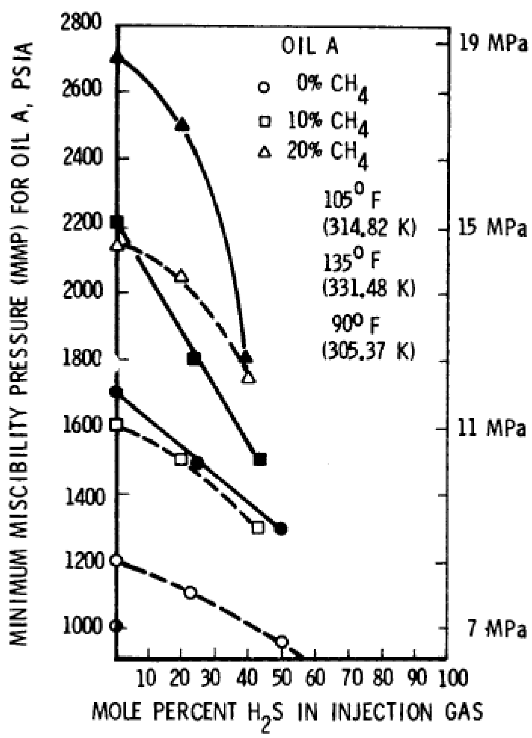


Figure 3: Relationship of pressure and temperature in determining CO_2 MMP
Reference: Injection Of Steam and Solvent for Improved Oil Recovery (Yellig, 1985)

Holm (1987) discovered that at reservoir temperature, miscibility displacement of oil with CO2 can be achieved at lower pressures, when the C_5 to C_{30} content is higher. He also stated that C_5 to C_{12} content of the oil has the highest effect on the miscibility pressure. However, high heavy oil components will require higher pressure to achieve miscibility.

Besides that, the presence of hydrogen sulfide (H_2S), ethane (C_2H_6), or intermediate hydrocarbons such as propane (C_3H_8) and butane (C_4H_{10}) can reduce the CO_2 MMP but an opposite outcome with the presence of methane (CH_4) or nitrogen (N_2) in CO_2 can significantly increase the CO_2 MMP (Holm, 1987).

Metcalfe (1982) observed that CO_2 streams containing H_2S and/or LPG components will lower the MMP compared to pure CO_2 streams, while methane in the CO_2 stream will increase the MMP. H_2S was found to be more effective in reducing the MMP at higher concentrations of methane. Propane and butane are effective in MMP reduction, depending on the concentration and levels involved.



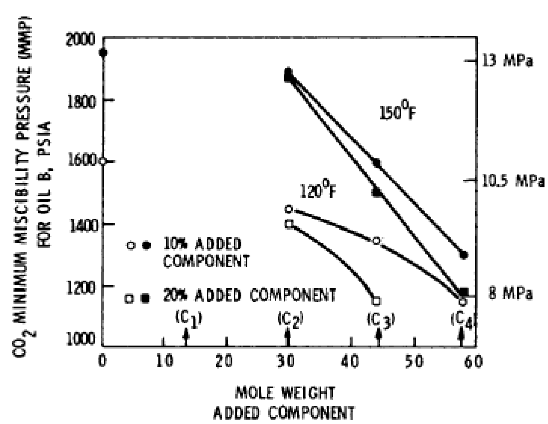


Figure 4 (Left): Effect of temperature and methane mixed with H_2S on CO_2 MMP

Figure 5 (Right): Effect of temperature and mole weight of added component on CO_2 MMP

Reference: Effects of Impurities on Minimum Miscibility Pressures and Minimum Enrichment Levels for CO2 and Rich-Gas Displacements (Metcalfe, 1982)

Rathmell *et al*., (1971) also conducted miscible displacement investigations using CO_2 and observed that the presence of methane content in the reservoir fluid will increase the minimum miscibility pressure.

2.4 Minimum Miscibility Pressure Determination Techniques

There are several ways to measure the MMP between a certain oil and CO2. The widely recognized experimental methods used to estimate gas-oil miscibility conditions under reservoir conditions are the slim tube, rising bubble and the vanishing interfacial tension technique.

2.4.1 Slim Tube Method

According to Aleidan & Mamora (2011), this experimental method is considered to be the most accurate approach and the industry regards the slim tube apparatus as the standard method in measuring the MMP.

The slim tube apparatus has been designed to create an environment where viscous fingering is minimized by transverse dispersion if the tube is small in diameter and low in displacement rate. Also, by making the slim tube longer in length, the relative length of any viscous fingering is small in comparison to the scale of the tube length (Mogensen *et al*., 2009).

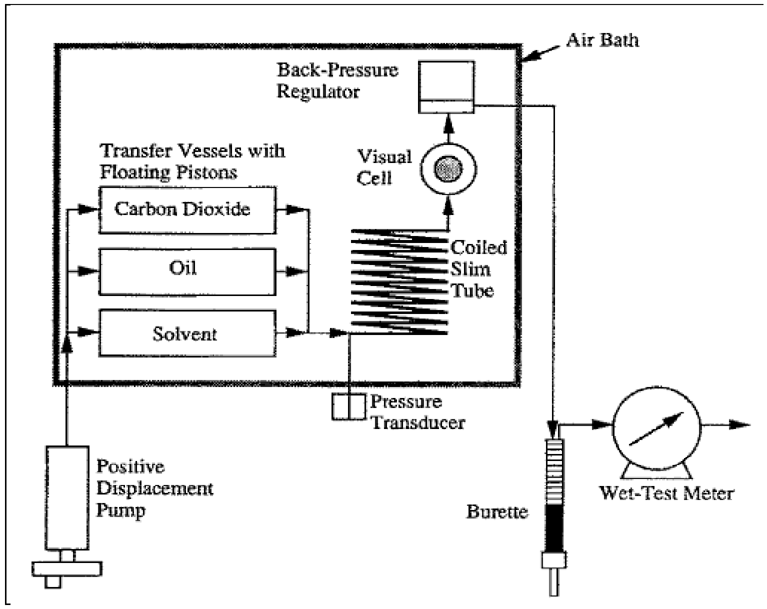


Figure 6: Slim Tube Apparatus Schematic

Reference: (Elsharkawy et al., 1992)

For this technique, the MMP is defined as the pressure where oil recovery approaches 80% at CO_2 breakthrough time or the final oil recovery reaches 90% - 95% at 1.2 pore volume of CO_2 injection. As the flooding pressure increases, the oil recovery will increase. However, the recovery range will become very small as the pressure increases at the MMP or higher. Thus the MMP is determined to be at the position of inflexion on the curve of oil recovery with flooding pressure (Ghedan, 2009).

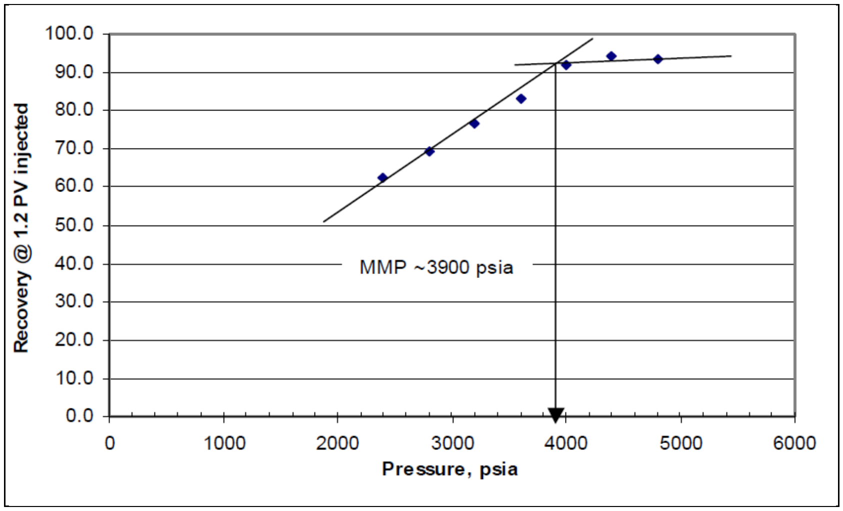


Figure 7: Recovery at 1.2 PV CO2 injected vs test pressure using slim-tube

Reference: Optimization of Carbon Dioxide Flooding For a Middle-Eastern Heterogeneous Oil Reservoir (Shedid et al., 2008)

Aleidan, (2011) conducted experimental studies using slim tube to determine CO_2 MMP with west Texas oil. He then made a comparison with nine published correlations and concluded that the Holm and Josendal, and Cronquist correlations predicted the MMP with the highest accuracy.

Shedid *et al*., (2008) conducted MMP investigations using slim tube with live crude oil, API ranging from 33.2 to 37.6 at the reservoir temperature and found the CO_2 MMP to be 3900 psia. He mentioned that a good initial estimate of MMP is required before the experiment is conducted as the accuracy of the slim tube depends on the intervals chosen.

Yongmao *et al*., (2004), conducted laboratory study using slim tube equipment to determine the MMP of the recombined reservoir fluid from Shengli Oil Field of China. The MMP was determined as 26 MPa and they concluded that it was better to determine the MMP by the position of inflexion on the curve of oil recovery versus flooding pressure rather than by reaching a special oil recovery point.

2.4.2 Rising Bubble Apparatus (RBA)

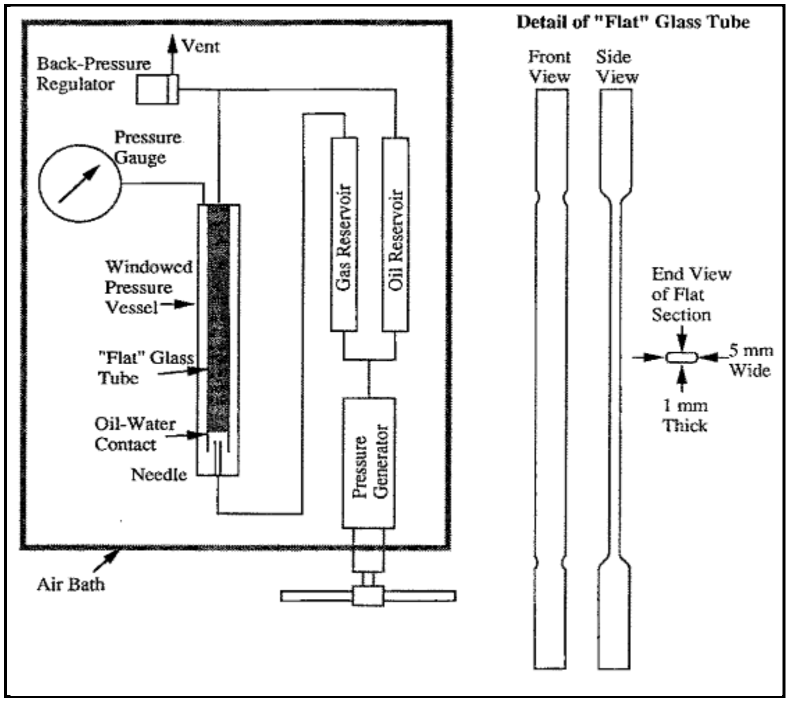


Figure 8: Rising Bubble Apparatus Schematic

Reference: (Elsharkawy et al., 1992)

The rising bubble apparatus was designed in the early 1980s, with features like a flat glass tube mounted vertically so that the evolution of shape of bubbles rising through the oil column can be observed clearly, and a hollow needle at the bottom is used to inject a bubble of gas, where the buoyant force of the gas will lift the gas bubble through the column and mix with oil.

Two advantages of using RBA to measure MMP is that RBA does not consume as much oil and gas as the slim tube method, and the RBA can visually demonstrate the pressure where miscibility occurs (Elsharkway *et al.*, 1992).

The behavior of the bubble rising through the oil column, changes into different shapes from “spherical”, to “ellipsoidal”, to “ellipsoidal cap”, and to “skirted ellipsoidal cap” as the interfacial tension between the oil and gas decreases to zero. The progress of shape of the bubbles indicates the MMP.

Elsharkawy *et al.,* (1992), Christiansen & Haines (1987) used RBA method to measure the MMP and made observations on the changes of the shape of bubble as it rises through the oil for vaporizing and condensing gas process.

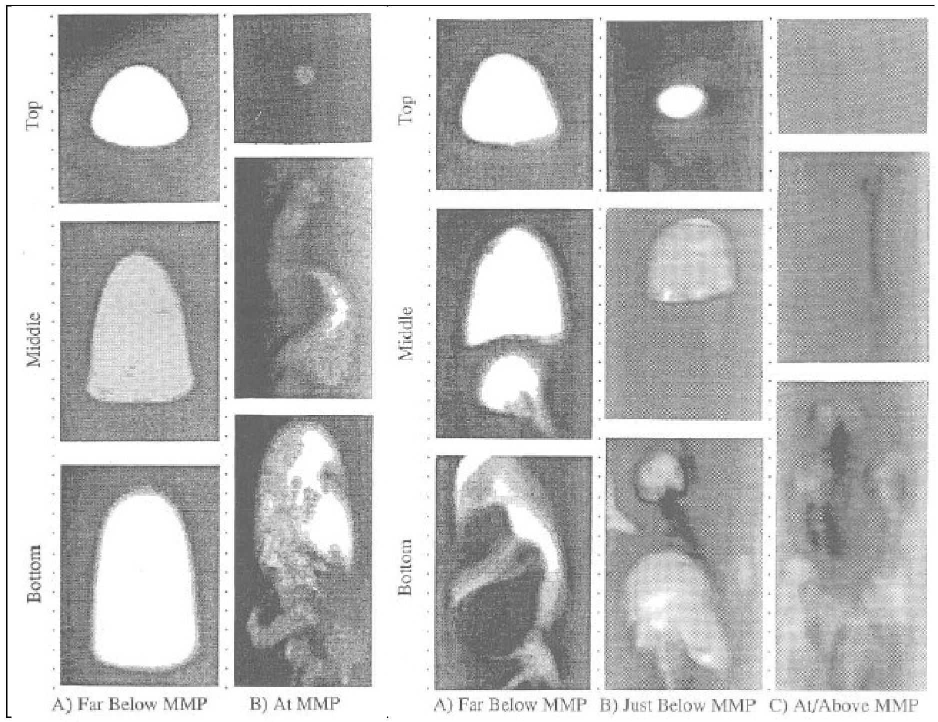


Figure 9 (Left): Bubble Behavior for Vaporizing Gas Process

Figure 10 (Right): Bubble Behavior for Condensing Gas Process

Reference: (Elsharkawy et al., 1992)

2.4.3 Vanishing Interfacial Tension

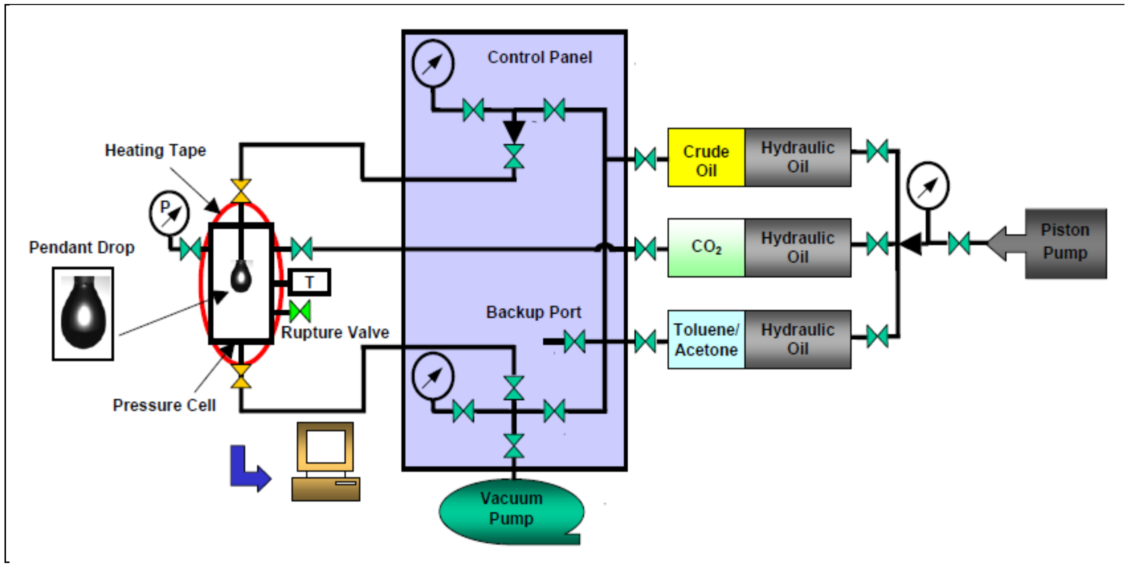


Figure 11: Block diagram of the experimental setup used to study the interfacial tension interactions

Reference: (Yang & Gu, 2004)

Rao & Lee (2003) defined that miscibility requires the absence of an interface between the injected gas and crude oil at reservoir conditions. The VIT concept is based on the concept that the interfacial tension between two immiscible fluids will continuously diminish and become zero at the point of miscibility.

According to Yang & Gu (2004), this method is the most advanced and accurate method of measuring the IFT at large range of pressures and temperatures. During the experiment, a pendant oil drop is produced at the tip of the syringe needle. By using an image acquisition system, the digital image of the drop is captured. Via computer digital image analysis and processing techniques, an accurate interfacial profile of the pendant drop is acquired. After that, by using the Laplace equation of capillarity, it will find the best fit for the numerically calculated interfacial profile to the physically observed drop profile, which will determine the IFT of the oil drop. The IFT measurements are repeated for at least four pendant drops to ensure that the results obtained are satisfactory.

Ayirala & Rao (2006) discussed the effectiveness of using the VIT method for gas/oil miscibility determination. The IFT between gas and oil is measured at reservoir temperature and different pressures. Miscibility conditions are observed by extrapolating the plot of IFT versus pressure to zero IFT. The VIT technique has been used effectively to optimize the injection gas compositions in two miscible gas-injection projects, one in Rainbow Keg River (RKR) reservoir, Alberta, and another in the Canadian Terra Nova offshore field. (Ayirala & Rao, 2006)

2.5 CO_2 MMP Published Correlations

Most published correlations predict CO_2 MMP as a function of three variables; temperature, the molecular weight of a plus fraction and the mole fraction of a light component in the reservoir oil. Holm & Josendal (1982) found that CO_2 displacement is equivalent to 59 mole % methane and 41 mole % propane mixtures. Holm and Josendal correlation uses temperature, C_{5+} molecular weight, C_5-C_{30} content, and CO_2 density. They concluded that C_2 through C_4 content has negligible effect on miscible displacement and methane content does not affect the MMP significantly.

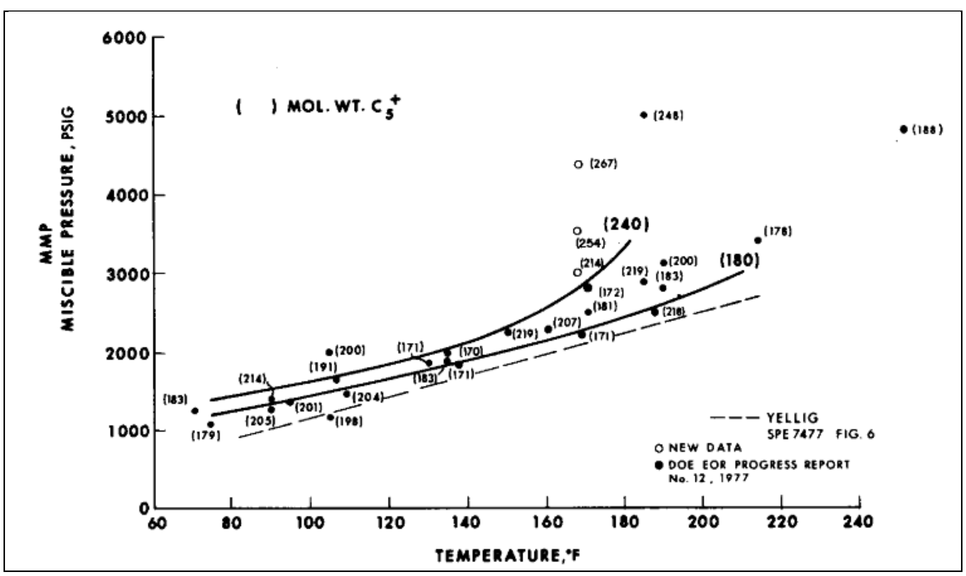


Figure 12: Holm-Josendal dynamic miscibility displacement correlation for CO_2

Reference: (Holm & Josendal, 1982)

Cronquist (1978) proposed correlation takes into account three parameters; reservoir temperature, molecular weight of C_{5+} and mole % of C_1. This correlation covers a wide range of API gravities and temperatures. Cronquist found that the molecular weight of C_{5+} fraction was a good correlation parameter for MMP.

Yellig & Metcalfe (1980) proposed a correlation which only varied as a function of temperature. Oil composition was concluded to have no minor or no significant effect on MMP. They suggested also that the CO_2 MMP should always be equal to or greater than the reservoir oil's bubble point pressure.

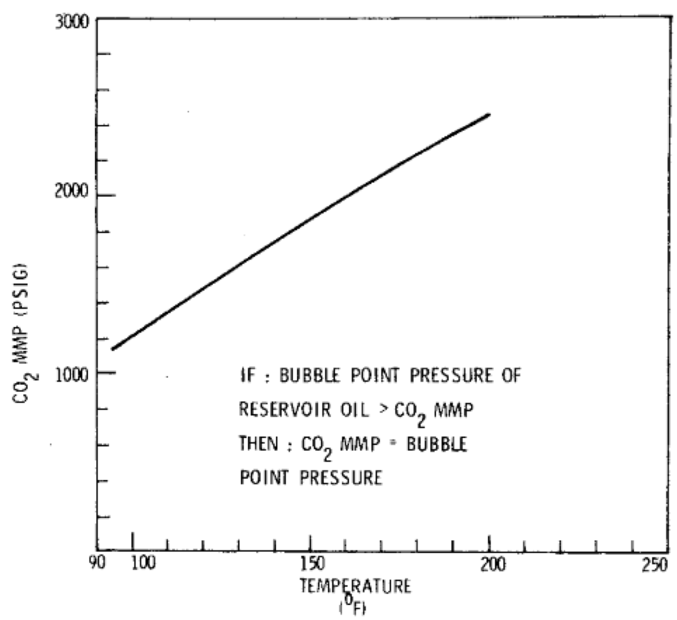


Figure 13: Temperature/bubble-point pressure of CO2 MMP correlation

Reference: (Yellig & Metcalfe, 1980)

Glaso (1985) proposed a generalized MMP correlation for N2, CO2, and LPG. His correlation was based on the Benham correlation, but predicted any multiple contact miscible displacement. He also found the CO2 solubility to be similar to the mixture containing 58 mole % methane and 42 mole % propane, which was similarly found by Holm and Josendal. Glaso correlation required input parameters are mole % of $C_2 - C_6$ intermediate content, molecular weight of C_{7+} and reservoir temperature.

Yuan *et al.* (2005) developed a correlation for pure and impure CO_2 displacements of multi-component oil using analytical gas flooding theory. The parameters for this correlation are reservoir temperature, molecular weight of C_{7+} and the intermediate component ($C_2 - C_6$) in the oil. For pure CO_2 MMP displacement correlation, a data set consisting of seventy analytically calculated MMPs from nine reservoir oils were used. Thus this correlation is limited to the nine oils used in their study.

According to Aleidan (2011), this method is often used for preliminary calculations of CO_2 MMP and he advised not to rely only on the correlations as the final result. He suggested using published correlations to get an estimate of the starting pressure for slim tube experiments. A total of nine correlations were used in his studies to estimate the MMP. He found that for dead oil samples, correlations that do not include light fractions will give a better predicted value.

Mogensen *et al.* (2009) discussed that empirical correlations are generally over predicting the MMP for light oils and underestimating the MMP for heavy oils. His studies indicated that the correlations had limited use when applied outside the range of data to which they were fitted.

2.6 Literature Review Summary

CO_2 flooding can increase the production life of light oil fields under waterflood by 15 to 20 years and is capable of recovering 15% to 25% of its original oil in place. The benefits of CO_2 flooding to the oil recovery are low interfacial tension, viscosity reduction, oil swelling, formation permeability improvement, solution gas flooding and density change of oil and water. Miscibility is achieved when two or more fluid substance mixes in all proportions and forms a single homogenous phase. There are two types of miscibility mechanisms, which are first contact and multiple contacts. First contact miscibility is only achievable for highly rich gases or at high pressures for lean systems, while multiple contact miscibility mechanisms consists of the vaporizing and condensing gas drives. The minimum miscibility pressure is defined as the minimum pressure required for the oil and gas (CO_2) to be miscible and flow together, thus obtaining higher oil recovery. The CO_2 MMP for a reservoir fluid can be affected by factors such as reservoir temperature, oil composition and purity of the injected CO_2. To determine the CO_2 MMP, the typical slim tube and rising bubble methods are popularly used. However, the VIT technique for pendant drop has recently gained popularity for its rapid miscibility determination. The VIT technique uses computer image analysis to capture the pendant drop and compute the IFT using Laplace Equation. IFT against pressure is plotted and extrapolated to zero IFT to determine the miscibility conditions. Published correlations such Cronquist (1978), Yellig & Metcalfe (1980), Glaso (1985) and Yuan (2011) were discussed about the limitations and parameters required of using their correlations. Published correlations are suggested to be used for starting pressure estimation in slim tube experiments. However, published correlations tend to over predict the MMP for light oils and under predict for heavy oils.

CHAPTER 3

METHODOLOGY / PROJECT WORK

3.1 Research Methodology

In the early stage of the project, researching, data gathering and literature reviews are done to further understand the research topic and the problem statement. Research work is done to gain more knowledge about the EOR methods, CO_2 flooding, miscibility mechanisms and MMP. After that, numerous methods for MMP determination were identified and the VIT method was chosen.

Before starting on the IFT measurements, the density of CO_2 and crude oil will have to be measured first. The density of CO_2 was obtained from a standard property table at different pressures and temperatures (http://webbook.nist.gov/chemistry/), while the density of the crude oil was measured using Anton Paar Density Meter, where the crude oil density was measured at different temperatures, and then extrapolated to the reservoir temperature.

Then, by using the VINCI Technologies Interfacial Tensometer, the IFT between the crude oil and CO_2 is measured at reservoir temperature and varying pressures. The IFT obtained is then plotted as a function of pressure to zero IFT. The CO_2 MMP for the measured crude oil is then determined.

Next, a detailed compositional analysis of each crude oil sample is obtained using the Shimadzu Gas Chromatograph GC-2010 and through empirical published correlations, a comparison is made between the MMP for each crude oil sample. For each light oil sample, the differences will be calculated and written down. An oral presentation about the analyzed results of the experiments will be done and a dissertation report is prepared.

3.1.1 Density Measurement

Figure 14: Anton Paar Density Meter 4500 M

To measure the density of each crude oil sample, the Anton Paar Density Meter is used. The density of the crude oil sample is needed for the VIT experiment using the IFT equipment.

Procedure:

1. Crude oil sample is prepared by heating and stirring before drawing some into a 3ml syringe. Heating and stirring of the crude oil is to ensure the sample is not too viscous to flow and is mixed evenly.

2. The density meter is turned on, and crude oil is injected into the density meter until the U-tube in the density meter is filled all the way.

3. Ensure that there is no air bubbles in the U-tube of the density meter. Air bubbles will affect the density measurements and the error will be highlighted in the density meter's graphical-user-interface (GUI).

4. From the GUI, the temperature is set to start from 40°C to 89°C. The density is recorded for every increment of 10°C, and then extrapolated to obtain the density of the crude oil at reservoir temperature.

3.1.2 Interfacial Tension Measurement

Figure 15: VINCI Technologies Interfacial Tensometer IFT 700

For this experiment, the VINCI Technologies Interfacial Tensometer IFT 700 is used to measure the interfacial tension between the crude oil and CO_2 gas with the VIT method. By producing a pendant drop in the chamber, the drop shape image is captured and computed. The interfacial tension is then calculated by solving the algorithm of the Laplace equation.

Procedure:

1. The equipment is setup for a pendant drop, where the capillary injector is plugged at the top of the cell.

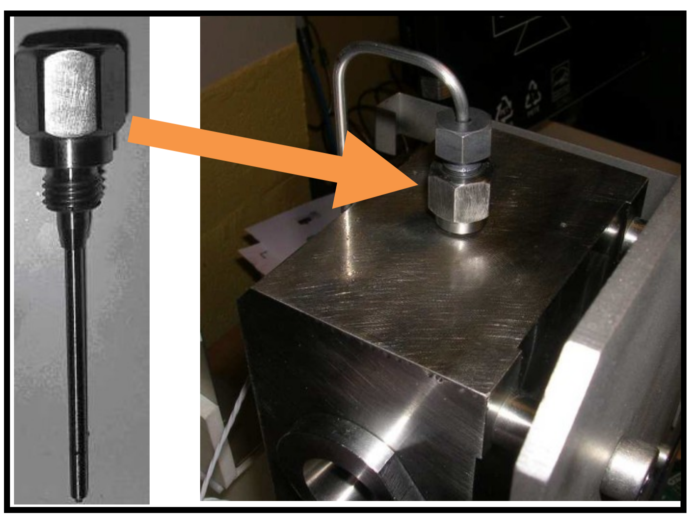


Figure 16: Configuration of capillary injector for pendant drop

2. Ensure that the IFT equipment has been correctly cleaned. Incomplete cleaning, dusts and traces of previous sample would affect the accuracy of the results.
3. Temperature of the accumulator and cell are set to reservoir temperature. Alarm is set 5°C above the reservoir temperature.
4. CO_2 gas is placed on the right inlet valve, while the crude oil sample is injected into the left side.
5. After reaching the desired temperature, the pressure is increased slowly to the desired test pressure by injecting CO_2. Increasing the pressure too rapidly could cause the CO_2 to fog the cell.
6. Allow some time for the fluids to achieve equilibrium state as the two temperature probes shown are the temperature of the stainless steel body, not the temperature of the fluid.
7. A drop of crude oil is then produced by using the hand pumps.
8. Video settings such as Focus, Histogram, Optical Calibration are carried out from the Workshop menu.
9. For the software measurement setup, the densities for crude oil and CO_2 are keyed in. Frontier setup and one image analysis are also attuned.
10. Measurements for IFT are then run for 30 seconds. For every second, there will be an IFT computed.
11. Results are then saved in Microsoft Excel form.
12. Step 8 is repeated for different test pressures, while Step 9 is repeated for every new drop produced.

3.1.3 Gas Chromatograph (GC)

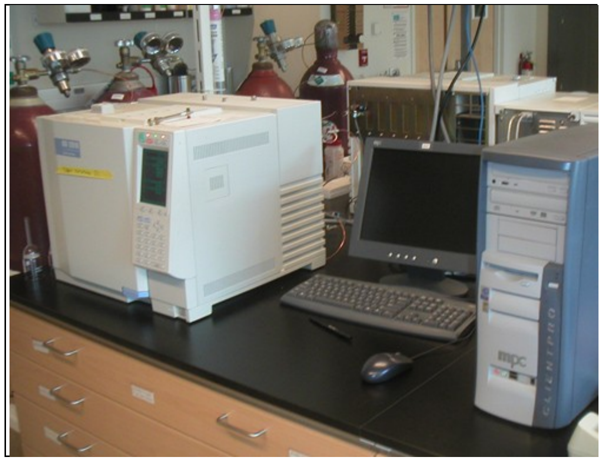


Figure 17: Shimadzu Gas Chromatograph GC-2010

The Shimadzu Gas Chromatograph GC-2010 was used to analyze the oil composition of the sample. The gas chromatograph separates the various components in the sample and determines the amount of each component is present. The important part of using this equipment are the injection port where the samples are loaded, a "column" which the components are separated, a regulated flow of a carrier gas (often helium) which will push the sample through the instrument, a detector and a data processor. The temperature of the injection port, column, and detector are controlled by thermosatted heaters.

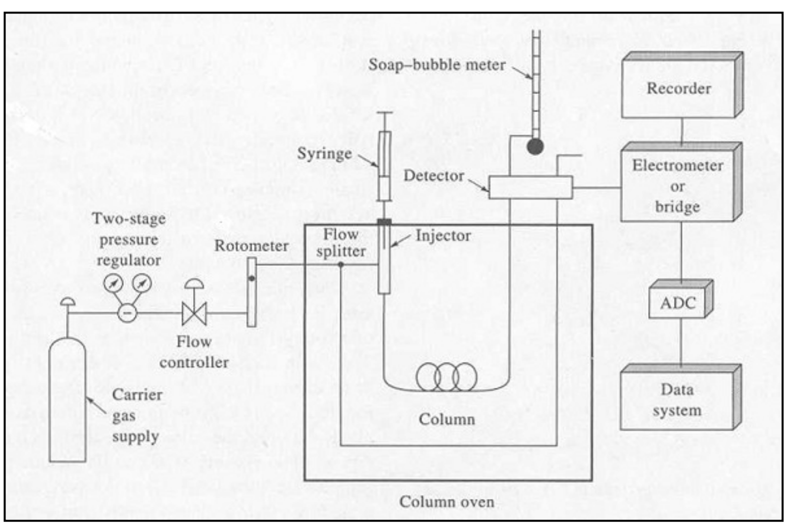


Figure 18: Schematic representation of a gas chromatograph

3.2 Project Activities

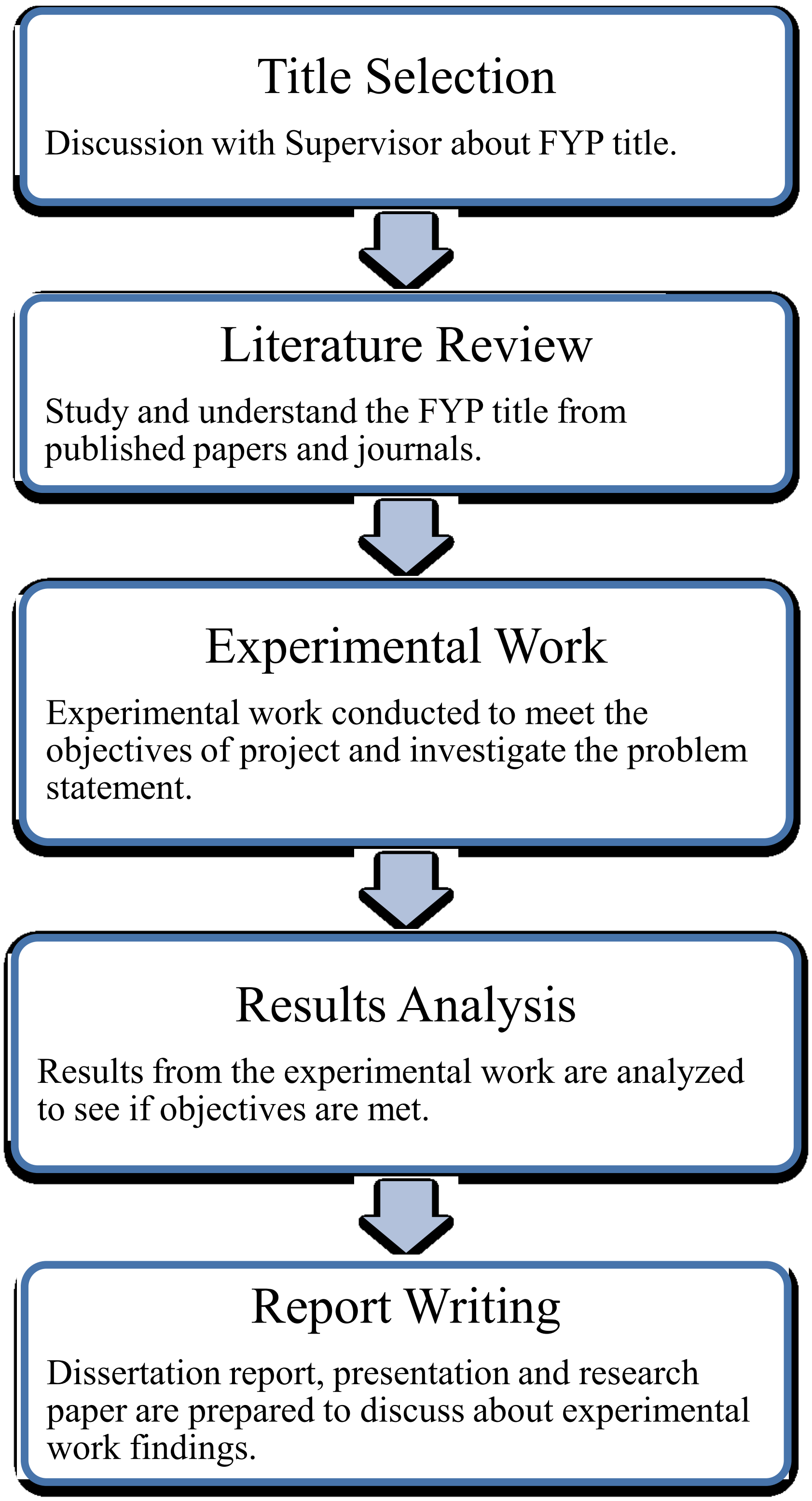


Figure 19: Project Activities Outline

3.3 Gantt Chart

Table 1: Gantt Chart

No.	Detail/Week	1	2	3	4	5	6	7	Mid – Semester Break	8	9	10	11	12	13	14	15	16
1	Project work commences	■	■	■	■	■	■	■		■								
2	Submission of Progress Report									■								
3	PRE-EDX combined with seminar/ Poster Exhibition/ Submission of Final Report (CD Softcopy & Softbound)												■					
4	SEDEX													■				
5	Delivery of Final Report to External Examiner/ Marking by External Examiner													■				
6	Final Oral Presentation															■		
7	Submission of hardbound copies																	■

3.4 Key Milestone

Table 2: Key Milestone

No	Action Item	Action By	Date	Note
1	**Briefing & update on students progress**	**Coordinator / Students / Supervisors**	**8 Feb**	**WEEK 3**
2	**Project work commences**	**Students**		**WEEK 1-8**
3.	**Submission of Progress Report**	**Students**	**16 March**	**WEEK 8**
4.	PRE-EDX combined with seminar/ Poster Exhibition/ **Submission of Final Report (CD Softcopy & Softbound)**	Students / Supervisor / Internal Examiner / Coordinator	**2 April**	**WEEK 11**
5.	EDX	Supervisors / FYP Committee	**9 April**	**WEEK 12**
6.	Delivery of Final Report to External Examiner / Marking by External Examiner	Students / Supervisors	**13 April**	**WEEK 12**
7.	Final Oral Presentation	FYP Committee / Coordinator	**23 April**	**WEEK 14**
8.	Submission of hardbound copies	Students	**11 May**	**WEEK 16**

3.5 Tools and Equipment

1.	Anton Paar Density Meter DMA 4500M
2.	VINCI Technologies Interfacial Tensometer (IFT 700)
3.	Shimadzu Gas Chromatograph GC-2010
4.	Carbon Dioxide Gas Tank
5.	Toluene & n - Heptane
6.	Heating Oven
7.	Degreaser
8.	Acetone

CHAPTER 4

RESULT AND DISCUSSION

4.1 Crude Oil Density Measurements

The Anton Paar Density Meter can only measure density up to a certain temperature. Thus to find the density at reservoir temperature, which is above the maximum temperature the equipment can achieve, extrapolation has to be made. The density measurement is taken at different temperatures and then extrapolated to the reservoir temperature to obtain the desired density for VIT experiment.

4.1.1 Dulang Oil Density

Table 3: Dulang Oil Density at Different Temperatures

Temp (°C)	Density (g/cm³)
40	0.8294
60	0.8191
70	0.8126
80	0.8061
85	0.8031
89	0.8005

Dulang reservoir temperature is at 102°C, thus extrapolation is required.

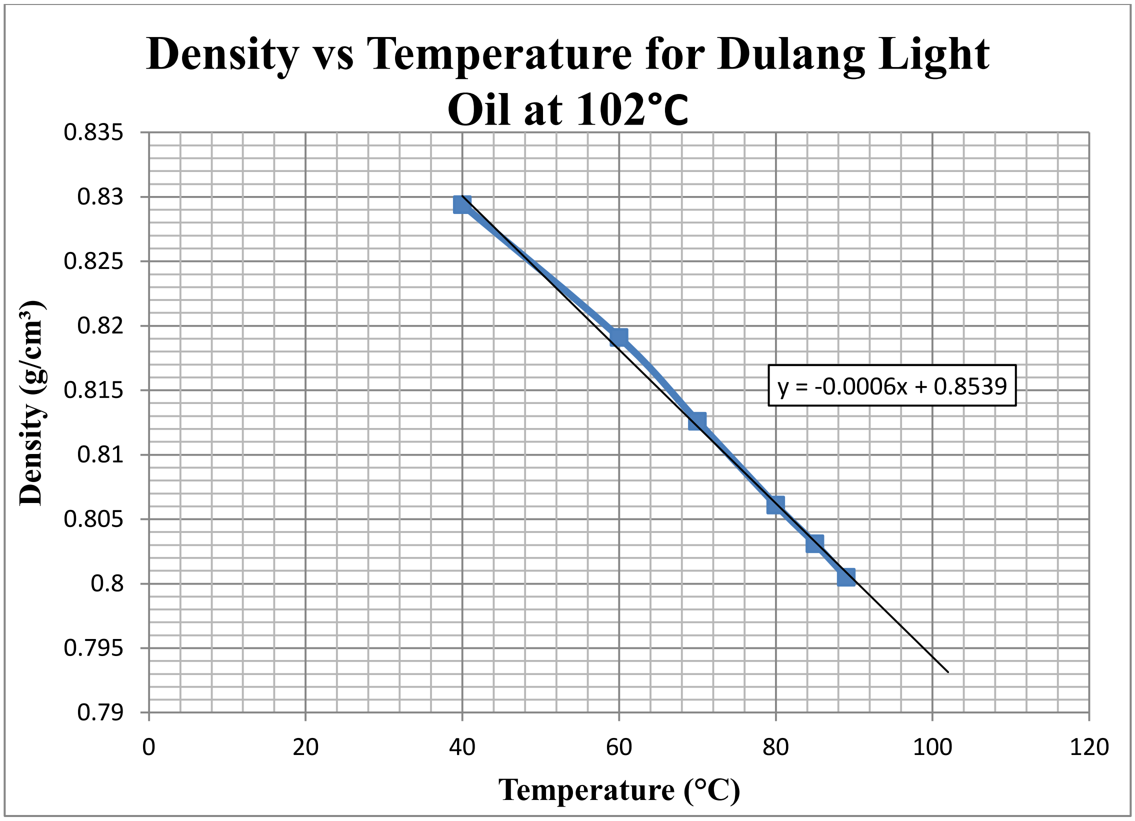


Figure 20: Dulang Density vs Temperature Graph

The density of Dulang crude oil at 102°C is 0.793 g/cm³.

To derive the API gravity, first we need to calculate the specific gravity using the density extrapolated earlier and applied into the formula below:

$$\text{SG oil} = \frac{\rho_{\text{oil}}}{\rho_{\text{H}_2\text{O}}}$$

SG for Dulang Oil @ 60°F = 0.84454

$$\text{API gravity} = \frac{141.5}{\text{SG}} - 131.5$$

API gravity for Dulang = 36.05°API

4.1.2 Angsi Oil Density

Table 4: Angsi Oil Density at Different Temperatures

Temp (°C)	Density (g/cm³)
60	0.7898
70	0.7832
80	0.7765
85	0.7731

Angsi reservoir temperature is 119°C, thus extrapolation is required.

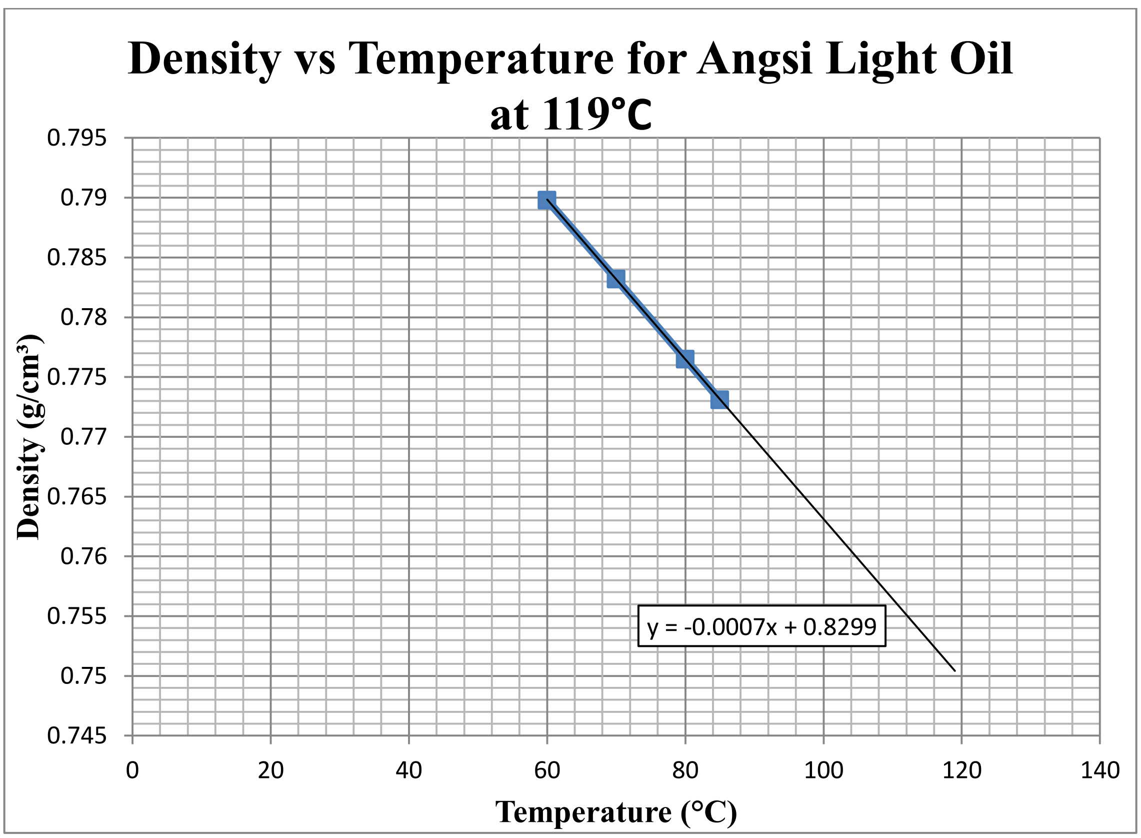


Figure 21: Angsi Density vs Temperature Graph

The Angsi crude oil density at 119°C is 0.7466 g/cm³.

To derive the API gravity, first we need to calculate the specific gravity using the density extrapolated earlier and applied into the formula below:

$$\text{SG oil} = \frac{\rho_{\text{oil}}}{\rho_{\text{H}_2\text{O}}}$$

SG for Angsi Oil @60°F = 0.8190

$$\text{API gravity} = \frac{141.5}{\text{SG}} - 131.5$$

API gravity for Angsi = 41.27°API

Figure 20 and **Figure 21** shows that extrapolation is done to obtain the density at reservoir temperature for Dulang and Angsi. Results show that Angsi has a lower density, and has a higher API gravity in comparison to Dulang. One of the difficulties faced in obtaining the measured density is during the injection of sample into the density meter. Air bubbles found in the U-tube will affect the density measurement, thus during injection, the sample has to be injected with a syringe at an appropriate injection rate.

4.2 IFT Measurements

The IFT for each crude oil sample was measured at reservoir temperature using the VINCI Technologies Interfacial Tensometer IFT 700. At least four experimental runs were conducted at four different pressures for each oil sample. The pressure versus interfacial tension for each run was plotted and shown at **Figure 22** for Dulang, and **Figure 23** for Angsi. Then the points are extrapolated to IFT equals zero, where this pressure is considered the MMP for this oil with CO_2. All experimental runs did not show complete miscibility between the oil and CO_2. Only good IFT measurements for different pressures are shown. At higher pressures, the pendant oil drop does not stay in the needle long, thus the IFT measurement gets tougher.

4.2.1 Dulang IFT

Table 5: Dulang IFT measurement with number of attempts

Pressure	800 psi		1200 psi		1600 psi	2000 psi
Attempt	1	2	1	2	1	1
Measured IFT over time	14.34	14.48	11.9	11.8	9	6.3
	14.37	14.58	11.93	11.88	9.04	6.64
	14.48	14.69	11.84	11.81	9.16	6.44
	14.49	14.38	11.83	11.78	9.05	6.21
	14.46	14.59	11.91	11.77	9.06	6.41
	14.42	14.52	11.94	11.77	8.97	6.42
	14.39	14.49	11.96	11.84	8.93	6.16
	14.51	14.81	11.93	11.75	9	7.29
	14.5	14.69	11.9	11.72	8.97	6.25
	14.42	14.43	11.92	11.73	9.02	6.72
	14.44	14.51	11.92	11.67	9.03	6.37
	14.47	14.57	11.91	11.75	9.01	6.51
	14.55	14.12	11.89	11.77	8.65	6.45
	14.46	14.24	11.9	11.79	9.03	6.48
	14.42	14.62	11.83	11.79	8.98	6.7
	14.48	14.39	11.95	11.74	9.01	6.49
	14.39	14.41	11.82	11.8	8.96	6.55
	14.38	14.33	11.86	11.79	8.93	6.61
	14.45	14.54	11.93	11.74	9.01	6.42
	14.35	14.44	11.86	11.74	8.99	5.95
	14.52	14.37	11.89	11.83	8.92	6.35
	14.44	14.69	11.87	11.78	8.98	6.74
	14.48	14.54	11.86	11.85	8.98	6.51
	14.44	14.26	11.93	11.75	8.89	6.5
	14.45	14.62	11.9	11.76	8.97	6.56
	14.47	14.65	11.88	11.74	8.98	6.53
	14.59	14.46	11.9	11.78	9.01	6.28
	14.5	14.42	11.83	11.74	9	6.38
	14.58	14.31	11.86	11.81	9.01	7.21
SUM	419.24	420.15	344.85	341.47	260.54	188.43
AVERAGE	14.45655	14.48793	11.89138	11.77483	8.984138	6.497586
AVERAGE 2	14.47224138		11.83310345			

Table 6: Dulang IFT at different pressures

Pressure (psia)	800	1200	1600	2000
IFT (N/m)	14.47224	11.8331	8.984138	6.497586

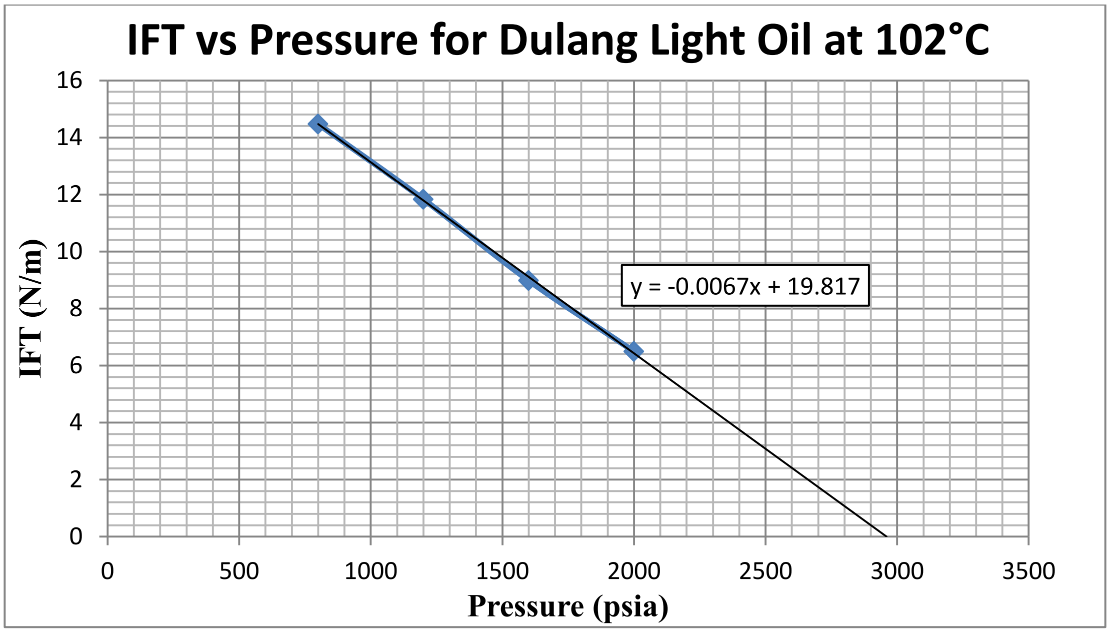


Figure 22: MMP Determination through plot of IFT versus Pressure for Dulang

For Dulang, the MMP extrapolated at zero IFT is 2957 psia.

4.2.2 Angsi IFT

Table 7: Angsi IFT measurement with number of attempts

Pressure	800 psi		1600 psi	2000 psi			2400 psi		
Attempt	1	2	1	1	2	3	1	2	3
	10.79	10.69	7.52	7.44	4.55	5.23	3.84	16.22	3.7
	10.85	10.7	9.92	5.56	4.62	5.46	3.68	3.49	3.67
	11.03	10.71	8.67	6.75	4.94	5.47	3.69	3.73	3.83
	10.73	10.86	8.81	6.6	4.84	4.91	3.73	3.6	3.83
	11.05	10.83	8.19	8.62	4.77	4.47	3.82	3.89	3.89
	11.12	11.19	8.2	4.38	5.1	4.75	3.64	3.59	3.7
	11.1	10.9	8.81	6.44	4.98	5.65	3.7	3.31	3.95
	11.14	11.12	10.32	8.21	4.92	4.93	3.47	3.51	3.43
	10.87	11.16	8	6.35	6.26	4.69	3.49	3.79	3.43
	11.27	10.97	8.84	6.2	5.65	4.63	3.8	3.6	3.61
	11.06	11.17	8.51	5.01	4.47	5.21	3.71	3.55	3.75
	11.15	11.28	7.7	4.97	5.07	5.11	3.73	3.67	3.66
Measured IFT over time	11.29	11.65	8.06	4.2	5.2	4.95	3.84	3.67	3.95
	11.89	11.32	8.81	5.53	4.88	5.02	3.74	3.69	3.74
	11.21	11.16	8.32	3.44	4.66	5.2	3.63	3.61	4.12
	11.14	11.38	7.53	3.92	5.17	4.97	3.65	3.71	4.05
	11.19	11.19	8.08	3.83	4.97	4.79	3.67	3.66	3.9
	11.25	11.08	7.45	4.3	5.37	5.48	3.77	3.85	3.46
	11.24	11.38	7.86	3.35	5.3	5.65	3.81	3.77	4.16
	11.18	11.32	7.83	4.38	5.64	5.33	3.84	3.8	3.7
	11.46	11.29	8.05	3.79	5.45	6.29	3.51	3.9	3.71
	11.61	11.11	9.16	3.79	5.46	5.72	3.47	3.83	3.85
	11.41	11.38	7.83		5.48	5.85	3.72	3.69	3.76
	11.52	11.14	8.91	4.03	5.41	5.4	3.85	3.75	3.99
	11.18	11.48	7.97	4.46	5.66	6.02	3.71	3.71	3.62
	11.48	11.47	7.18	7.11	5.78	6.05	3.64	3.67	3.74
	11.54	11.39	7.5	2.08	4.9	5.65	3.85	3.56	3.61
	11.23	11.38	6.97	4.11	5.45	5.74	3.93	3.84	3.98
	11.65	11.18	7.47	3.19					
SUM	325.63	323.88	209.07	139.96	144.95	148.62	103.93	99.44	105.79
AVERAGE	11.2286	11.168276	8.041154	5.183704	5.176786	5.307857	3.711786	3.682963	3.778214
AVERAGE 2	11.19844828			5.222782187			3.724320988		

Table 8: Angsi IFT at different pressures

Pressure (psia)	**800**	**1600**	**2000**	**2400**
IFT (N/m)	11.1984	8.223103	5.377901	3.81679

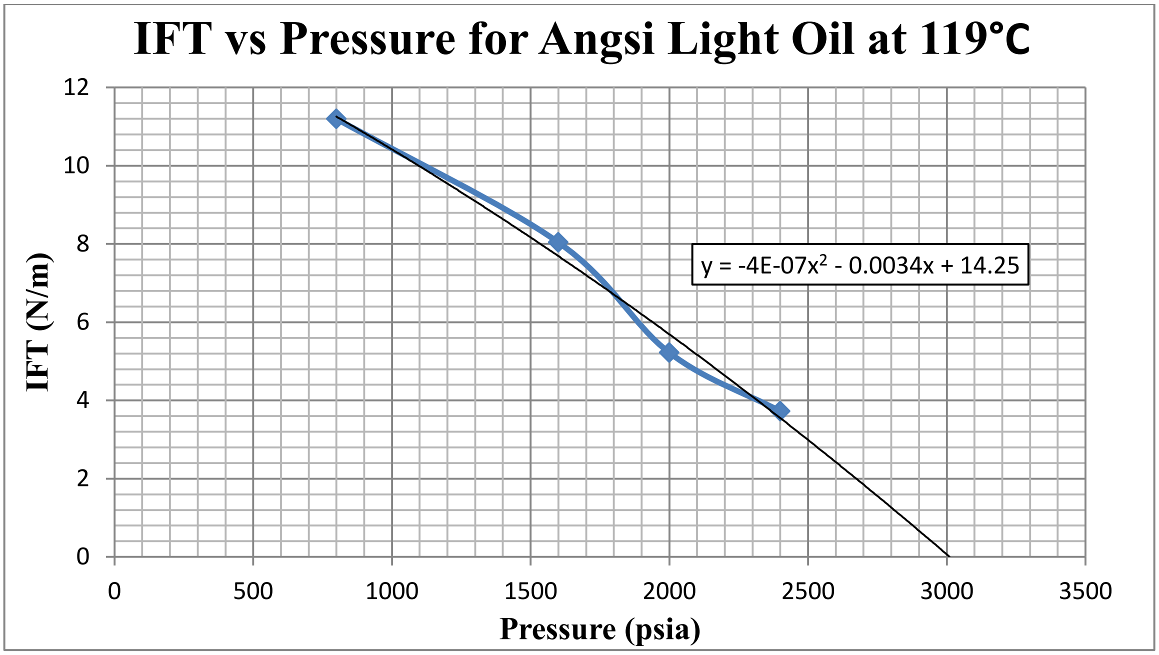


Figure 23: MMP Determination through plot of IFT versus Pressure for Angsi

For Angsi, the MMP extrapolated at zero IFT is 3077 psia.

The IFT measurement pressure for each sample was planned to be run at 800 psi, 1200 psi, 1600 psi and 2000 psi. However for Angsi sample, the IFT measurement at 1200 psi was not accurate due to the oil sticking to the needle, shown in **Figure 24**. Thus the experiment proceeded with the next set of pressure at 1600 psi, 2000 psi and 2400 psi. Higher pressure was not preferred due to the risks and difficulty of measuring the IFT as the drop will disappear rapidly at near miscibility pressure. From the VIT experiment, the MMP for Dulang is 2957 psia while Angsi is 3077 psia.

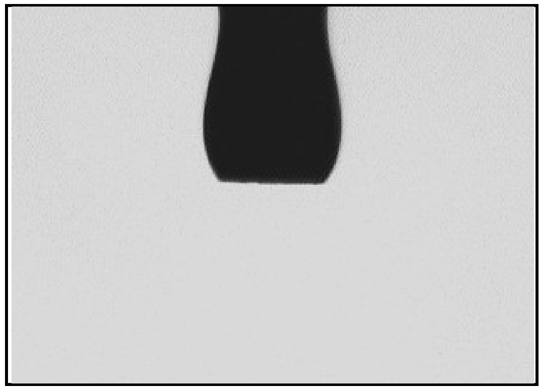


Figure 24: Oil sticking to the needle during run at 1200 psi pressure

4.2.3 Comparison between CO_2 MMP Determination Techniques

It is widely known that for CO_2 MMP Determination, the standard experimental method is to use the slim tube. However, the conventional slim tube test is too costly, time consuming and might not necessarily represent the true thermodynamic miscibility. Consequently, the Rising Bubble Apparatus was introduced to reduce the time taken to determine MMP. This method is also relatively cheaper compared to the slim tube. But, this test is subjective to the interpreter as miscibility is obtained from visual observations. Hence, the VIT technique was introduced and relies on the theory that at miscibility, the interfacial tension between the fluids must become zero due to the absence of an interface. The VIT technique has been applied effectively to optimize injection gas compositions for two miscible gas injection projects at the Rainbow Keg River and Canadian Terra Nova fields. (Ayirala & Rao, 2006)

4.3 Gas Chromatograph

Table 9: Dulang Oil Composition

COMPONENT	MOL %
CO2	20.743
N2	0.109
C1	15.062
C2	3.007
C3	2.710
iC4	1.032
nC4	0.854
iC5	0.415
nC5	0.283
C6	2.917
C7	2.833
C8	1.285
C9	2.470
C10	2.357
C11+	43.923
TOTAL	100.000

Table 10: Angsi Oil Composition

COMPONENT	MOL %
CO2	1.91
N2	0.15
C1	35.83
C2	7.24
C3	6.26
iC4	2.82
nC4	2.10
iC5	1.75
nC6	1.14
C6	2.96
C7	3.90
C8	5.69
C9	4.10
C10	3.70
C11	3.04
C12+	17.41
TOTAL	100.00

4.3.1 Effect of Oil Composition on CO_2 MMP

From the MMP determination using VIT method, we found that Angsi has a higher CO_2 MMP at 3077 psia while Dulang is at 2957 psia. From the gas chromatograph analysis in **Table 9** and **Table 10**, the oil composition of Dulang and Angsi is known. According to Dong (1999), the CO_2 MMP for a reservoir fluid can be affected by reservoir temperature, oil composition and purity of the CO_2 injected. By comparing **Table 9** and **Table 10**, we found that Angsi has a higher content of methane. According to Rathmell *et al.* (1971), the presence of methane affects the miscibility between CO_2 and the reservoir oil, thus increasing the MMP.

Besides that, the Dulang oil showed higher weight percent content of C_{5+} (56.4%) compared to Angsi (43.7%). Thus, Dulang has a slightly higher C_{5+} content which is considered important for CO_2 vaporizing mechanism. The greater the concentration of extractable hydrocarbons in the reservoir oil, the lower the MMP should be (Holm & Josendal, 1982). The light fractions are responsible for condensing CO_2 while the medium fractions are extracted by CO_2 (Aleidan, 2011). However, it is

4.3.2 Effect of Temperature on CO_2 MMP

The reservoir temperature for Dulang and Angsi are 102°C and 119°C respectively. Yellig & Metcalfe (1980) found that the reservoir temperature affects the CO_2 MMP significantly. The CO_2 MMP is increases at higher reservoir temperatures. By comparing between Angsi and Dulang, Angsi has a higher reservoir temperature thus from VIT, we confirmed that Angsi has a higher CO_2 MMP compared to Dulang.

4.4 CO_2 MMP Correlation

There are many CO_2 MMP published correlations that can be used to estimate the MMP for different oils. Published correlations uses several key input parameters such as reservoir temperature, molecular weight of C_{5+}, ratio of volatiles (C_1 and N_2) and intermediates (C_2-C_4, H_2S and CO_2). List of published empirical correlations used are shown in **APPENDIX A**.

Table 11: Summary of Published Correlation Results

Method	**Dulang (psia)**	**Error (%)**	**Angsi (psia)**	**Error (%)**
VIT	2957.00		3077.00	
Cronquist (1977)	2741.69	7.28%	3313.63	7.69%
Glaso xC2-C6>18%	2654.26	10.24%	3191.35	3.72%
Glaso xC2-C6<18%	4778.57	61.60%	5299.83	72.24%
Alston (1985) LO	3361.29	13.67%	4216.19	37.02%
Alston (1985) STO	3059.86	3.48%	3849.39	25.10%
Yellig and Metcalfe (1980)	2666.73	9.82%	3054.80	0.72%
Yuan (2005)	3596.91	21.64%	3993.40	29.78%

4.4.1 Comparison between CO_2 MMP Published Correlation

The results of the published correlations are summarized in **Table 11**. The table shows a comparison between the published correlations and the MMP measured from the VIT by calculation the resulted error. In Appendix A, a summary of the published correlations with the equations were included as a reference. From the table, it was noticed that (Cronquist, 1977) correlation gave the best predicted MMP for both oil sample. The correlation takes into account three parameters: reservoir temperature, molecular weight of C_{5+} and mole % of C_1. This correlation covers a wide range of oil gravity, from 23.7 to 44° API, and reservoir temperature ranging from 21.67 to 120°C. (Yellig & Metcalfe, 1980) correlation gave the best prediction for Angsi, however this correlation only depends on temperature. It is also evident that it is more accurate to predict the MMP for dead oil, as shown in the (Alston, 1985) live oil (LO) and stock tank oil (STO) correlation. Glaso's correlation gave better prediction when C_2 – C_6 contents were included.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

Rapid CO_2 MMP determination via VIT technique for Angsi and Dulang has been conducted in different pressure and reservoir temperature. The minimum miscibility pressure (MMP) for Dulang and Angsi are 2957 psia and 3077 psia respectively. Thus, immiscible CO_2 flooding is only possible as the initial reservoir pressure for Dulang and Angsi are 1800 psig and 2515 psig. The MMP obtained from VIT technique have been compared with several published correlations, and (Cronquist, 1977) correlation gave the best predicted MMP for both reservoir oil. Comparison of the published correlations was discussed, with each correlation requiring specified input parameters. MMP predictions for dead oil was found to be more accurate compared to live oil MMP predictions. On the other hand, these correlations have shown the importance of parameters that affects the CO_2 MMP for instance, the reservoir temperature, the oil composition, presence of volatile components and intermediate fractions.

The VIT technique was also justified in comparison to other CO_2 MMP determination methods like slim tube and rising bubble apparatus. VIT technique was opted due to time constraint and it is hoped that with more research time, more CO_2 MMP samples could be further investigated and compared.

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APPENDICES

Appendix A

Empirical Correlations

Cronquist (1977):

$$MMP_{pure} = 15.988T^{0.744206+0.0011038M_{C5+}+0.0015279C_1}$$

T = reservoir temperature at °F, M_{C5+} = molecular weight of C_{5+}, C_1 = mole % of C_1

Glaso (1985):

$$MMP(CO_2) = \begin{cases} 810.0 - 3.404M_{C_{7+}} + 1.7\times10^{-9} M_{C_{7+}}^{3.73} \exp(786.8M_{C_{7+}}^{-1.058})T & , x_{C_2-C_6} > 18\% \\ 2947.9 - 3.404M_{C_{7+}} + 1.7\times10^{-9} M_{C_{7+}}^{3.73} \exp(786.8M_{C_{7+}}^{-1.058})T - 121.2x_{C_2-C_6} & , x_{C_2-C_6} < 18\% \end{cases}$$

T = reservoir temperature at °F, M_{C7+} = molecular weight of C_{7+}, x_{C2-C6} = mole % of $C_2 - C_6$ intermediate content

Alston (1985)

$$p_{CO_2-STO} = 8.78\times10^{-4}(T_R)^{1.06}(M_{C5+})^{1.78}$$

$$P_{CO2-LO} = 8.78 \text{ x } 10^{-4} (T_R)^{1.06} (M_{C5+})^{1.78} . \left(\frac{x_{vol}}{x_{int}}\right)$$

T_R = reservoir temperature at °F, M_{C5+} = molecular weight of C_{5+},

x_{vol} = mole fraction of volatile (C_1 and N_2)

x_{int} = mole fraction of intermediate ($C_2 - C_4$, CO_2 and H_2S)

Yellig and Metcalfe (1980):

$$MMP_{pure} = 1833.717 + 2.2518055T + 0.01800674T^2 - \frac{103949.93}{T}$$

T = reservoir temperature at °F

Yuan (2005):

$$MMP(CO_2) = -1463.4 + 6.612M_{C_{7+}} - 44.979x_{C_2-C_6} + \left(2.139 + 0.11667M_{C_{7+}} + 8166.1\frac{x_{C_2-C_6}}{M^2_{C_{7+}}}\right)T$$
$$+ \left(-0.12258 + 0.0012283M_{C_{7+}} - 4.0152\times10^{-6}M^2_{C_{7+}} - 9.2577\times10^{-4}x_{C_2-C_6}\right)T^2$$

M_{C7+} = molecular weight of C_{7+}, x_{C2-C6} = mole percent of $C_2 - C_6$,

T = reservoir temperature at °F