A RELIABLE AND RAPID TECHNIQUE FOR THE LABORATORY DETERMINATION OF THE MINIMUM MISCIBILITY PRESSURE FOR CO_2 – LIGHT CRUDE OIL SYSTEMS USING THE SLIM TUBE METHOD.

A Thesis

by

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ABSTRACT

Minimum miscibility pressure is a crucial parameter for the design of any gas injection project. The techniques used for MMP determination can be categorized into either experimental or computational methods. Computational methods are convenient and fast. However, they do not lead to accurate values and are mainly used when only an approximation is needed. Experimentally, the most common ways of determining MMP are the slim tube method, the rising bubble method, and the method of vanishing interfacial tension. The vanishing interfacial tension and the rising bubble methods do not entirely address the multi-contact mechanisms. On the other hand, the slim tube experiment is considered to be the most accurate way to determine the MMP since it simulates the 1-D displacement of the reservoir crude oil by the injected CO_2 fully accounting for the thermodynamic phenomena taking place in the CO_2-oil system inside a sand packed coil.

I present a technique that enables the determination of the minimum miscibility pressure of a CO_2 – light crude oil system at low reservoir temperatures using a short 20 ft slim tube in less than two weeks, about a third of what it normally takes using the conventional 80 ft slim tube.

MMP is a crucial parameter in designing a CO_2 enhanced oil recovery project and its value needs to be known with a degree of accuracy that cannot be provided by the use of equations of state or correlations, and therefore, needs to be determined experimentally.

The slim tube technique is recognized to be the most accurate experimental method for determining the MMP. However, its use has not been favored because it is time-consuming.

The MMP for various CO_2 – crude oil systems were determined from the North Burbank Unit and the Oklahoma/Texas Panhandle. The reduction in the length of the slim tube from 80 ft to 20 ft resulted in a decrease in the total time of the experiment. The validity of the technique was proven by performing recovery factor measurements using a conventional 80 ft long slim tube. The MMP values obtained are valid when the length of the slim tube is sufficient to host the mixing zone and the velocity of the displacement is slow enough to enable the transverse dispersion to eliminate viscous fingering. In the case of light oil at low temperatures, the use of the 20 ft slim tube is justified as the length of the mixing zone is shorter. The results are also supported by the use of numerical simulation.

The reduction in the time required for slim tube experiment results in a fast, economical and accurate technique for the determination of MMP in CO_2 – light crude oil systems. Taking into account that CO_2 flooding is the most applied EOR technique in the US and that it is mainly applied to light oil reservoirs, this work can be of great impact by providing a rapid and reliable method for determining the MMP for designing a CO_2 enhanced oil recovery project.

DEDICATION

"Your time is limited, don't waste it living someone else's life. Don't be trapped by dogma, which is living the result of other people's thinking. Don't let the noise of other's opinion drowned your own inner voice. And most important, have the courage to follow your heart and intuition, they somehow already know what you truly want to become. Everything else is secondary." – ***Steve Jobs***

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It is with my sincere gratitude that I dedicate this thesis work to:

My father, my mother, my brother and my sister

For all your support and letting me find my own path

For all the times I have been unreachable

For all the distance between us

For all the important moments I have missed

For all your love and patience

For all you have sacrificed for me

Thank you for providing me the best education possible.

Thank you for always being patient, understanding and loving

I would not be at this stage without all of your combined love and support

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This work was supervised by a thesis committee consisting of Dr. David Schechter, Chair of the Committee, Dr. Maria Barrufet and Dr. Hadi Nasrabadi of the Department of Petroleum Engineering.

The Numerical Validation and The Quality Control sections presented in Chapter IV were completed in collaboration with Francisco Tovar, Ph.D candidate in the Department of Petroleum Engineering. All other work for the thesis was completed by the student, under the advisement of Dr. David Schechter of the Department of Petroleum Engineering.

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NOMENCLATURE

EOR	Enhanced Oil Recovery
EOS	Equation of State
MMP	Minimum Miscibility Pressure, psig
NBU	North Burbank Unit
OOIP	Oil Originally in Place
FCM	First Contact Miscibility
MCM	Multi-Contact Miscibility
VGD	Vaporizing Gas Drive
CV	Condensing/Vaporizing Gas Drive
MME	Minimum Miscibility Enrichment
PV	Pore Volumes
CCE	Constant Composition Expansion
Sor	Residual Oil Saturation
E	Overall Displacement Efficiency
E_V	Macroscopic Displacement Efficiency
E_D	Microscopic Displacement Efficiency
IFT	Interfacial Tension
VIT	Vanishing Interfacial Tension
PVT	Pressure Volume Temperature

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CHAPTER I

INTRODUCTION AND LITERATURE REVIEW

General Background on Enhanced Oil Recovery

Oil recovery is separated into three different stages: primary, secondary, and tertiary oil recovery. Primary recovery uses displacement energies naturally existing in the reservoir such as solution gas drive, gas cap drive, natural water drive, gravity drainage and fluid and rock expansions. Primary recovery leads to only around 35% of the oil originally in place (OOIP) in light to medium reservoir oils (Lake 2010). In secondary recovery, the natural energy in the reservoir is augmented by water injection or immiscible gas injection to sustain reservoir pressure and displace more oil towards producing wells. Secondary recovery usually leads to an additional 20% recovery of the OOIP. Hence, half of OOIP is left unproduced in the reservoir due to poor sweep efficiency, conning problems, and capillary forces and only a fraction of the oil contacted by the water displaced.

Oil overall displacement efficiency (E) is governed by the pore scale and the volumetric scale displacements.

$$E = E_D * E_V \qquad (1)$$

Microscopic displacement efficiency (E_D) represents the amount of residual oil saturation (Sor) in areas contacted by displacing fluids in pours media at the microscopic level.

$$E_D = \frac{\text{Volume of Oil Displaced}}{\text{Volume of Oil Contacted by the Injected Fluid}} \tag{2}$$

Macroscopic (Volumetric) displacement efficiency (E_V) is the product of areal sweep efficiency (Ea) and vertical sweep efficiency (Ev). E_V represents the fraction of the volume of oil contacted by the injected fluid over the volume of oil originally in place. The volumetric displacement efficiency is governed by the heterogeneity of the reservoir as well as the mobility ratio between the injected fluid and the reservoir oil.

$$E_V = Ea * Ev = \frac{\text{Volume of Oil Contacted by the Injected Fluid}}{\text{Volume of Oil Originally in Place}} \tag{3}$$

Tertiary/Enhanced oil recovery (EOR) is applied using external sources of energy and/or materials to recover oil that cannot be produced, economically by conventional means (Thomas 2007). EOR can be achieved through a reduction in interfacial tension (IFT), a reduction in oil viscosity, oil swelling or wettability alteration and can be divided into three main categories: thermal, chemical or miscible gas EOR processes.

Thermal EOR Processes

Thermal methods reduce residual oil saturation (Sor) by thermal driving mechanisms such as in-situ combustion, steam injection, steam assisted gravity drainage (SAGD), hot water drive and electrical heating that lead to a reduction in the oil viscosity and thus result in a more favorable mobility ratio. In in-situ combustion, hot air and hot water are injected to support movement in the heated front towards the wellbore. Injecting steam downhole is also used but in a cyclic pattern. Eventually, steam condenses into hot water leading to a better mobility ratio and better sweep efficiency. Environmental activists oppose thermal recovery methods due to high combustion temperatures in the power plant, producing large air pollutant emissions.

Chemical EOR Processes

Chemical flooding consists of injecting a water based solution with surfactants, polymers, microbes, or a combination of two or more of these chemicals. Surfactant flooding is used to recover the oil confined by the capillary pressure, reducing the interfacial tension between water and oil. The surfactant prevents the free oil from being trapped. Polymers, on the other, hand are added to either water or surfactant flood to enhance the mobility ratio in the flooding process. Polymers decrease the relative permeability to water but increase its viscosity which improves the microscopic sweep efficiency. Polymers are also used in mobility control for the permeability reduction in high permeability zones which results in a better vertical sweep efficiency. Finally, microbial flooding consists of injecting bacteria and nutrients that make the surface rock more slippery to oil, altering the wettability, increase the water density, and reduce the oil density.

Miscible EOR Processes

The miscible displacement flooding consists of injecting CO_2, inert gas (N_2), or hydrocarbon gases at a pressure where the injected gas and the in situ crude oil are miscible. Nitrogen gas is cheaper than CO_2 and is used for pressure maintenance and miscible displacement fluid in high pressure/light oil reservoirs. CO_2 miscibility in oil leads to a lower viscosity and density of the mixture allowing for better sweep efficiency.

For decades, carbon dioxide has been a successful enhanced oil recovery (EOR) technique, and its use has consistently grown in recent years becoming the most used EOR method in the US. In 2012 alone, the Oil & Gas Journal (2012) reported that CO_2 miscible flooding accounted for 41% of the total US EOR production, the most out of all types of EOR. Operators favor CO_2 injection because it can significantly reduce oil viscosity, swells the oil due to its high solubility in hydrocarbons, and can decrease override effects (Tovar et al. 2014). From an environmental standpoint, injection of CO_2 into oil reservoirs is also appealing to reduce global warming effects through CO_2 sequestration (Denney 2010; Izgec et al. 2005). Furthermore, for any gas injection EOR process, reaching miscibility greatly increases ultimate recovery, and CO_2 requires a lower pressure to achieve miscibility with crude oil compared to nitrogen or hydrocarbon gases.

Miscibility Development and Minimum Miscibility Pressure in Gas Flooding

Miscibility can be achieved in one of two way: first contact (FCM) or multi-contact miscibility (MCM). First contact miscibility is achieved if the oil and the gas form a single phase when brought into contact at any ratio. First contact miscibility is rarely achieved between oil and gas; miscibility is usually achieved by mass transfer and components exchange between the injected gas and the in situ oil through flow in the porous medium. This miscibility is named multi-contact miscibility. MCM can be achieved either by a condensing drive displacement, a vaporizing gas drive (VGD) displacement, condensing/vaporizing-gas (CV) drive displacement or CO_2 displacement.

In the vaporizing gas drive displacement, the gas contacts the oil and the light to intermediate hydrocarbon molecules transfer from the oil to the gas by vaporizing into the injected gas and enriching it. After several contacts between the oil and the enriched gas, when the gas reaches the minimum miscibility enrichment (MME), multiple contact miscibility is reached. In general, the vaporizing gas drive occurs when injecting a lean gas such as dry gas, nitrogen.

On the other hand, the condensing drive displacement is the result of the in situ transfer and condensation of intermediate hydrocarbon molecules from the injected rich gas; mostly methane through butane; into the relatively heavy reservoir oil leading to a lighter oil. The injected gas, in this case, has to contain a major fraction of intermediate components. After constant contact, MME is reached and the enriched lighter oil then achieves a multi contact miscibility with the injected gas.

The condensing/vaporizing-gas drive mechanism consists of both condensation and vaporization gas drives. In this type of displacement, neither the oil nor the gas becomes rich enough to reach miscibility through vaporization or condensation alone. Multi-contact miscibility is reached by both the condensation of intermediate hydrocarbon molecules from the injected gas and the vaporization of mid-range hydrocarbon molecules from the reservoir oil (Zick 1986; Stalkup 1987).

Minimum miscibility pressure (MMP) is one of the most crucial parameters in any gas injection project. The MMP is the lowest pressure at which the injected gas and the reservoir oil become miscible through a multi-contact process at the reservoir temperature (Elsharkawy *et al.*, 1992). In order to optimize recovery in any gas injection project, it is essential to operate near above this critical value. Hence, the measurement of MMP is key in the design of any miscible CO_2 flooding project.

CHAPTER II

PROBLEM DESCRIPTION AND RESEARCH OBJECTIVES*

Experimental Methods for Estimating the Minimum Miscibility Pressure

The MMP can be determined using several methods that are categorized as experimental or computational. In the laboratory, the slim tube experiment, the rising bubble method, the mixing cell method and the method of vanishing interfacial tension are the most common. However, the vanishing interfacial tension and the rising bubble methods do not entirely address the multi-contact mechanisms (Teklu et al. 2012), and the mixing cell method is unable to measure the MMP for a condensing/vaporizing drive (Bryant and Monger 1988). Therefore, the slim tube experiment is considered to be the most accurate way to determine the MMP since it simulates the 1-D displacement of the reservoir crude oil by the injected CO_2 fully accounting for the thermodynamic phenomena taking place in the CO_2-oil system inside a sand packed coil (Ekundayo and Ghedan 2013).

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*Part of the problem description and research objectives sections presented in this chapter have been reprinted from “Fast-Slim Tube: A Reliable and Rapid Technique for the Laboratory Determination of MMP in CO2 - Light Crude Oil Systems” by Imad A. Adel, Francisco D. Tovar, and David S. Schechter. SPE Paper 179673. Copyright 2016 by the Society of Petroleum Engineers. Reproduced with permission of SPE. Further reproduction prohibited without permission.

Vanishing Interfacial Tension Method

The vanishing interfacial tension (VIT) experiment was first introduced for MMP determination by Rao (1997). This method consists of measuring the interfacial tension between the injected gas and the crude oil at different working pressures and a fixed reservoir temperature. The experiment is conducted by injecting a crude oil drop (~ 10% of the cell volume) through a capillary tube into a high temperature, high-pressure cell filled with injection gas (Rao and Lee 2002). The contact angle and shape of the hanging oil drop, as well as the densities of the oil and the gas, are used to determine the interfacial tension (IFT). The pressure is then increased by adding more gas into the cell, and the measurement of the IFT is repeated. The MMP is then determined by extrapolating the IFT vs. pressure graph to zero.

This method, however, is limited when using multi-component mixtures. Orr and Jensen (2007) argue that “mixture compositions that are linear combinations of the initial oil and injection gas are quite different from the critical mixture that forms at the MMP in a gas–oil displacement in a porous medium” Orr and Jensen also claimed that the mixtures formed in the VIT cells for multi-component oil mixtures do not lead to reliable MMP values.

Rising Bubble Method

The Rising bubble was first introduced as a quick substitute for the slim tube experiment by Christiansen and Haines (1987). The method consists of eight inches long transparent glass column kept at the desired pressure and temperature. Gas is injected at the bottom of the oil column through a needle. The formed gas bubble then rises through the column and the shape of the rising bubble is used to evaluate the MMP criteria (Christiansen and Haines 1987).

This method tries to reproduce the forward contact of oil and gas in the reservoir. The gas makes contact with crude oil as it rises through the column becoming richer and richer as it approaches the top. The rising bubble method can determine the MMP for a vaporizing gas drive as miscibility will develop at the front of the advancing gas. However, it cannot accurately determine the MMP for a condensing drive or condensing/vaporing gas drive (Zhou and Orr 1998).

This method is faster and cheaper when compared to the slim tube method, however, it faces a major problem in accuracy when miscibility is achieved by a condensing drive mechanism.

Mixing Cell Method

The mixing cell method is used to determine the MMP by analyzing the phase behavior of the injected gas and the crude oil mixture (Bryant and Monger 1988). In a pressure volume temperature (PVT) visual cell, the oil and gas are mixed at defined ratios and brought to equilibrium. The gas and crude oil are repeatedly mixed in the PVT cell in a forward or backward contact.

In a forward contact, when equilibrium is reached, the enriched gas is preserved, and the oil in the PVT cell is replaced with new fresh oil that is brought to contact with the gas from the previous step. In a backward contact, it is the oil that is preserved and the gas is replaced with fresh gas. The procedure is repeated at different pressures until the repeated mixtures lead to a single phase that can be seen through the glass on the PVT cell.

The main disadvantage of the mixing cell method is its inability to determine MMP for a condensing/vaporizing gas drive. This method is fast and cheap and can be a good alternative to the slim tube method when the miscibility drive mechanism is known in advance to be either a condensing or a vaporizing gas drive.

Slim-Tube Method

The slim tube experiment is the most established and accepted experimental method to determine the MMP. The slim tube is a long sand packed stainless steel coiled column. The slim tube’s length ranges from 5 to 120 ft and its diameter varies from 0.12 to 0.63 in, regularly being 0.25 inches (Elsharkawy et al. 1992; Orr et al. 1982). The slim tube experiment is very close to a one-dimensional displacement due to the large length to diameter ratio focusing only on the effect of displacement efficiency and phase behavior and avoiding negative conditions such as gravity effect, unfavorable viscosity ratios, and fingering.

Generally speaking, the slim tube experiment procedure consists of injecting CO_2 to contact and displace the reservoir crude oil that saturates a grain packed coil at stabilized conditions of pressure and temperature that resemble those in the reservoir. Elsharkawy *et al.* (1992) define the MMP value as the intersection between the two lines defined by the recovery factor as a function of pressure, both under miscible and immiscible conditions. On the other hand, Holm and Josendal (1974) standards for the MMP are 80% recovery at gas breakthrough. All recovery factors are measured at 1.2 PV of injected gas.

The slim tube method has a significant drawback as the experiment requires considerable time to run and is very expensive. Nonetheless, despite these drawbacks, the slim tube experiment is the most reliable method of determining MMP in industry. It is the only method that simulates the 1-D displacement of the reservoir crude oil by the injected CO_2 fully accounting for the thermodynamic phenomena taking place in the CO_2-oil system inside a sand packed coil (Ekundayo and Ghedan 2013).

Problem Description

To standardize the slim tube experiments, long coil lengths and low injection rates have been recommended in literature to avoid composition variations, effects of fingering and to ensure a stable thermodynamic front (Elsharkawy *et al.*, 1992). Flock and Nouar (1984) reviewed several studies addressing the effect of the coil length on the MMP.

Based on their experimental work, the authors recommended the use of a coil with a length more than of 40 ft to achieve a stable displacement. The coils commonly used are therefore of 60 to 80 ft in length. As a consequence of this, the determination of MMP by the slim tubing technique usually takes at least five weeks for one oil sample when the recovery factor is measured at only four pressures. It will be argued later that measuring only four points does not allow the ability to check for the consistency of the experiments. This long time translates into high costs that have resulted in a reduction of the use of the slim tubing technique despite its accuracy.

Tovar *et al.* (2015) measured the MMP for a CO_2 – North Burbank Unit (NBU) dead crude oil system using the slim tube technique with a 20 ft short packed coil instead of the conventional 80 ft packed coil. This alteration enabled a substantial decrease in the experimental time. The MMP was experimentally determined to be 1563 psig, which was lower than the 1687 psig value found in the test reports performed by a commercial laboratory using a 40 ft packed column. The results are not expected to be identical for three reasons. The first one is that the commercial laboratory only performed three recovery measurements**,** at 1400 psig, 1600 psig and 1900 psig, assuming a 100%

recovery at 2400 psig. The pressure at which the 100% recovery factor is assumed, can shift the MMP by a few psig. The second one is that the short coil experiment used dead oil, whereas the long one used recombined oil. Finally, the third one is that the samples were collected from the same reservoir but at different times.

The use of dead oil was justified because the NBU produces at a very low gas to oil ratio (Tovar *et al.*, 2015). The results of Tovar *et al*. (2015) were consistent with core flooding experiments. Due to the significant reduction in time, the fast version of the slim tube technique using a 20 ft coil for similar light crude oil samples was adopted.

Research Objectives

The primary objective of this thesis is to show that the slim tube technique for CO_2 – light crude oil systems can be made more affordable with considerably reducing the experimental time, without significant loss in the accuracy of the conventional method. I argue that a 20 ft slim tube coil is enough to reach a stable thermodynamic front**,** and leads to a reliable MMP value. The results obtained using the fast-slim tube method are validated by comparing them to the ones obtained using a longer 80 ft coil with similar diameter and packing, and by the use of numerical simulation.

CHAPTER III

EXPERIMENTAL PROCEDURE*

A schematic of the experimental apparatus is represented in **Figure 1**. A short 20 ft coil that allows a rapid determination of MMP values was used for the experiments. The coil is made of stainless steel with an inside diameter of 0.25 in, a thickness of 0.063 in, and is packed with 100 – 150 mesh Ottawa sand.

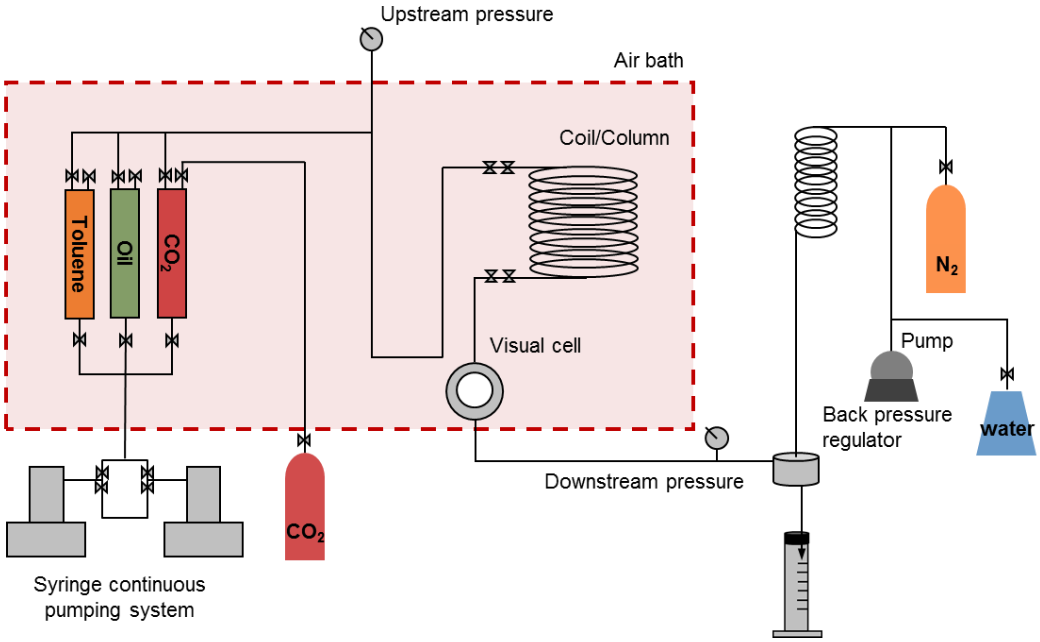

Figure 1. Schematic of the set up for the MMP determination apparatus using the slim tubing technique (Tovar *et al.*, 2015)

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*Parts of the procedures presented in this chapter have been reprinted from "Fast-Slim Tube: A Reliable and Rapid Technique for the Laboratory Determination of MMP in CO2 - Light Crude Oil Systems" by Imad A. Adel, Francisco D. Tovar, and David S. Schechter. SPE Paper 179673. Copyright 2016 by the Society of Petroleum Engineers. Reproduced with permission of SPE. Further reproduction prohibited without permission.

Slim Tube Column Pore Volume Measurement

The pore volumes of the different slim tube columns used were calculated in order to perform recovery measurements and to determine the amount of fluid to be injected. The slim tube is proved to be clean and dry by weighing the slim tube and comparing it to its original weight. The dead volume at the inlet and outlet of the slim tube is then calculated by filling the valves and the associated pipes with water and weighing them. The slim tube is submitted to vacuum for two to six hours depending on the length of the column. A vacuum gauge is placed in the opposite side to where the vacuum pump is connected to verify that the vacuum was successfully applied and held.

Before connecting the toluene accumulator to the slim tube, the pipes connecting the accumulator to the slim tube are fully saturated with toluene. The pump is then set to a constant pressure of 500 psi and the volume in the pump is recorded. The valve that connects the accumulator to the slim tube is then opened, and the new volume in the pump is recorded after it stabilizes. The difference is the pore volume. The compressibility of toluene is used to make the pore volume calculation more accurate by increasing the pressure in the pump to 1000, 2000 and 3000 psig and recording the variation in the pore volume.

Slim tube Column Clean Up

To guarantee the porous media is clean and dry before each run, 2 pore volumes (PV) of toluene are injected at a rate of 0.2 cm^3/min followed by 2 PV of supercritical CO_2 at a rate of 0.06 cm^3/min. The slim tube coil is then weighted and compared to its original weight to verify that it is fully clean and dry. If the weight difference is more than one gram the cleaning and drying procedure is repeated.

Recovery Measurements

The dry coil is submitted to vacuum conditions. During that stage**,** the absence of leaks is verified with the assistance of a vacuum gauge. Then the temperature of the air bath is set to reservoir temperature. Later, crude oil is injected at a rate of 1 cm^3/min while gradually increasing the back pressure until the desired test pressure is reached**.**

After the stabilization of pressure and temperature, 2 PV of the dead crude oil are injected at a rate of 0.2 cm^3/min to guarantee the system is fully saturated. Following this, 1.2 PV of CO_2 is pumped at a rate of 0.06 cm^3/min to displace the oil. The effluents are measured throughout the experiment to calculate the recovery factor at each of the different displacement pressures.

MMP Determination

The resulting recovery factors are plotted as a function of pressure, and two regions are identified, the immiscible region, where the recovery factor is a strong function of pressure, and the miscible region, where the recovery factor is a weak function of pressure. The MMP is the intersection point between these two regions (**Figure 2**).

To check for consistency, six runs are made for each MMP determination, three in the immiscible region and three in the miscible region. For all most cases, the pressures for the displacement were 500, 750, 1000, 1800, 2500 and 3500 psig.

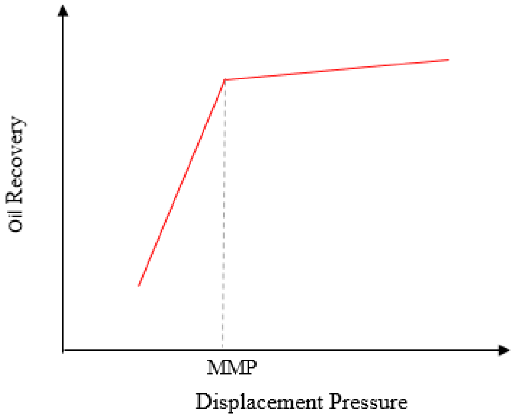

Figure 2. Schematic of oil recovery as a function of the displacement pressure

CHAPTER IV

RESULTS AND DISCUSSIONS*

MMP Determination Results Using a 20 ft Coil

Table 1 represents the results for six runs at 3500, 2500, 1800, 1000, 750 and 500 psig, for light crude oil samples 1 – 5 at their respective reservoir temperatures of 122 °F. The recovery factors measured at the three lower pressures of 500, 750 and 1000 psig show a strong dependency on pressure, which indicates that miscibility has not yet been reached. On the other hand, at the higher pressure data points of 1800, 2500 and 3500 psig, the recovery factors are not strongly affected by the increase of pressure, indicating that miscible behavior has been reached. The identification of these two regions is the main criteria adopted for the determination of the MMP from our experimental data.

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Table 1. Recovery factor as a function of pressure for light oil samples 1 – 5

Test Pressure (psig)	Recovery factor (% of OOIP) Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
3500	98.38	92.50	94.98	93.78	91.34
2500	92.38	86.50	91.98	89.78	87.16
1800	88.18	82.30	89.88	86.98	83.51
1000	62.62	56.42	66.26	56.00	54.80
750	40.12	38.92	51.26	36.00	41.75
500	17.62	21.42	36.26	16.00	29.23
API °	42.91	38.31	34.90	38.8	37
Res Temp (°F)	122	122	122	122	122
MMP (psig)	**1247**	**1330**	**1373**	**1366**	**1545**

Table 2 represents the results for six runs for sample 6 at its reservoir temperature of 152 °F and sample 5 at a temperature of 165°F. Sample 6 is relatively heavier than the other sample with an API of 29.82° and is categorized as a medium oil. It has been pointed out in literature raising the temperature tends to delay extraction, probably due to increased surface energy, which prevents the hydrocarbon and CO2 molecules from coherence (Wang and Knight, 1982), leading to a higher MMP. The MMP for sample 5 was redetermined at a higher temperature of 165°F to explore the effect of temperature on the MMP and the effect of reducing the coil length in CO2 – crude oil systems presenting higher MMP values. The recovery factors indicated early that the MMP for theses samples are higher than the MMPs for samples 1 – 5 at a temperature of 122°F.

Hence, the displacement pressures had to be accordingly adjusted to stay out of the transition zone and keep within the linear trends of the miscible and immiscible regions.

The increase in the experimental temperature from 122 °F to 165 °F let to an increase of 2200 psig in the minimum miscibility for the CO2 – sample 5 system. These results show the great impact temperature has on the MMP value and are in agreement with literature and our numerical simulation results.

Table 2. Recovery factor as a function of pressure for samples 5 and 6

Test Pressure (psig)	**Recovery factor (% of OOIP) Sample 6**
5500	86.5
4000	82.46
2500	77.24
1500	42.30
1300	27.66
1000	4.18

API °	29.8
Res Temp (°F)	152
MMP (psig)	**1980**

Test Pressure (psig)	**Recovery factor (% of OOIP) Sample 5**
6500	98.12
5500	97.35
5000	96.51
3000	78.22
2000	55.38
1500	43.78

API °	37
Res Temp (°F)	**165**
MMP (psig)	**3748**

The MMP of each sample was determined by fitting a straight line through each group of data points representing either the miscible or the immiscible region and solving for the interception point of the two trend lines. **Figures 3– 9** show the plots of the recovery factor as a function of pressure for the light crude oil – CO2 systems. The consistency of our experiments is shown in the fact that all data points fall within linear trends. Notice that we have performed three measurements both above and below the MMP. This enables an immediate quality assessment of the consistency of all runs, which is prohibitive when a longer coil is used, as can be observed commonly in miscibility studies performed by commercial laboratories.

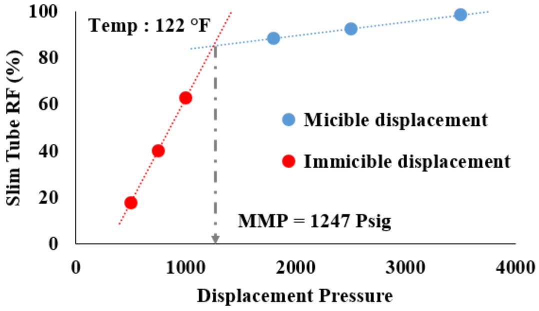


Figure 3. MMP plot for sample 1 using a 20 ft coil

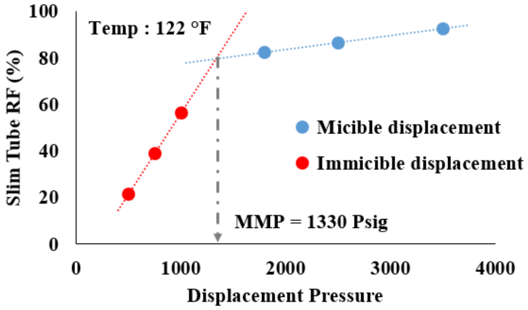


Figure 4. MMP plot for sample 2 using a 20 ft coil

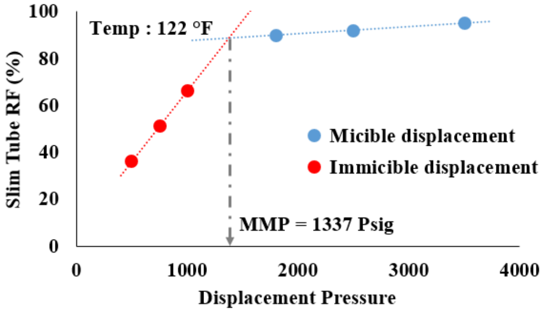


Figure 5. MMP plot for sample 3 using a 20 ft coil

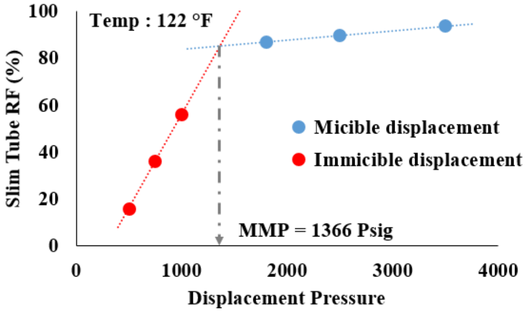


Figure 6. MMP plot for sample 4 using a 20 ft coil

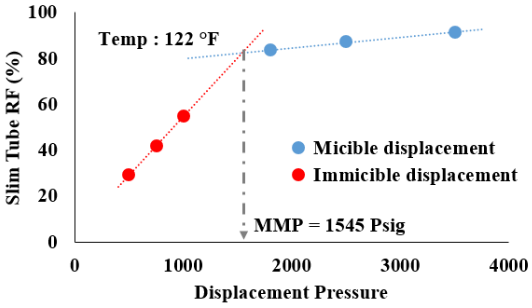


Figure 5. MMP plot for sample 5 using a 20 ft coil

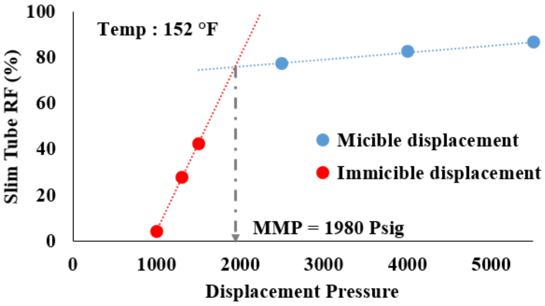


Figure 6. MMP plot for sample 6 using a 20 ft coil

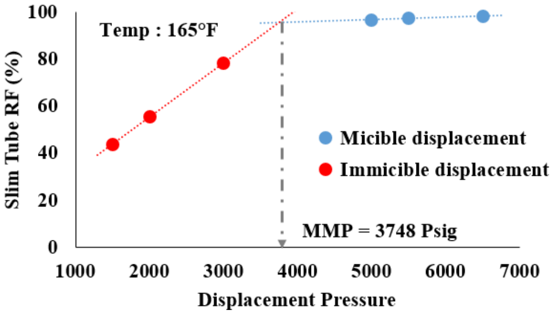


Figure 9. MMP plot for sample 5 using a 20 ft coil at 165°F

Experimental Validation Using an 80 ft Coil

The MMP determination was repeated for samples 3, 4, 5 and 6 using a longer slim tube to validate our results. We chose the samples to cover the whole range of API values in **Tables 1 - 2**. In this case**,** the 80 ft coil was employed instead of the 20 ft described earlier. Apart from the length, all other slim tube characteristics remained the same. The experimental procedure already described, as well as pressure and temperature conditions**,** were also preserved. Four runs were performed for each of the samples to compare the recovery factors and the resulting MMP values. **Figures 10, 11, 12, 13 & 14** represent the results and compare them with the ones obtained with the 20 ft coil.

Test Pressure	Sample 3- Oil recovery (% of OOIP)	
(psig)	20 ft. coil	80 ft. coil
3500	94.98	96.20
2500	91.98	93.08
1000	66.26	67.40
500	36.26	37.12
MMP (psig)	1373	1364

Error
9 Psig ~ 0.6 %

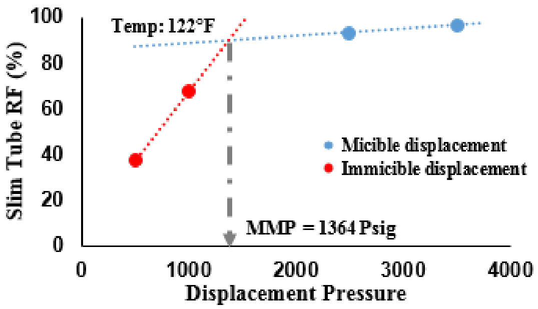


Figure 10 –(a – left) Table comparing the 20 ft and 80 ft slim tube experiment results for sample 3 (b – right) Minimum miscibility pressure plot for sample 3 using an 80 ft coil

The use of the 80 ft slim tube coil consistently led to a lower MMP value when compared with the one obtained using the 20 ft coil. Also, the recovery factors obtained with the long 80 ft coil were always higher than the ones obtained with the 20 ft coil **(Figures 10a, 11a and 12a)**. These results are consistent with the conclusions made by Ekundayo and Ghedan (2013) and by Folck and Nour (1984) who attributed this phenomenon to the stabilizing effect on the transition zone inside the coil that results from the use of a longer coil. However, the differences of 9, 13 and 15 psig found in the MMP values are negligible for the purposes of EOR project design and supports the reliability of the use of a 20 ft slim coil tube for similar light crude oils.

Test Pressure (psig)	Sample 4- Oil recovery (% of OOIP)	
	20 ft. coil	80 ft. coil
3500	93.78	95.31
2500	89.78	92.15
1000	56.00	59.78
500	16.00	18.72

MMP (psig)	1366	1351

Error
15 Psig ~ 1.1 %

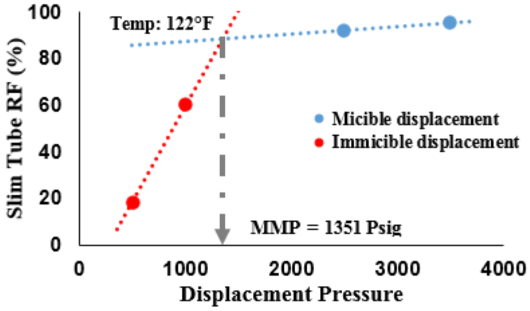


Figure 11 –(a – left) Table comparing the 20 ft and 80 ft slim tube experiment results for sample 4 (b – right) Minimum miscibility pressure plot for sample 4 using an 80 ft coil

Test Pressure (psig)	Sample 5- Oil recovery (% of OOIP)	
	20 ft. coil	80 ft. coil
3500	91.34	92.50
2500	87.16	88.80
1000	54.80	56.80
500	29.23	30.10

MMP (psig)	1545	1532

Error
13 Psig ~ 0.85 %

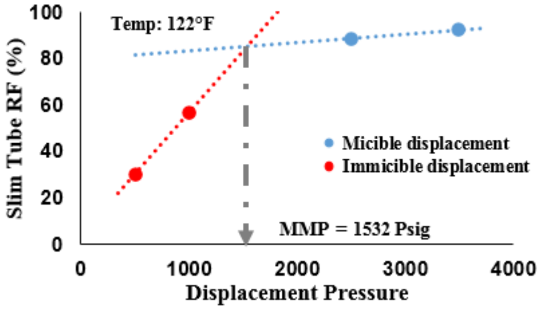


Figure 12 –(a – left) Table comparing the 20 ft and 80 ft slim tube experiment results for sample 5 (b – right) Minimum miscibility pressure plot for sample 5 using an 80 ft coil

Figure 13 represents the redetermination of the MMP for sample 6 using the longer slim tube coil. Note that sample 6 has a higher reservoir temperature of 152 °F and a high MMP value of 1980 psi when compared to the oil samples in Table 1. The 80 ft coil again led to a lower MMP and higher recovery factors which agree with our previous results. However, the difference in MMP of 87 psig is a relatively higher than the differences found in sampels 1-5 at 122 °F.

Test Pressure (psig)	Sample 6- Oil recovery (% of OOIP) 20 ft. coil	80 ft. coil
4000	82.46	84.85
2500	77.24	78.38
1500	42.30	44.69
1000	4.18	5.26
MMP (psig)	1980	1893

Error
87 Psig ~ 4.6 %

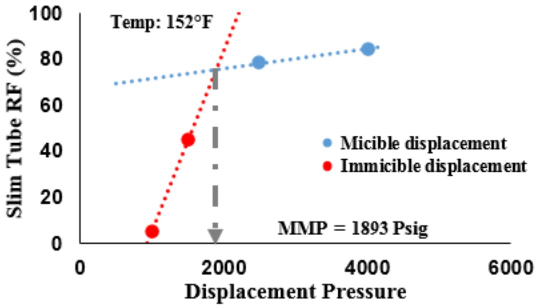


Figure 13 –(a – left) Table comparing the 20 ft and 80 ft slim tube experiment results for sample 6 (b – right) Minimum miscibility pressure plot for sample 6 using an 80 ft coil

Figure 14 represents the redetermination of the MMP for sample 5 at 165 °F using the longer 80 ft coil. Note that this sample has a very high MMP value at these conditions. The redetrmaination of the MMP was conduceted in order to investigate the effect of reducing the coil length in CO_2 – crude oil systems presenting higher MMP values. The 80 ft coil again led to a lower MMP and higher recovery factors which agree with our previous results. However, in this case, the difference in MMP of 180 psig is considerably higher than the differences found in oil sampels with lower MMP values and lower reservoir temperatures.

Test Pressure (psig)	Sample 5- Oil recovery @ 165°F (% of OOIP) 20 ft. coil	80 ft. coil
6500	98.12	98.92
5000	96.51	97.56
3000	78.22	81.93
1500	43.78	44.12
MMP (psig)	3748	3568

Error
180 Psig ~ 5.04 %

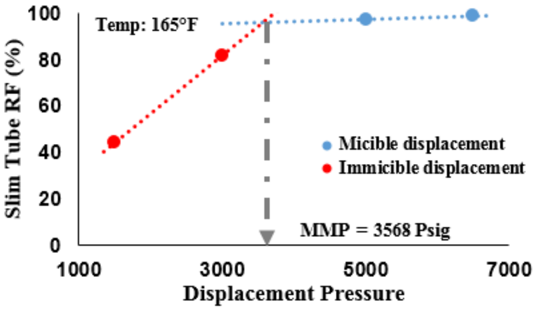


Figure 14 –(a – left) Table comparing the 20 ft and 80 ft slim tube experiment results for sample 5 at 165 °F (b – right) Minimum miscibility pressure plot for sample 5 at 165 °F using an 80 ft coil

The results are in agreement with the numerical validation results that are discussed in the next section. The deviation in the MMP obtained with a 20 ft coil will be more significant at higher reservoir temperatures, higher minimum miscibility pressures and heavier crude oils when compared to the one obtained with an 80 ft coil.

Therefore, exerting caution is recommend when using this technique at high temperatures for CO_2 – crude oil systems with a likely MMP close or higher to 2000 psi until a more precise application window is established. In this way, we were able to explore the effect of reducing the coil length in CO_2 – crude oil systems presenting higher MMP values.

Numerical Validation

A numerical simulation model was also used to validate the results. The PVT behavior of the NBU crude oil (Sample 7) was modeled using the Peng-Robinson (Robinson and Peng 1978) equation of state (EOS) with the Péneloux volume shift (Péneloux et al. 1982). A commercial laboratory provided the composition of this sample using 40 components, which was lumped into eight pseudo-components (Table 3) for modeling purposes. The EOS was tuned using solubility and swelling data for the original reservoir fluid and mixtures resulting from the addition of 50, 100, 150, 200, 250, and 300 mole % of CO2. The partial CCE expansion data was available for all the mixtures while the viscosity data was only available for three of the mixtures.

Table 3. Fluid pseudo-components properties of the NBU oil (Adel *et al.*, 2016)

Component	Composition, fraction	Molar Weight	Critical Pressure (Bar)	Critical Temperature (k)	Acentric Factor
CO_2	0.18	44.01	73.76	304.61	0.22500
N2 + C1	1.78	17.19	44.12	180.82	0.01298
C2 – nC4	5.84	48.20	40.42	393.22	0.16762
iC5+ nC5+	7.20	78.01	31.60	405.82	0.27077
C7 – C12	41.43	124.42	31.55	546.56	0.47127
C13 – C19	23.11	213.20	22.67	603.14	0.81817
C20 – C35	14.11	337.83	18.54	641.19	1.23480
C36 – C80	6.35	775.24	15.10	861.60	1.00745

The simulation model used was Cartesian one-dimensional. The grid size was fixed to 30 cm, and the number of cells was varied to accommodate the different lengths slim tubes. In all cases, the final recovery factor was measured at 1.2 PV of injected CO_2, which was accomplished by varying the injection time. To observe the effect of coil length in MMP, three coil lengths, of 20, 40 and 80 ft were selected. For each length, 12 different simulation runs were performed at various pressures to plot the recovery factor at 1.2 PV of CO_2 injected as a function of pressure. In a similar fashion, as in the case of the laboratory experiments, the MMP was determined by the intersection between a line describing the immiscible trend and another line depicting the miscible one.

To observe the effect of coil tube length for higher MMP values, the temperature was increased for the same system CO_2 – crude oil. The increment in temperature results in higher MMP (Wang and Knight, 1982). Therefore, additional simulation runs were performed at 130, 140, 150 and 160 °F, besides the original reservoir temperature of 122 °F. A total of 180 simulation runs were required to obtain the data presented in this section.

An experimentally determined MMP value was available from a commercial laboratory. The slim tube technique was used with a coil of 40 ft and three data points at 1400 psig, 1600 psig and 1900 psig. A fourth point**,** with 100% recovery at 2400 psig was assumed. We will argue later**,** that the use of only 3 or 4 data points can lead to errors in the MMP determination using the slim tube technique. Therefore, we did not make strong efforts to match the experimental MMP reported of 1687 psig. Our simulation result corresponding to the original reservoir temperature of 122 °F and a similar slim tube length of 40 ft yielded to an MMP of 1607 psi, which is considered close enough to the experimental data reported by the commercial laboratory. It is also close to the value of 1563 psig experimentally measured with a 20 ft coil and using dead oil from the same field by Tovar *et al*, (2015).

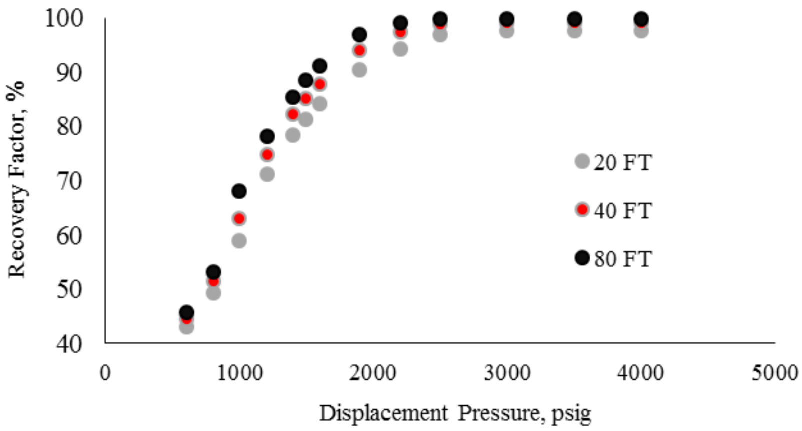


Figure 15. Simulated recovery factors as a function of pressure for different coil lengths

Figures 15 and 16 represent the simulated MMP results for sample 7 at 122°F for three different coil lengths. The MMP value slightly decreases with the increase in coil length which is in agreement with our experimental results, and the conclusions of Ekundayo and Ghedan (2013), as it was argued earlier. The difference of 36 psig resulting in an error of 2.29 % found in the MMP value between the 20 ft and 80 ft simulation results is negligible for the purposes of EOR project design and supports the reliability of the use of a 20 ft slim tube coil for similar light crude oils at low temperatures.

Column length (ft)	**80**	**40**	**20**
MMP (Psig)	1588	1607	1625
RF @ MMP	98.82	98.50	96.21
Error %	--	1.16	2.29
Difference (Psig)	--	18.5	36.48

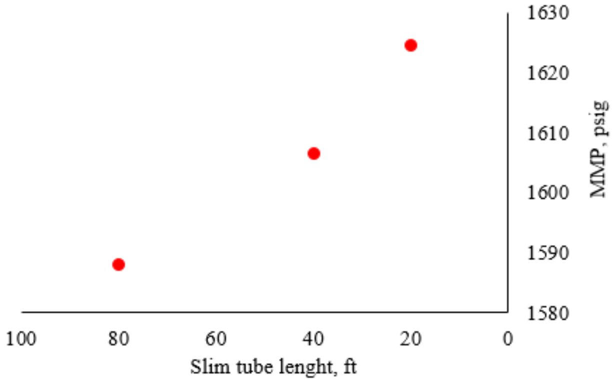


Figure 16 –(a – left) Table is comparing the simulated slim tube experiment results for the NBU oil at different coil lengths. (b – right) Plot of the simulated MMP as a function of coil length for the NBU oil

The MMP for this system CO_2 – NBU crude oil was artificially augmented by increasing the reservoir temperature. As it has been pointed out in literature raising the temperature tends to delay extraction, probably due to increased surface energy, which prevents the hydrocarbon and CO_2 molecules from coherence (Wang and Knight, 1982), leading to a higher MMP. In this way, we were able to explore the effect of reducing the coil length in CO_2 – crude oil systems presenting higher MMP values. The results are represented in **Figure 17**.

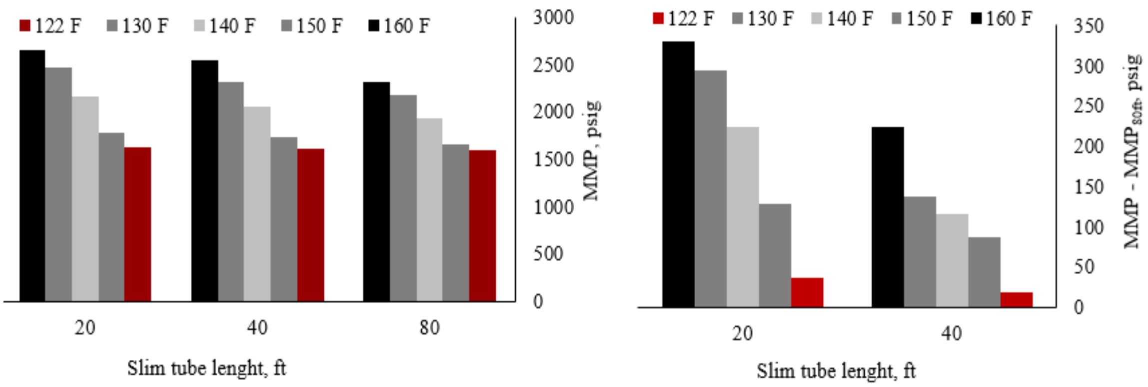


Figure 17 –(a – left) Simulated MMP as a function of coil length and temperature. (b – right) Deviation in the MMP value from the one obtained with the 80 ft coil using different coil lengths and temperatures for the NBU oil

The plots show that the increase of the MMP value with coil length reduction is higher in CO_2 – crude oil systems presenting higher MMP (**Figure 17 – b**). For MMP values in the range of our experimental results, the simulation also shows small increments in MMP that are not relevant and that support the use of 20 ft coil. However, as the MMP gets closer and exceeds 2000 psig, the deviation in the MMP obtained with a 20 ft coil is significantly higher compared to the one achieved with an 80 ft coil. Nevertheless, since these results are limited to only one CO_2-crude oil system and the MMP is being artificially incremented by increasing the temperature we must not generalize this trend.

Our experimental and simulation data suggest that it is safe to use the fast-slim tube technique when the MMP is significantly below 2000 psig, as it would be expected for most light oils of common interest for CO_2 EOR applications. As the MMP gets close to or exceeds 2000 psig, or if the reservoir temperature is too high, we recommend exerting caution. We believe it is possible that heavier oils and high temperatures conditions need a longer coil to host a larger transition or mixing zone.

It becomes evident that more experimental and simulation work is required to establish a better window of applications for the fast slim tube technique. Moreover, due to the complexity of crude oil samples, it may not be possible to extend the application of this technique to all light oils as defined in terms of only API gravity, but instead, several parameters like SARA fractions, acid numbers, and heavy component fractions may have to be considered.

In favor of the fast-slim tube technique**,** is the fact that produces higher MMP values than the regular technique. This is important because falling below the MMP can have a hugely detrimental impact on the expected ultimate recovery because of the high dependence of the recovery factor on pressure below the MMP. Consequently, any significant deviation in MMP that may be possible for heavier crude oil will not reduce the expected ultimate oil recovery having a minor impact on economics. This minor impact would be caused by the over design of the injection facilities to inject at a pressure which is slightly higher.

Quality Control in MMP Determination

Figure 18 represents the simulated results for the NBU crude oil (sample 7) at 122°F and 40 ft coil length. We performed 12 simulation runs at different pressures to show that even when the immiscible region and the miscible region are well defined with linear trends, the transition from one to the other is smooth. As a consequence, the selection of the pressures at which the experimental runs are performed is crucial.

When a total of four runs are performed, with only two data points to define each one of the linear trends, above and below MMP, this is of particular importance. Due to the long time required for each run when using an 80 ft coil, doing more than four experimental runs may be prohibitive, and therefore commercial laboratories only perform three or four points. We argue in this section that this practice can lead to errors in the resulting MMP.

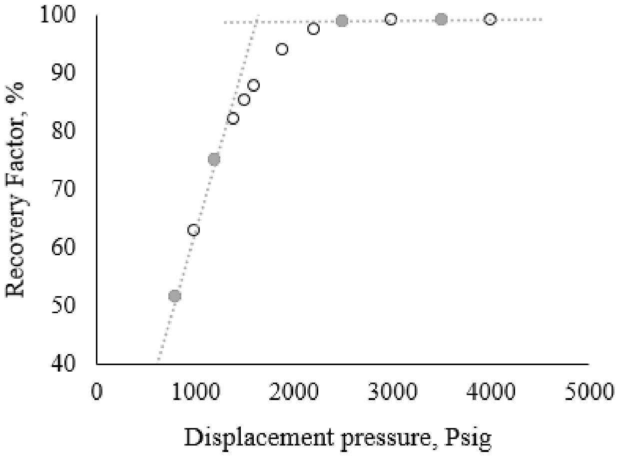


Figure 18. Simulated Slim tube experiment results at 122°F using a 40 ft coil length

Figures 19 and 20 use the same data represented in **Figure 18**, but they show additional trends to exemplify how the selection of one point inside the transition zone can lead to substantial deviations in the resulting MMP. In all the examples, if the rest of the points are ignored, it is not possible to know that one of the points is actually not part of the linear trend.

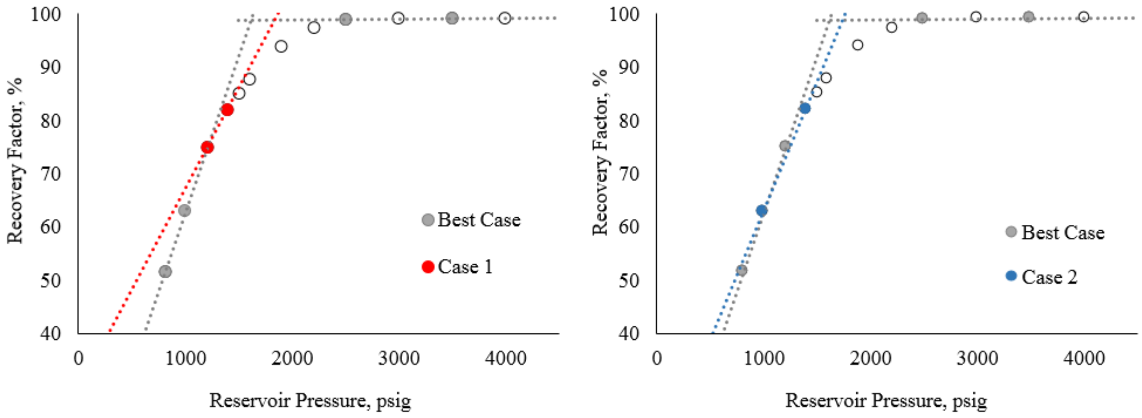
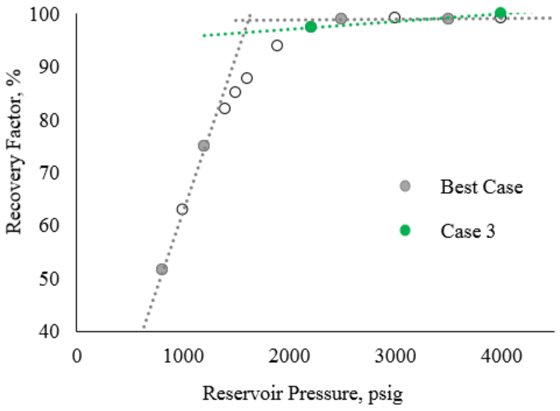


Figure 19. MMP plots showing the impact of immiscible pressure points selection on the value of the MMP



Case	MMP (psig)	MMP Deviation (psig)	Error %
Best Case	1607		--
Case 1	1832	**225**	14.00
Case 2	1736	**129**	8.03
Case 3	1571	**36**	2.24

Figure 20 –(a – left) MMP plot indicating the impact of miscible pressure points selections on the MMP value. (b – right) Table comparing the MMP values and the error at each set of pressure points selection

One of the main advantages of the fast – slim tube technique we propose is that it enables the performance of three measurements, both above and below the MMP, automatically providing for an assessment of the consistency of the results. Hence, it is guaranteed that the data points are within the linear trends and not in the transition zone, while still significantly reducing the time and cost of the experiment.

Performing six runs using the 20 ft coil leads to a 50% reduction in the experimental time when compared to only performing four runs using the longer 80 ft coil (**Table 4**). Consequently, the use of the fast-slim tube technique yields to an MMP value slightly higher than the one determined with the conventional technique. Such deviation can be offset with the potential errors resulting from using four data points with longer coils, meaning that the utilization of an 80 ft long coil does not guarantee a more precise determination of the MMP in light oils.

Experimental Time Reduction

The MMP determination using the fast-slim tube technique with six points and a 20 ft length coil takes about half the time when compared to the conventional technique using four points and an 80 ft coil. If the fast-slim tube technique is employed with only four points as well, it requires about a third of the time the conventional method would.

Table 4. Experimental time for the MMP determination (Adel et al., 2016)

		Time required (hours)
6 runs	20 ft coil	234
4 runs	80 ft coil	481

Time reduction	**51.35%**

CHAPTER V

CONCLUSIONS*

We have shown that is possible to significantly reduce the time required to measure the MMP for light crude oils by using a short 20 ft coil instead of a traditional 80 ft without losing the accuracy of the method. Therefore, making the technique more cost effective.

The resulting MMP values using the fast-slim tube technique are slightly higher than the ones obtained with the traditional method; however, the difference between them is considered irrelevant for EOR project purposes.

A 20 ft coil is believed to be sufficient to host the mixing zone in the displacement of light crude oil with CO_2 at low temperatures when the velocity of the displacement is slow enough to enable the transverse dispersion for the elimination of viscous fingering.

We recommend the use of three data points below MMP, and three more above it, in the procedure for the fast-slim tube technique for quality control and to ensure the consistency of the experiments.

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*Parts of the conclusions presented in this chapter have been reprinted from “Fast-Slim Tube: A Reliable and Rapid Technique for the Laboratory Determination of MMP in CO2 - Light Crude Oil Systems” by Imad A. Adel, Francisco D. Tovar, and David S. Schechter. SPE Paper 179673. Copyright 2016 by the Society of Petroleum Engineers. Reproduced with permission of SPE. Further reproduction prohibited without permission.

We have illustrated with examples that the use of three to four data points can lead to errors in the determination of the MMP that could offset the benefits of using the conventional slim tube technique with long coils over the fast-slim tube procedure proposed.

The fast – slim tube method enables the performance of three measurements, both above and below the MMP, automatically providing for an assessment of the consistency of the results. Hence, it is guaranteed that the data points are within the linear trends and not in the transition zone, while still significantly reducing the time and cost of the experiment.

The MMP determination using the fast-slim tube technique with six points and a 20 ft length coil takes about half the time when compared to the conventional technique using four points and an 80 ft coil. If the fast-slim tube technique is employed with only four points as well, it requires about a third of the time the conventional technique would.

Exerting caution is recommend when using this technique at high temperatures for CO_2 – crude oil systems with a likely MMP close or higher to 2000 psi until a more precise application window is established. Higher MMP would potentially be linked to heavier crude oils and high temperature conditions that may need a longer coil in order to host the mixing zone. In this way, we were able to explore the effect of reducing the coil length in CO_2 – crude oil systems presenting higher MMP values.

In favor of the fast-slim tube technique**,** is the fact that produces higher MMP values than the conventional technique. This is important because falling below the MMP can have a hugely detrimental impact on the expected ultimate recovery because of the high dependence of the recovery factor on pressure below the MMP. Consequently, any significant deviation in MMP that may be possible for heavier crude oil will not reduce the expected ultimate oil recovery having a minor impact on economics. This minor impact would be caused by the over design of the injection facilities to inject at a pressure which is slightly higher.

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