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Fast-Slim Tube: A Reliable and Rapid Technique for the Laboratory Determination of MMP in CO_2 - Light Crude Oil Systems

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Abstract

We present a technique that enables the determination of the minimum miscibility pressure (MMP) of a CO_2 – oil system 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.

We determined the MMP for five CO_2 – crude oil systems 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 our technique was proven with 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, the use of the 20 ft slim tube is justified as the length of the mixing zone is shorter. We support our results with the use of numerical simulation.

The reduction in the time required for slim tube experiments results in a fast, economic 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 to determine the MMP for designing a CO_2 enhanced oil recovery project.

Introduction

For decades carbon dioxide has been a popular 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 (Izgec *et al*. 2005, Denney 2010). 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.

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.

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, 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). 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).

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) defines 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. All recovery factors are measured at 1.2 PV of injected gas.

In order 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 in excess 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. We will argue 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, we adopted the fast version of the slim tube technique using a 20 ft coil for similar light crude oil samples. In this paper we show the MMP determination for five light crude oil samples from the North Burbank Unit and the Oklahoma/Texas Panhandle area using a 20 ft packed coil. All five samples are light crude oils with API's of 42.91, 38.8, 38.31, 37 and 34.90 °API, which are close to the properties of the NBU sample of 37.42 °API used by Tovar *et al.* (2015).

The main objective of this work is to show that the slim tube technique for light crude oil-CO_2 systems can be made more affordable with considerably reducing the experimental time, without significant loss of the accuracy of the conventional method. We 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.

Experimental Procedure

Minimum Miscibility Pressure Determination

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 inside diameter of 0.25 in, thickness of 0.063 in, and is packed with 100 – 150 mesh Ottawa sand.

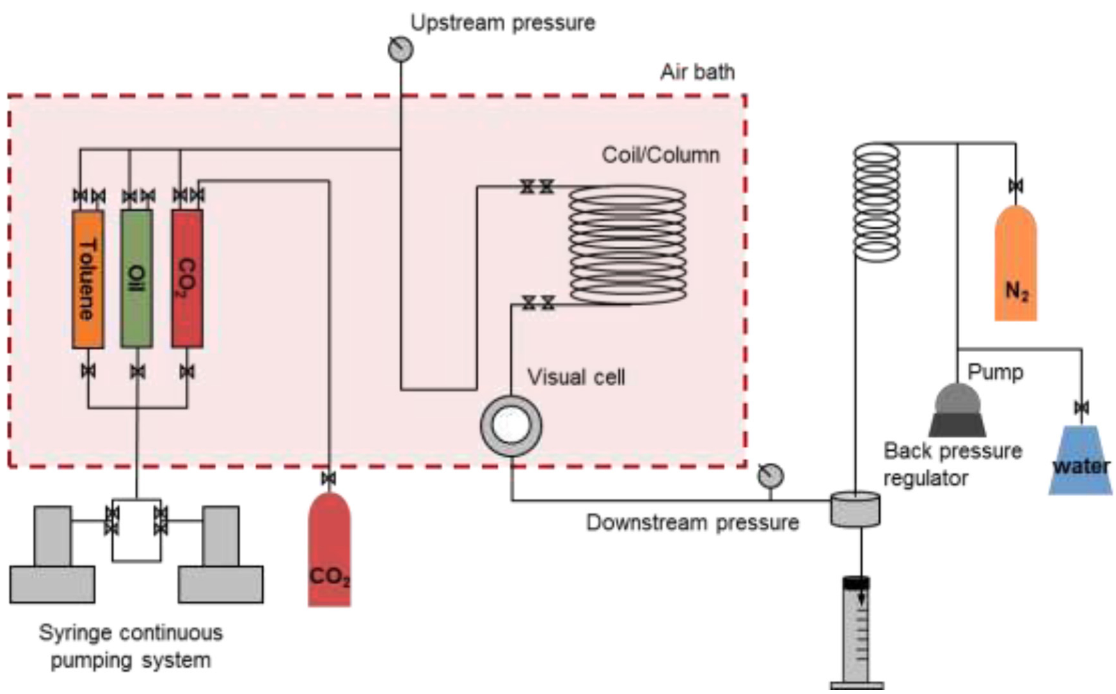


Figure 1—Schematic of the set up for the MMP determination apparatus using the slim tubing technique (Tovar *et al.*, 2015)

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.

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, which for all of our experiments was 122 °F. 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 cm3/min to displace the oil. The effluents are measured along the duration of the experiment to calculate the recovery factor at each pressure. 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 between these two regions (Figures 2–6).

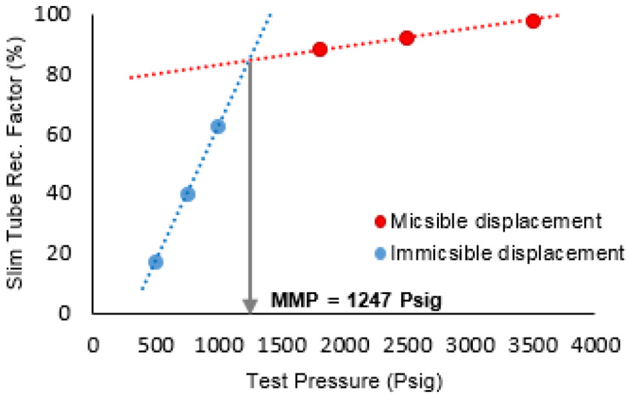


Figure 2—MMP plot for sample 1 using a 20 ft coil

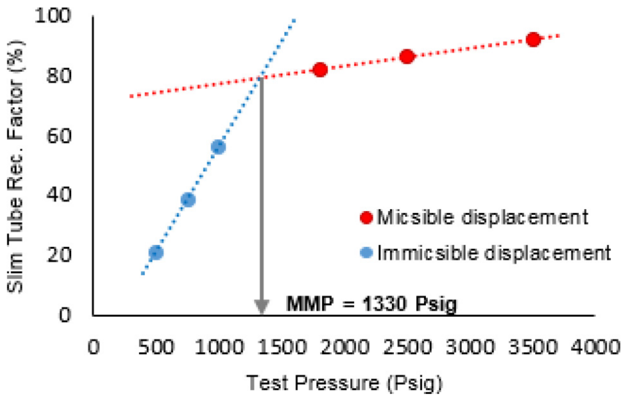


Figure 3—MMP plot for sample 2 using a 20 ft coil

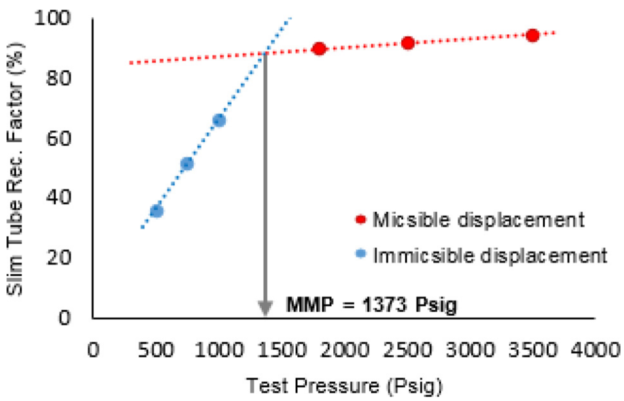


Figure 4—MMP plot for sample 3 using a 20 ft coil

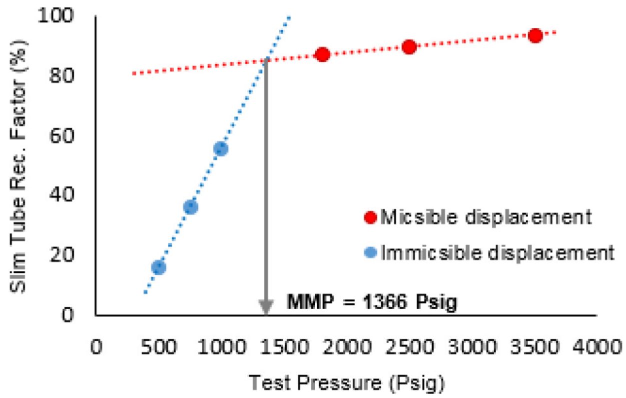


Figure 5—MMP plot for sample 4 using a 20 ft coil

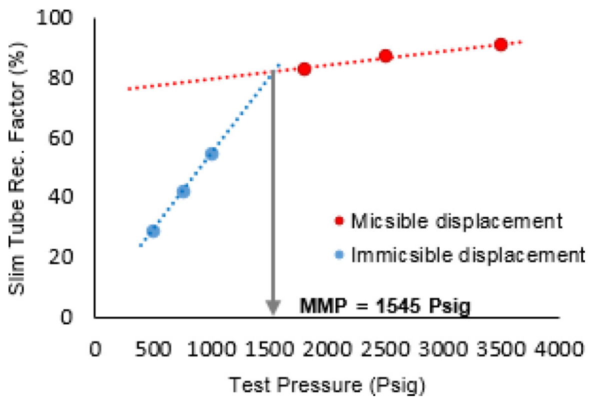


Figure 6—MMP plot for sample 5 using a 20 ft coil

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 cases, the pressures for the displacement were 500, 750, 1000, 1800, 2500 and 3500 psig.

Results and Discussions

Table 1 represents the results for the six runs at 3500, 2500, 1800, 1000, 750 and 500 psig, for the five light crude oil samples used in our work. 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.

Table 1—Recovery factor as a function of pressure

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
MMP (psig)	**1247**	**1330**	**1373**	**1366**	**1544**

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 2–6 show the plots of the recovery factor as a function of pressure for the light crude oil – CO_2 systems. The consistency of our experiments is shown with the fact that all data points fall within the 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.

Experimental Validation

The MMP determination was repeated for samples 3, 4 and 5 using a longer slim tube to validate our results. We chose the samples to cover the whole range of API in Table 1. In this case, an 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 the pressure and temperature conditions, were also preserved. Four runs were performed for each of the samples at the pressures of 3500, 2500, 1000 and 500 psig. Figures 7, 8 & 9 represent the results and compare them with the ones obtained with the 20 ft coil.

Test Pressure (psig)	Sample 3- Oil recovery (% of OOIP) 20 ft. coil	80 ft. coil	Error
3500	94.98	96.20	
2500	91.98	93.08	
1000	66.26	67.40	
500	36.26	37.12	
MMP (psig)	1373	1364	9 Psig ~ 0.6 %

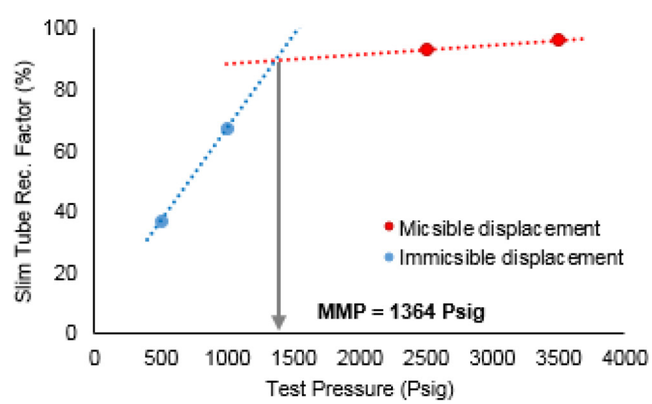


Figure 7—(a - left) Table comparing the slim tube experiment results for sample 3 using 20 ft and 80 ft packed coils. (b - right) MMP plot showing the recovery factor as a function of pressure for the MMP determination of sample 3 using an 80 ft packed coil.

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 %

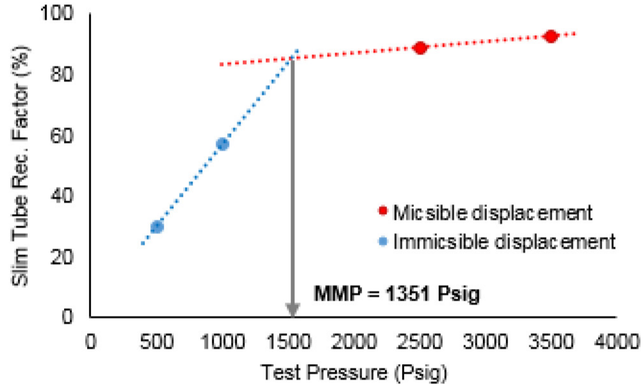


Figure 8—(a - left) Table comparing the slim tube experiment results for sample 4 using 20 ft and 80 ft packed coils. (b - right) MMP plot showing the recovery factor as a function of pressure for the MMP determination of sample 4 using an 80 ft packed coil.

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 %

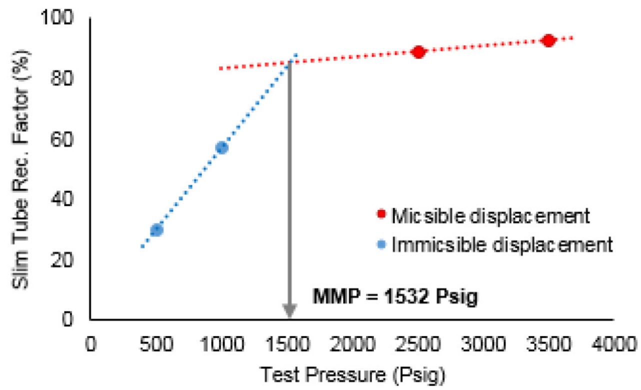


Figure 9—(a - left) Table comparing the slim tube experiment results for sample 5 using 20 ft and 80 ft packed coils, (b - right) MMP plot showing the recovery factor as a function of pressure for the M MP determination of sample 5 using an 80 ft packed coil.

The use of the 80 ft slim tube coil 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 consistently higher than the ones obtained with the 20 ft coil (Figures 7a, 8a, 9a). These results are consistent with the conclusions made by Ekundayo and Ghedan (2013) and by Folck and Nour (1984) who attributed this phenomena 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 is 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.

Numerical Validation

A numerical simulation model was also used to validate the results. The PVT behavior of the NBU crude oil (Sample 6) was modeled using the Peng-Robinson (Robinson and Peng 1978) equation of state (EOS) with the Péneloux volume shift (Péneloux, Rauzy, and Fréze 1982). A commercial laboratory provided the composition for this sample using 40 components, which was lumped into 8 pseudo-components (Table 2) 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 CO_2. The partial CCE expansion data was available for all the mixtures and the viscosity data for three of them.

Table 2—Sample 6 fluid pseudo-components propreties

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 different pressures to plot 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.

In order to observe the effect of coil tube length for higher MMP values, the temperature was increased. For the same system CO_2 – crude oil, 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 an MMP of 1607 psi, which we considered to be 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).

Figures 10 and 11 represent the simulated MMP results for sample 6 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 the 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 coil tube for similar light crude oils.

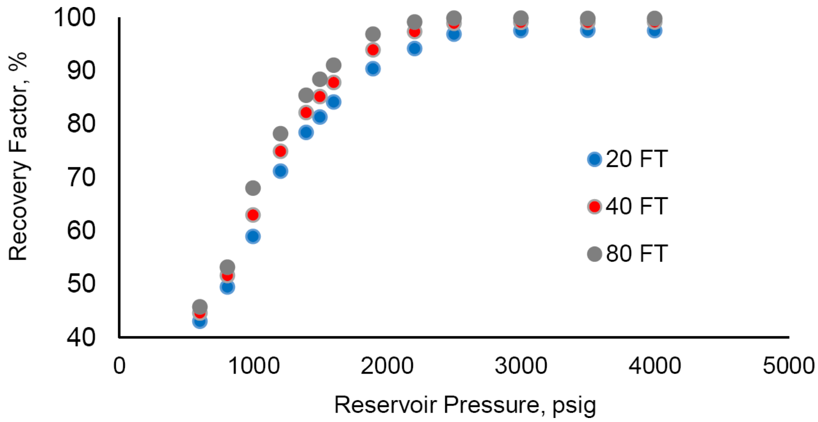


Figure 10—Simulated recovery factors as a function of pressure

Colum 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

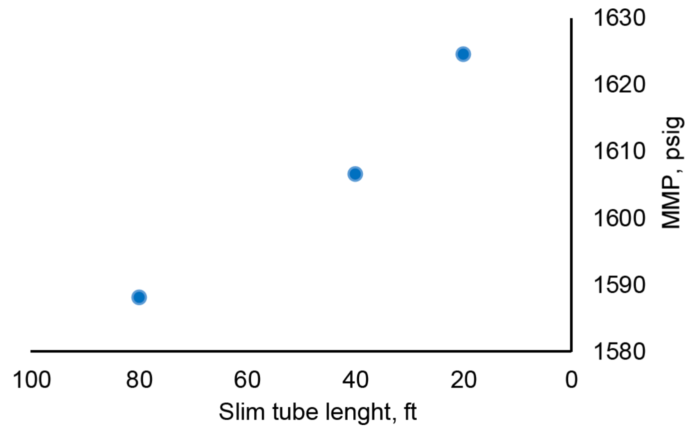


Figure 11—(a – left) Table comparing the simulated slim tube experiment results for sample 6 at different coil lengths. (b – right) Simulated MMP plot of sample 6 as a function of coil length.

The MMP for this system CO_2 – NBU crude oil was artificially incremented 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 12.

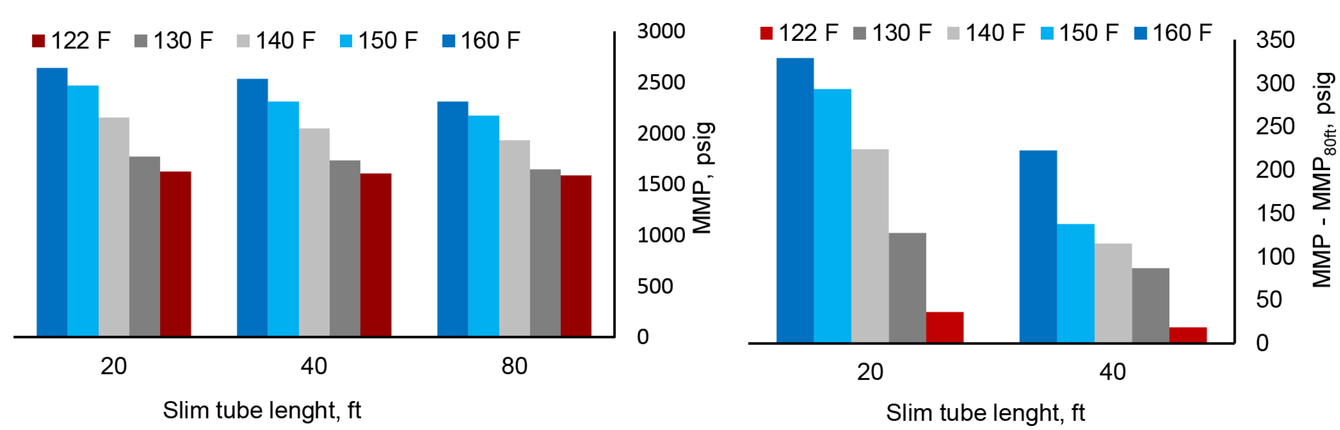


Figure 12—(a - left) Simulated MMP as a function of coil length and temperature, (b - right) Deviation in the MMP value from the one obtained with the 80 ft coil using different coil lenghts and temperatures.

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 12 – b). For MMP values in the range of our experimental results, the simulation also show 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 obtained 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, we recommend exerting caution. We believe it is possible that heavier oils need a longer coil in order to host a larger transition or mixing zone.

It becomes evident that more experimental and simulation work is required to better establish a window of application for the fast slim tube technique. Moreover, due to the complexity of hydrocarbons 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 components 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 huge detrimental impact in the expected ultimate recovery because of the high dependence of recovery factor on pressure below the MMP. Consequently, any meaningful deviation in MMP that may be possible for heavier crude oil will not reduce the expected ultimate oil recovery having a minor impact in 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 13 represents the simulated results for the NBU crude oil (sample 6) 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 especially important. 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 do three or four points. We argue in this section that this practice can lead to errors in the resulting MMP.

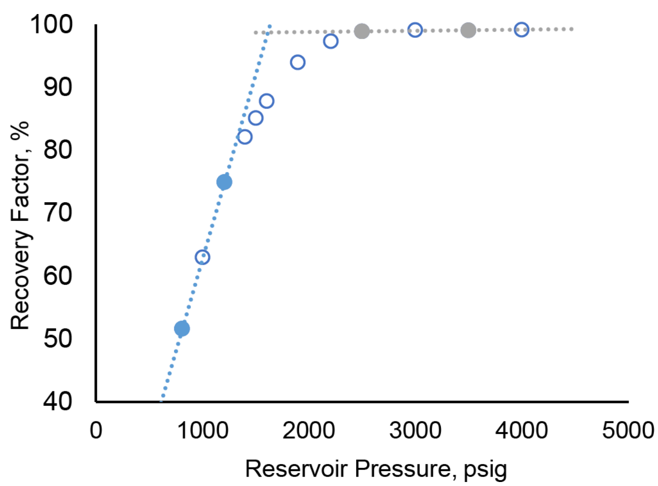


Figure 13—Simulated Slim tube experiment results at 122°F and 40 ft coil

Figures 14 and 15 use the same data represented in Figure 13, but they show additional trends to exemplify how the selection of one point inside the transition zone can lead to important 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.

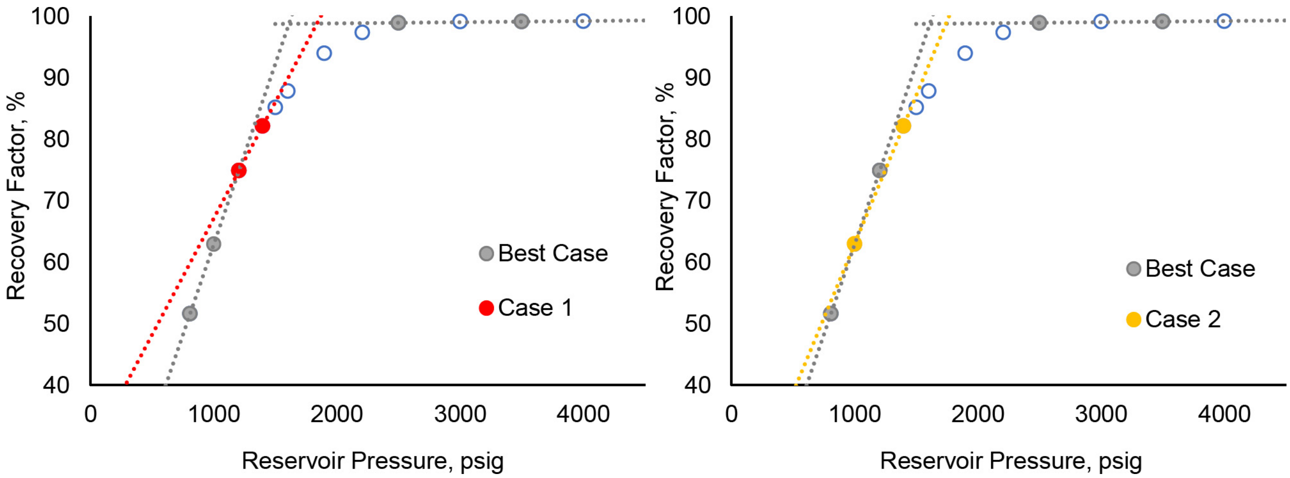
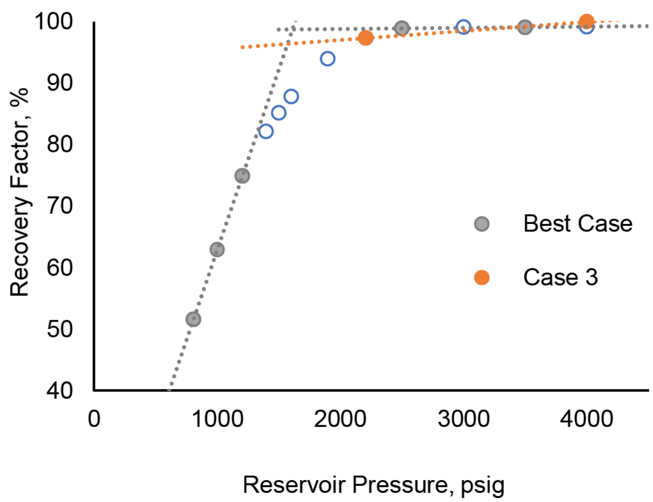


Figure 14—MMP plots showing the impact of immicible pressure points selctions on the MMP value



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 15—(a - left) Minimum Mcsibility Pressure plot showing the impact of micsible pressure points selctions 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 inside 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 3). Consequently, whereas, the use of the fast-slim tube technique yields 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 use of a 80 ft long coil does not guarantee a more precise determination of the MMP in light oils.

Table 3—Experimental time for MMP determination

		Time required (hours)
6 runs	20 ft coil	234
4 runs	80 ft coil	481
Time reduction		**51.35%**

Conclusions and Recommendations

1. 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.
2. 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.
3. A 20 ft coil is believed to be sufficient to host the mixing zone in the displacement of light crude oil with CO_2 when the velocity of the displacement is slow enough to enable the transverse dispersion for elimination of viscous fingering.
4. 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.
5. 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.
6. 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.
7. We recommend to exert caution in using this technique for CO_2-crude oil systems with MMP close or higher to 2000 psi until a more precise application widow is established. Higher MMP would potentially be linked to heavier crude oils that may need a longer coil in order to host the mixing zone.

Nomenclature

EOR	Enhanced oil recovery
EOS	Equation of state
MMP	Minimum miscibility pressure, psig
NBU	North Burbank Unit
OOIP	Oil originally in place
PV	Pore volumes
CCE	Constant composition expansion

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