Carbon Dioxide Minimum Miscibility Pressure with Nanopore Confinement in Tight Oil Reservoirs

R S Mohammad¹, *, S Zhang¹, E Haq², X Zhao³ and S Lu¹

1. Oil and Gas Field Development Engineering Department, China University of Petroleum-Beijing, Changping, China
2. Geological and Geosciences Department, China University of Petroleum-Beijing, Changping, China
3. Centre of Geoscience Computing, School of Earth and Environmental Science, University of Queensland, Brisbane, Australia
E-mail: 2846921094@qq.com

Abstract. CO₂-injection is one of the capable processes in EOR from low-permeable reservoirs and MMP determination is a key factor in estimating the displacement efficiency of the CO₂ in the EOR processes. The laboratory procedures for MMP determination recognized in the oil industry are slim-tube, rising-bubble, or vanishing interfacial tension (VIT). However, the presence of nanopores in tight formations influences phase equilibrium, causing reduction in MMP. Instead, the existing MMP correlations need to be modified for tight reservoirs that might result in reliable MMP. Therefore, MMP measurement is performed using WinProp to validate correlations for tight oil samples. This study presents MMP determination experimentally using VIT for tight oil samples in both recombined-oil and dead-oil conditions. Subsequently, results obtained from VIT are compared with slim-tube results and the relative error was found 4.86% for recombined-oil and 23.36% for dead-oil. A huge deviation between VIT and slim-tube is noted while measuring MMP for dead-oil, due to deficiency of multiple contacts miscibility and stabilization of heavier fractions. subsequently, an already incorporated correlation for MMP is utilized, considering the effect of nanopore confinement. This study provides an appropriate technique for predicting MMP considering the capillary pressure and solubility on well performance of tight reservoirs.

1. Introduction
Carbon dioxide enhanced oil recovery (EOR) is the main choice to execute CO₂ geological sequestration due to the extra economic advantage, prevailing infrastructure and acquaintance of petroleum and its operations. A CO₂ Miscible injection process have become commonly used procedure for the improved oil recovery worldwide. Thus, many CO₂ field applications have been recorded as successful [1], [2]. In miscible CO₂ injection process, the main task is to displace the trapped oil using miscible CO₂ injection process, which increases the displacement efficiency and enhance the oil recovery. minimum miscibility pressure (MMP) determination is a key parameter in estimating the capability of injected fluid in the EOR processes. The miscibility is achieved at the MMP, when interfacial tension among the CO₂ and reservoir fluid is approaching zero, which
consequences in potential transfer of molecules across the interface leading to mutual miscibility and homogeneous fluid formation [3].

There are many experimental procedures for the determination of MMP such as Slim-tube (ST), vanishing interfacial tension (VIT) and raising bubble (RB). A number of studies presented the slim-tube for different gas-oil systems for calculation of MMP and formed various standards for its prediction. However, VIT experiment is achieving cohesion within the oil industry because it is requiring extremely short time and less expenses in estimating CO$_2$-Oil MMP while slim-tube needs extended time. The concept of VIT is to predict the miscibility situations by calculating the interfacial tension through the fluid phases against differing injectivity pressures or composition of the injected fluid and subsequently calculates the MMP by extrapolation [4]. The miscibility mechanism is achieved, as interfacial tension (IFT) among injected CO$_2$ and the reservoir fluid reaches zero and transmission of molecules take place between fluid bulks that results in miscibility development. Holm et al. introduced the MMP as oil recovery reaches 80% at gas breakthrough [5]. Waqar et al. conducted experimental work and compared VIT and ST test, however they did not consider the nanopore confinement effect in their study [6]. Rao et al. applied VIT to realize the miscibility conditions for a couple of gas–oil samples by altering the compositions of the injected fluid and injectivity pressure [7], [8]. Orr & Jessen have questioned the viability of this approach, but recent studies by Ayirala and Rao [9] showed that the experimental VIT MMP results were within 5 to 8 percent of the slim-tube MMP values [10]. Zhang et al. proposed correlation for CO$_2$-Oil MMP with nanopore confinement effect which lacks experimental analysis [11] in contrast to this study.

On the other hand, a diversity of corrections is in appreciation of CO$_2$-Oil MMP, which help engineers in making decisions to develop injectivity status and to design appropriate downstream facilities [12]. The central factors impacting CO$_2$-Oil MMP correlations are reservoir fluid compositions, reservoir temperature and CO$_2$ injectivity conditions [13]. In the previous studies, reservoir temperature was considered the most significant parameter influencing the CO$_2$-Oil MMP. However, few techniques have huge inconsistencies with the data obtained from laboratory work. It was noted that MMP is further affected by molecular weights of crude oil [14]. Subsequently, the prognosis of CO$_2$-Oil MMP is required to deem crude oil composition for computing MMP. Higher concentration of lighter components in the crude oil increases the MMP, while higher concentration of intermediate components reduces the MMP [15]. Most of the pores in tight reservoirs are on the order of a few nanometers varying from 3nm to 100nm and from 40nm to 1500nm for shales and sandstones, respectively [16].

Many researches pointed that the fluid phase behavior in these nanopores diverges notably then that of macro pores, which cause variations in Van der Waals forces, molecules orientation in such a nanoscale pore structure [17], as presented in Figure 1. The molecular orientation can be transformed due to nanopore confinement effect as proposed by Pitakbunkate et al [18]. They show that the conventional liquid-vapor equilibrium (VLE) calculations could be extended to unconventional reservoirs by including capillary pressure and shift in critical temperature and pressure in VLE calculations. A mathematical algorithm is used to calculate VIT by integrating the extended VLE calculation to any MMP algorithm, such as the multiple mixing cells (MMC) model of Ahmadi and Johns [19]. In this approach, incremental pressure increase as IFT approaches zero.

Nanopore confinement can cause fluid phase equilibrium alteration with shifting critical properties [20] resulting in different fluid behavior. Yangyang et al., observed that the CO$_2$ density near pore walls is higher than that in pore centres [21]. Since the crude oil phase equilibrium and fluid properties in a confined system deviate from those at their bulk state, CO$_2$-Oil MMP is decreased with confinement due to critical properties shift. Therefore, the consequence of nanopore confinement on CO$_2$-crude oil MMP is studied and incorporated into a new novel CO$_2$-Oil MMP correlation. The recommended screening technique is carried out by phase behavior module of CMG (Winprop) using tight reservoir crude oil samples.
As a result of the above-mentioned experimental MMP approaches, a study for MMP measurements using VIT experimental procedure is performed. The crude oil of tight reservoir is being utilized with CO$_2$ in both the stock tank oil (dead oil) and recombined oil (live oil) forms individually for displacement study. Furthermore, a comparative estimation of CO$_2$-crude oil MMP with confinement effect calculating correlations with VIT measured MMP was also carried out using CMG WinProp to find an appropriate correlation for precise MMP prediction.

2. Methodology

2.1. MMP Correlation

Many researchers developed correlations for computing crude oil - CO$_2$ MMP. Holm & Josendal (1974) modified correlation to predict the CO$_2$ MMP based on molecular weight of C$_5^+$ and the desired temperature of the reservoir. Lee J. provided a model based on reservoir temperature and CO$_2$ vapor pressure for estimating MMP. Furthermore, procedure by Orr and Jensen was almost suitable correlation for predicting MMP of low temperature reservoirs. Yuan et al. presented correlation to predict CO$_2$-oil MMP using intermediates and heavier mole frictions of reservoir fluid and reservoir temperature. Shokir developed a new model for the prediction of both impure and pure CO$_2$ displacements. However, none of above-mentioned CO$_2$-crude oil MMP determining correlations might prove to be reliable MMP with confinements effect. Therefore, a novel correlation is proposed to take into account the effect of nanopore confinement in determining the CO$_2$-oil MMP. The solubility parameters for hydrocarbon components can be measured by [22]:

$$
\sigma = \frac{\alpha \delta^2 V^{0.33}}{A}
$$

Eq. (1)

Where

- $\delta$ is solubility parameters,
- $\sigma$ is interfacial tension,
- $V$ is molar volume,
- $\alpha$ is constant and
- $A$ is coefficient for solubility parameters.
Furthermore, the oil solubility parameters can be determined from average molecular weights and reservoir temperature [23].

\[ \delta_{oil} = 0.01M + 6.54 - 0.01(T - 25) \]  
\[ \text{Eq. (2)} \]

Where

- \( T \) is reservoir temperature,
- \( M \) is molecular weight.

The solubility parameters for CO\(_2\) can be estimated by the following expression [24]

\[ \delta_{CO_2} = bP_c^{0.5} \left( \frac{\rho_r}{\rho_{r(lig)}} \right) \]  
\[ \text{Eq. (3)} \]

Where

- \( P_c \) is critical pressure,
- \( \alpha \) is constant,
- \( \rho_r \) is reduced density and
- \( \rho_{r(lig)} \) is reduced density of gas compressed to a liquid state.

The pressure condition that corresponds to \(|\delta_{oil} - \delta_{g}| \approx 3.0 \text{ (cal/cm}^3\text{)}^{0.5}\) is the CO\(_2\)-crude oil MMP [23].

At reservoir temperature, the relationship between gas solubility and reduced density can be calculated by Equation (3). Then the CO\(_2\) density can be obtained. According to Figure 2, the CO\(_2\)-oil MMP can be estimated.

\[ \text{Figure 2. Density of CO}_2 \text{ required for miscible displacement [17].} \]
Taking the confinement effect into consideration, a critical property shift is given by the following equations [20]:

\[
\Delta P_c = \frac{P_c - P_{cp}}{P_{cp}} = 0.9409 \frac{\sigma}{r_p} - 0.2415 \left(\frac{\sigma}{r_p}\right)^2 \tag{4}
\]

\[
\Delta T_c = \frac{T_c - T_{cp}}{T_{cp}} = 0.9409 \frac{\sigma}{r_p} - 0.2415 \left(\frac{\sigma}{r_p}\right)^2 \tag{5}
\]

\[
\sigma = 0.2444 \sqrt[4]{\frac{T_c}{P_c}} \tag{6}
\]

Where \(\Delta P_c\) and \(\Delta T_c\) are the critical pressure and critical temperature shift due to nanopore confinement effect, respectively. \(P_c\) and \(T_c\) are the critical pressure and critical temperature, respectively. \(P_{cp}\) and \(T_{cp}\) are the pore critical pressure and pore critical temperature, respectively. \(r_p\) is the pore radius and \(\sigma\) is the Lennard-Jones size parameter.

Subsequent to the above steps, the \(\text{CO}_2\) density can be obtained. Consequently, it requires to be modified by the following equation at frequent pore radius in 50 nm or less as proposed by Zhang et al [11]. Then the modified \(\text{CO}_2\)-Oil MMP corresponding to Figure 2 with the nanopore confinement effect can be achieved.

\[
\rho_{\text{confined}} = -0.0003(\ln r_p)^4 + 0.0088(\ln r_p)^3 - 0.0973(\ln r_p)^2 + 0.4578 \ln r_p + 0.2179 \tag{7}
\]

2.2. Experimental Work

2.2.1. Recombination of Downstream Fluids

The live crude oil sample utilized in this study were collected from downstream system (gas from first-stage separator and dead oil from stock tank) and experimentally recombined at reservoir conditions based on the saturation pressure to reproduce a representative fluids of tight oil reservoir as validated in our previous studies [25], [26]. This study does not explain the recombination related processes, though it represents the reservoir fluid and recombined fluid compositions used in this work in Table 1. The carbon dioxide utilized in this study has a purity > 99.95%. The rest of PVT properties of tight reservoir fluid such as oil viscosity, density, gas-oil ratio and formation volume factor were measured using CMG WinProp as shown in Table 2 resulting in a good correlation with tight reservoir fluid data.

| Comp. | Pc (psi) | Tc (°C) | Mol. Weight | Reservoir Fluid (Mole %) | Recombined Fluid (Mole %) | Average Absolute Error (%) |
|-------|----------|---------|-------------|--------------------------|-----------------------------|---------------------------|
| N\(_2\) | 492.31 | 126.2 | 28.013 | 1.3643 | 1.3843 | 0.0828 |
| CO\(_2\) | 1069.86 | 304.2 | 44.010 | 6.7853 | 6.8817 | 1.4213 |
| C\(_2\)H\(_4\) | 667.19 | 190.6 | 16.043 | 27.034 | 27.426 | 1.4514 |
| C\(_6\)H\(_6\) | 708.34 | 305.4 | 30.070 | 5.5795 | 5.6450 | 1.1733 |
| C\(_8\)H\(_8\) | 615.76 | 369.8 | 44.097 | 5.7348 | 5.7831 | 0.8422 |
| iC\(_9\)H\(_10\) | 529.05 | 408.1 | 58.124 | 1.8855 | 1.8839 | 0.0828 |
| nC\(_9\)H\(_10\) | 551.09 | 425.2 | 58.124 | 3.3868 | 3.3806 | 0.1839 |
| iC\(_{10}\)H\(_{12}\) | 490.84 | 460.4 | 72.151 | 2.6760 | 2.6544 | 0.8066 |
| nC\(_{10}\)H\(_{12}\) | 489.37 | 469.6 | 72.151 | 3.7068 | 3.6693 | 1.0108 |
| C\(_{18}\)H\(_{14}\) | 477.03 | 507.5 | 86.010 | 5.1205 | 5.0579 | 1.2230 |
| C\(_{4+}\) | 145.69 | 905.1 | 274.00 | 36.755 | 36.233 | 1.4193 |

**Table 1. Reservoir fluid and recombined fluid components.**
Table 2. Comparison between PVT properties of tight oil reservoir and predicted properties using WinProp.

| PVT Properties | Sample | Model (Winprop) | Average Absolute Error (%) |
|----------------|--------|----------------|---------------------------|
| Bubble Point (psi) | 2652   | 2651.864       | 0.005130                  |
| Viscosity (cp)    | 0.270  | 0.2698         | 0.074070                  |
| Oil Density (lb/ft³) | 36.849 | 36.900         | 0.138402                  |
| Gas-Oil Ratio (scf/stb) | 76.50  | 76.84          | 0.444444                  |
| Oil FVF (bbl/stb) | 1.317  | 1.319          | 0.151860                  |
| AARE (%)         |        |                | 0.162780                  |

2.2.2. Vanishing Interfacial Tension Test
Vanishing interfacial tension (VIT) is a fast route to calculate the minimum miscibility pressure to economically recover oil during CO₂ injection process. VIT offers a method to directly measure the IFT between the two phases. The experimental schematic demonstration utilized in this work is illustrated in Figure 3. The apparatus comprises of two high pressure and temperature PVT cells and syringe pump (ISCO-260D). The upper cell contains recombined fluid phase and CO₂ phase is injected from syringe pump until a little head of free CO₂ is seen at the top, confirming that the recombined fluid is fully saturated with CO₂ phase. The measurement system is consisting of high pressure syringe pump to inject CO₂ as a solvent into the lower cell at stable desirable temperature. The PVT cell contains a moveable piston to manipulate the pressure inside the cell by pumping CO₂ on to the back of PVT cell as a pressurizing fluid. The valve between both the cells allows us to displace the system, while keeping the upper cell at a relatively higher pressure than the lower cell pressure. As the valve is opened the upper cell system is likely to pump the recombined fluid into the lower cell and once the droplet is formed the valve is closed or the moveable piston is utilized to pressurize and depressurize the system. The IFT measurements are conducted with few test droplets until the system have excess oil at the bottom of the lower cell, this is to achieve a stable droplet at the tip of the capillary tube. As the stabilized droplet of recombined fluid is introduced in the lower cell which is fully saturated with CO₂, in terms the CO₂ instantly swells the droplet which dissolves in the oil, thus significantly extending the volume of the oil phase, leading the lighter components of the oil phase to isolates into the vapor phase and achieve solubility through CO₂ phase. In the process one may achieve slight stability in terms of droplet. Through saturating/presaturating the oil phase with CO₂ (upper cell) and CO₂ phase with the oil (lower cell), some of that mass transfer upon the formation of the droplet at the tip of capillary is removed. However, the lower cell which has two lateral windows for optical observations; one of the lateral windows is utilized for lighting source and the other for camera to detect the shape of droplet and the magnified picture is digitized and stored in the operating system. Afterwards, the digitized droplet analyzes drop shapes through Laplace equation to fit the experimental drop shape for the determination of interfacial tension with the knowledge of density difference (density of the oil saturated with CO₂ at desired pressure and temperature and density of the CO₂ phase saturated with oil at desired pressure and temperature).

\[ \Delta P = \gamma \left( \frac{1}{R_1} + \frac{1}{R_2} \right) \]  

Eq. (8)
3. Results and Discussion

3.1. VIT measured MMP

MMP measurements using VIT technique are done in a high-pressure PVT cell. Crude oil is introduced as a drop phase into the chamber filled with the injected fluid. Pendant drop shape analyses are utilized to calculate the interfacial tension. The pressure is gradually increased by pumping more injection fluid on the back of PVT cell as pressurizing fluid. The interfacial tension is determined at different pressures at reservoir temperature. VIT technique for estimating MMP between reservoir fluid and injected fluid (CO₂) with the nanopore confinement effect of tight reservoir besides the slim-tube test data is validated at the reservoir temperature of 238 °F. The reservoir fluid compositions and critical properties are presented in Table 1. Vanishing interfacial tension measurement for minimum miscibility pressure data for both of live oil (recombined oil) and dead oil (stock tank oil) samples are illustrated in Figures. 4 and 5, respectively. However, it was observed that the live oil MMP estimated using VIT procedure is 3478 psi, which is comparatively higher in comparison with the calculated data of 3316 psi utilizing slim-tube technique with AARE of 4.86%. The close agreements designate that VIT procedure can precisely predict the MMP of the reservoir fluid in confined phase. The measurements are done at 9 different pressures, after which the line is extrapolated to zero IFT value. In contrast, stock tank oil (dead oil) MMP determination using VIT was 1821 psi, whereas ST measurement was 2376 psi with AARE of 23.36%. It is noted that a huge deviation between VIT and ST data occurred due to heavier fractions of dead oil. Thus, light fractions of dead oil are isolated into CO₂ gas phase, considered too low to be enriched for solubility with crude oil. In case of stock tank oil with high stabilized heavier fractions, this difference can be correlated to lack of multiple contact miscibility development between crude oil and gas phases compared to achieved miscibility in slim-tube case at respective injection pressure.

In order to consider the capillary pressure effect on the minimum miscibility pressure, the MMP is calculated with different pore sizes. The MMP for the pore size 5 nm is 2810 psi, whereas MMP increased to 3104 psi with the pore size of 10 nm. Therefore, the minimum miscibility pressure reduces as the pore size is reduced. Thus, the reservoir fluid and injected fluid (CO₂) will achieve miscibility at lower pressures and subsequently is helpful for CO₂ EOR. Figure 6 shows the determined IFTs and MMP considering capillary pressure effect for tight oil reservoirs.
3.2. Correlation calculated MMP
A CO₂-Oil MMP can be measured in several methods and the correlation provides one of the easiest and attractive solutions to estimate MMP. Though, the correlations are used in certain conditions, however they are yet to be considered as an efficient method to roughly determine CO₂-Oil MMP. Mostly, the MMP correlations are function of the few parameters including reservoir temperature, molecular weights and mole fractions. Most of CO₂ MMP correlations are summarized in Table 3.

Table 3. Common CO₂ MMP Correlations.

| Author          | Correlation                                           |
|-----------------|-------------------------------------------------------|
| Holm & Josendal | Graphical correlation as shown in Figure 2.           |
| Cronquist       | \[ \text{MMP} = 16T^{(0.744+0.0015X_{\text{vol}}+0.0011MW_{C5+})} \] |
Orr & Jensen

\[ MMP = 0.101386 \exp \left[ 10.91 - \frac{2015}{255.372 + 0.5556(1.8T_R + 32)} \right] \]

Emera & Sarma

\[ MMP = 5.0093 \times 10^{-5}(1.8T_R + 32)^{1.164} \left( \frac{MW_{c5+}}{X_{\text{vol}} / X_{\text{int}}} \right)^{0.1073} \]

Yelling & Metcalfe

\[ MMP = 12.6472 + 0.01553(1.8T_R + 32) + 1.24192 \times 10^{-4}(1.8T_R + 32)^2 - \frac{716.9427}{(1.8T_R + 32)} \]

Shokir

\[ MMP = -0.068616Z^3 + 0.31733Z^2 + 4.9804Z + 13.432 \]

Alston

\[ MMP = 0.000878T^{1.06} \left( \frac{MW_{c5+}}{X_{\text{vol}} / X_{\text{int}}} \right)^{0.136} \]

Lee

\[ MMP = 7.3924 \times 10^{-2.772} \left[ \frac{1519}{(1492 + 1.8T_R)} \right] \]

Glaso

\[ MMP = 180 - 3.404MW_{c5+} + \left( 1.7 \times 10^{-9}MW_{c7+}^{3.730} e^{7.868MW_{c7+}^{1.058}} \right) T \]

Shengli

\[ MMP = 675.01 - 6.9MW_{c6+} + 1.7(T - T_{\text{critical}}) \]

Yuan

\[ MMP = a_1 + a_2MW_{c7+} - a_3X_{\text{int}} + \left( a_4 + a_5MW_{c7+} + a_6 \frac{X_{\text{int}}}{MW_{c7+}} \right)(1.8T_R + 32) + \left( a_7 + a_8MW_{c7+} - a_9MW_{c7+}^2 - a_{10}X_{\text{int}} \right)(1.8T_R + 32)^2 \]

Recombined crude oil sample shown in Table 1 is used to validate the CO₂-Oil MMP correlations. Results in Table 4 are shown based on the correlation parameters. In general, the correlations involving molecular weights and temperature give a good prediction of CO₂-Oil MMP. The correlations only taking temperature into consideration significantly deviates from the results provided by the Winprop simulator. On the contrary results of MT and MTF correlations are similar but the equations become more complex if the crude oil composition is involved in the correlations.

**Table 4. CO₂–Oil MMP correlation validation for each parameter.**

|                         | Recombed Fluid (Live Oil) | Stock Tank Oil (Dead Oil) |
|-------------------------|---------------------------|--------------------------|
| Temperature (MMP in psi) | 2892.052                  | 2240.832                 |
| Temp. MW (MMP in psi)   | 3250.295                  | 2630.984                 |
| Temp., MW & MF (MMP in psi) | 3150.219             | 2570.068                 |
| CMG Winprop (MMP in psi) | 3319.913                  | 2619.381                 |
Afterwards, CO₂ MMP is predicted with a confinement effect at 20nm and 10nm pore radii as shown in Table 5. The correlation proposed by Zhang K., [11] gives a reasonable prediction of CO₂-Oil MMP, which also incorporates the alteration of CO₂–Oil MMP with nanoscale pore confinement. Comparison between experimental MMP and calculated MMP ITF values shown in Figure 7 suggest that the results from the correlation considering confinement effect have good coincidence which measured values.

| Correlation (psi) | CMG (psi) |
|-------------------|-----------|
| r = 20 nm         | r = 20 nm |
| r = 10 nm         | r = 10 nm |
| Recombined Fluid  | 3343.118  | 3505.561 |
| Stock Tank Oil    | 2214.726  | 2407.626 |
|                   | 2968.154  | 3084.046 |
|                   | 1849.760  | 2086.213 |

Figure 7. Calculated MMP IFT by confined correlation vs. experimental MMP IFT.

4. Conclusion
The VIT technique is efficient, less expensive and a reproducible technique in determining the MMP compared to irreproducible, most expensive and time consuming ST procedure. VIT test needs at most one day to estimate one MMP, whereas slim-tube requires a minimum of two weeks for one MMP determination. For recombined crude oil, VIT estimated MMP was found significantly good in contrast to slim-tube determined MMP that validates the multiple contact miscibility between recombined fluid and injected CO₂ gas. While in case of dead oil sample, higher deviation was noticed between ST and VIT estimated MMPs. This may be due to deficiency of multiple contacts miscibility and stabilization of crude oil heavier fractions.

With reference to common CO₂–Oil MMP correlations, it was observed that for both of the crude oil samples (recombined oil and dead oil samples), no correlation is able to calculate MMP in close consent with VIT and ST determined MMP. It emphasizes the correlations validity only for certain number of crude samples considered during correlations development. Therefore, most of the common CO₂–Oil MMP correlations are validated by Winprop simulator. Subsequently, a solubility depended technique is proposed and confirmed to predict the CO₂–Oil MMP considering the modifications by nanopore confinement. Commonly, the correlations with molecular weights and reservoir temperature can give a rough estimation of CO₂–Oil MMP. For tight oil reservoirs, taking confinement effect into
account, CO₂-Oil MMP is reduced. This study entails the combination of solubility techniques and the nanopore confinement effect that provide an efficient method for MMP calculations.

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