

**EVALUATION OF FOAM SUPER-CRITICAL CO₂ INJECTION IN
THE COMPOSITE LIMESTONE CORE USING NEW MULTIPLE
FRACTURE SYSTEM**

BY

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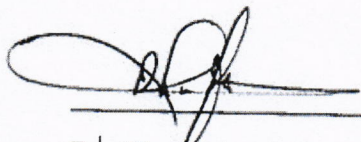
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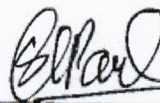


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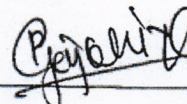


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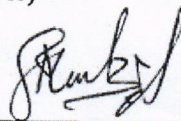
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[To my great father, to my amazing mother, to my dear family]

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LIST OF ABBREVIATIONS

EOR	:	Enhanced Oil Recovery
OOIP	:	Oil Originally in Place
SAG	:	Surfactant-Alternating-Gas
WOR	:	Water-Oil-Ratio
AOS	:	Alpha-Olefin-Sulfonate
MMP	:	Minimum Miscibility Pressure
Swi	:	Initial Water Saturation
Sor	:	Residual Oil Saturation
OIIP	:	Oil Initially in Place
WAG	:	Water-Alternating-Gas
NMR	:	Nuclear Magnetic Resonance
CT	:	Computerized Tomography

ABSTRACT

Full Name : [JABER ALI AL-SHUQAYYIQI]
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[CO₂ flooding reduces residual oil saturation, which leads to producing substantial amounts of oil that were not producible by other tertiary recovery means. A significant failing of this gas-driven oil recovery method is gas channeling and phase segregation due to the great gas mobility and high-density differences between oil and injected gas. Even though CO₂ is a supercritical fluid at reservoir conditions, its density is still lower than oil density and its mobility greater than oil mobility. The foam was used to solve the mobility issue and resulted in an increased oil recovery and a reduced gas mobility ratio at a laboratory scale. However, this increase in the recovery factor is not usually achieved at the reservoir due to fracture system's complexity. This work intends to examine the foam sweep efficiency using a new fractured system configuration at high pressure and high-temperature condition.

In this research, a novel approach was developed to quantify oil recovery from each composite core plug. This novel approach integrates X-ray computerized tomography (CT) scans and nuclear magnetic resonance (NMR) diffusion which estimates the individual oil recoveries added to the total measured recovery as shown in Table 5 and Figure 44. This approach was able to detect the density override in the composite core. The new approach

facilitates the composite core flooding analysis by allowing researchers to have more details about the residual oil distribution.

Six core flooding experiments were conducted which investigated several enhanced oil recovery (EOR) schemes that involve secondary recovery, CO₂ foam flooding and surfactant-alternating gas (SAG). The Alpha-Olefin Sulfonate (AOS) surfactant was used to generate the foam. Three experiments investigated the CO₂ foam flooding with different foam qualities and SAG without water flooding. The results showed that the CO₂ foam flooding reached the ultimate recovery faster than the SAG flooding. However, SAG showed the ability to overcome the gravity override issue. The other three experiments with flooding by seawater then investigated the same CO₂ foam flooding schemes. These experiments showed that the recovery of CO₂ foam flooding with 80% foam quality was faster than the recovery of CO₂ foam flooding with 70% foam quality.

]

ملخص الرسالة

الاسم الكامل: جابر علي أحمد الشقيقي

عنوان الرسالة: تقييم حقن ثاني أكسيد الكربون الفائق بالرغوة في الصخور الجيرية بمحاكاة التصدعات الطبيعية

التخصص: هندسة البترول

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يقلل غمر ثاني أكسيد الكربون من تشبع الزيت المتبقي ، مما يؤدي إلى إنتاج كميات كبيرة من النفط التي لم تكن قابلة للإنتاج بوسائل الاسترداد الثلاثية الأخرى. يتمثل أحد أوجه القصور الكبيرة في طريقة استخلاص الزيت التي يحركها الغاز في توجيه الغاز وفصل الطور بسبب الحركة الكبيرة للغاز والاختلافات عالية الكثافة بين الزيت والغاز المحقون. على الرغم من أن ثاني أكسيد الكربون عبارة عن سائل فوق حرج في ظروف الممكن ، إلا أن كثافته لا تزال أقل من كثافة الزيت وحركته أكبر من حركة الزيت. تم استخدام الرغوة لحل مشكلة التنقل وأدت إلى زيادة استخراج النفط وتقليل نسبة تنقل الغاز على نطاق معمل. ومع ذلك ، فإن هذه الزيادة في معامل الاسترداد لا تتحقق عادة في الخزان بسبب تعقيد نظام الكسر. يهدف هذا العمل إلى فحص كفاءة اكتساح الرغوة باستخدام تكوين نظام تكسير جديد في ظروف الضغط العالي ودرجات الحرارة المرتفعة.

في هذا البحث ، تم تطوير نهج جديد لتحديد مقدار استخلاص الزيت من كل المكونات الأساسية المركبة. يدمج هذا النهج الجديد التصوير المقطعي المحوسب بالأشعة السينية (CT) وانتشار الرنين المغناطيسي النووي (NMR) الذي يقدر عمليات استرداد الزيت الغريني المضافة إلى إجمالي الاسترداد المقاس كما هو موضح في الجدول 5 والشكل 44. كان هذا النهج قادرًا على اكتشاف الكثافة تجاوز في النواة المركبة. يسهل النهج الجديد تحليل الفيضان الأساسي المركب من خلال السماح للباحثين بالحصول على مزيد من التفاصيل حول توزيع الزيت المتبقي.

تم إجراء ست تجارب غمر أساسية بحثت في العديد من مخططات الاستخلاص المعزز للنفط (EOR) التي تتضمن الاسترداد الثانوي ، وغمر رغوة ثاني أكسيد الكربون والغاز السطحي المتناوب (SAG). تم استخدام الفاعل بالسطح Alpha-Olefin Sulfonate (AOS) لتوليد الرغوة. فحصت ثلاث تجارب غمر رغوة ثاني أكسيد الكربون بخصائص رغوية مختلفة و SAG بدون غمر المياه. أظهرت النتائج أن غمر رغوة ثاني أكسيد الكربون وصل إلى

الانتعاش النهائي بشكل أسرع من فيضان SAG. ومع ذلك ، أظهرت SAG القدرة على التغلب على مشكلة تجاوز الجاذبية. التجارب الثلاث الأخرى مع الفيضانات بمياه البحر ثم فحصت نفس مخططات غمر رغوة ثاني أكسيد الكربون. أوضحت هذه التجارب أن استعادة رغوة ثاني أكسيد الكربون المغمورة بجودة رغوة بنسبة 80% كانت أسرع من استعادة رغاوي ثاني أكسيد الكربون المغمورة بجودة رغوة 70% [

CHAPTER 1

INTRODUCTION

The main resources of energy worldwide are oil and gas for which the demand is continuously growing. However, most conventional petroleum reservoirs are already drained by the depletion drive mechanism or secondary drive mechanism such as water flooding. Petroleum production has reached the ultimate recovery through these drive mechanisms. Therefore, the focus is now on developing and investigating new techniques that can extract residual oil that was not recoverable by conventional methods. The new techniques are known as Enhanced Oil Recovery techniques (EOR). EOR methods are mainly implemented in the oil reservoir after primary and secondary driving method deployment, or whenever these methods are not financially feasible. Generally, the primary and secondary recovery mechanisms' ultimate recovery is from 20% to 50% of the initial oil in place (IOIP), depending on reservoir rock and fluids properties [1]. EOR methods are generally divided into three main categories: chemical, thermal, and gas injection methods. The chemical methods are aimed to change the reservoir rock properties to increase oil mobility or alter the wettability. The thermal methods are aimed to induce heat to the reservoir to reduce heavy oil viscosity. The gas injection methods are aimed to improve the overall oil displacement by injecting gases into the reservoir such as nitrogen, hydrocarbon gases, or carbon dioxide. CO₂ injection is one of the most effective methods to recover residual oil from depleted reservoirs. It has several properties that favor its

application among other gases. Carbon dioxide is a solvent for mobilizing and producing residual oil left behind after flooding the reservoir with water flooding. At the reservoir pressure and temperature conditions and oil composition, carbon dioxide can achieve miscibility with oil, which eventually reduces oil viscosity and cause the oil to swell and lower its interfacial tension. Also, carbon dioxide is available in large quantities either in nature or in industrial facilities as a byproduct.

Moreover, with the increasing concern with green-house gas emissions and its role in causing global warming, many attempts were made to capture carbon dioxide and store it underground by implementing it in EOR projects. Despite the features of CO₂ injection, it still has some limitations that reduce its success in recovering the remaining oil saturation. CO₂ has higher mobility than the oil in the porous media because of its lower viscosity. This unfavorable mobility ratio leads to viscous fingering, gas channeling, and CO₂ overriding, which results in an early gas breakthrough in the producers. Ultimately, the reservoir is partially swept with CO₂ flooding, which results in poor volumetric sweep efficiency. The issue of viscous fingering is more severe if the reservoir is heterogeneous. This unfavorable mobility ratio could be reduced by using foam with the injected gas. Gas breaks into bubbles separated and stabilized by the surfactant. The foam causes a reduction in gas mobility, which is essential for improving the volumetric sweep efficiency during immiscible or miscible gas flooding of oil reservoirs. For the foam to be generated in the porous media, gas and surfactant are injected simultaneously or alternatively at three specific conditions. Foam has other applications in the petroleum industry besides acting as an EOR mobility control agent, such as acid diversion, gas-shutoff, and water-shutoff [2]. This thesis aims to investigate foam supercritical (SC)-CO₂ injection for enhanced oil

recovery in fractured limestone cores through experimental laboratory work with several flooding modes. The injection modes are continuous SC-CO₂ with surfactant, surfactant alternating gas (SAG), and seawater flooding, followed by the two former modes. This thesis introduces a new core composite model with multiple horizontal and vertical fractures. This work investigates the effect of foam quality (using AOS Alpha-Olefin Sulfonate surfactant) on the sweep efficiency, SC-CO₂ mobility, and gravity segregation at high-pressure, high-temperature conditions (3200 psi and 90 C) using fractured limestone cores. In addition, CT scans and NMR are integrated to estimate the residual oil saturation the ultimate recovery of each plug of the composite core. The thesis is organized as follows: Chapter 2 is the literature review of the EOR methods that involve carbon dioxide and foam generation, the introduction of foam into EOR, and foam's behavior in lab tests and field applications. Chapter 3 explains the objectives behind conducting this research and identifies the challenges of using foam in EOR. Chapter 4 explains, in detail, the methodology, equipment and materials, and work strategy employed to conduct this research. Chapter 5 illustrates all the results of the experiments accompanied by a thorough analysis and discussion. Chapter 6 presents the conclusions and recommendations for future research in this area of study.]

CHAPTER 2

LITERATURE REVIEW

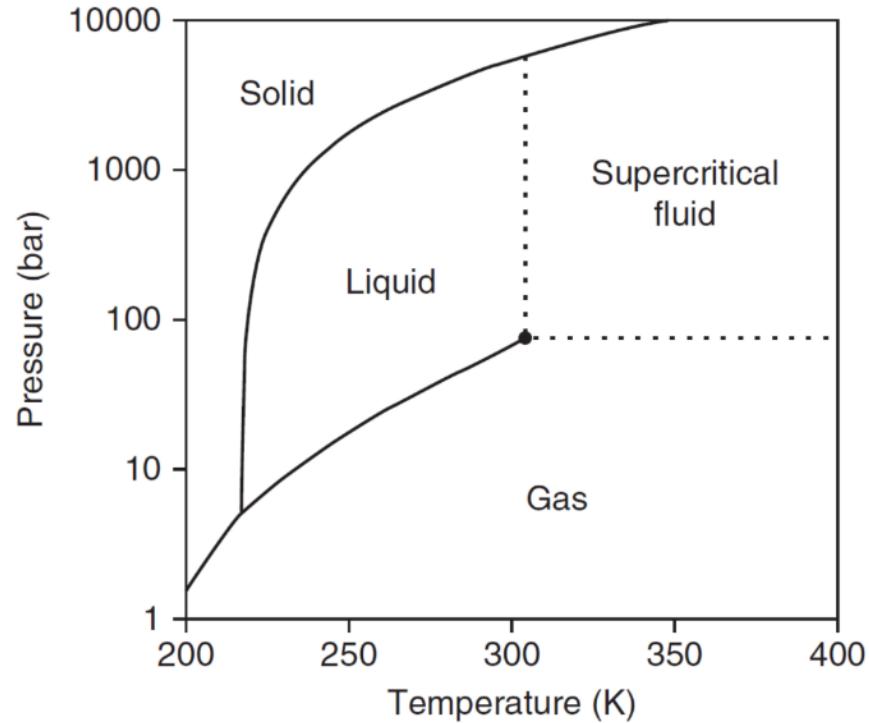
2.1 Carbon Dioxide Flooding

CO₂ flooding has improved several oil reservoirs' recoveries by recovering an additional 15% to 25% of the initial oil in place [2], [3]. The oil displacement by CO₂ is classified as miscible or immiscible displacement. The governing factor in determining the type of displacement is the minimum miscibility pressure (MMP). Miscible displacement occurs at reservoir pressures above MMP. Immiscible displacement takes place at reservoir pressures less than MMP [4]. CO₂ miscible flooding increases the reservoir recovery through three mass transfer mechanisms: diffusion, solubility, and dispersion [5], [6].

2.2 Advantages of Carbon Dioxide

CO₂ and reservoir oil can achieve miscibility at lower pressure compared to other gases. This enhances the microscopic sweep efficiency. Theoretically, CO₂ can displace oil completely at a microscopic scale. CO₂ is denser than other injection gases at high pressures and temperatures, which reduces the tendency of gas overriding during flooding compared to other hydrocarbon gases. Figure 1 shows the phase diagram of CO₂. It shows that at 1067.5 psi and 87.7 °F, CO₂ is a critical fluid, which means at typical reservoir conditions, CO₂ density is high which can potentially reach the density of some types of reservoir oil. CO₂ can exhibit properties between gases and liquids. Its density is similar to

a liquid, and its viscosity similar to gas. These properties mitigate gravity segregation and gas fingering, leading to a stable displacement front.



3

Figure 1: CO₂ pressure - temperature phase diagram (Balaban & Ferrentino, 2012)

2.3 Disadvantages of Carbon Dioxide

There are some limitations to using CO₂ as displacing fluid. CO₂ has a low volumetric sweep efficiency because of its high mobility ratio compared to reservoir crude oils. CO₂ is more movable than oil due to CO₂ low viscosity, which leads to viscous fingering and a quick CO₂ breakthrough. The low macroscopic sweep efficiency means that the injected CO₂ bypasses the reservoir fluid without being displaced or getting in contact. Due to the high difference between CO₂ and reservoir oil densities, gravity segregation occurs in the reservoir [8]. This issue is more severe in fractured and high vertical permeability reservoirs. To overcome these issues, multiple techniques were introduced to enhance the

macroscopic sweep efficiency of CO₂ injection. Water alternating gas (WAG) was used as an injection scheme to improve displacement efficiency. The main advantage of WAG is combining the enhanced macroscopic displacement of water and with enhanced microscopic displacement of CO₂. This injection scheme reduced the CO₂ mobility resulting in improved oil recovery. However, WAG did not completely solve the unfavorable mobility ratio issue. Other solutions tried to improve the overall sweep efficiency by using surfactants to generate foam, which controls gas mobility.

2.4 Miscible Displacement

Miscible displacement has successfully increased the oil recovery factor over the last decades by improving the microscopic displacement efficiency [9]. Miscibility is the ability of two or more fluids to be mixed at different proportions while interfacial tension (IFT) does not exist between the fluids [10]. Miscible displacement reduces the residual oil saturation to zero in the flooded region. Miscible displacement has two types: first contact and multi-contact miscible displacements. The first contact miscibility (FCM) is defined as the process at which the injected solvent will have existed as a single phase with oil. Several light and heavy hydrocarbons have been utilized as solvents in first contact miscibility processes. Multi-contact miscible (MCM) displacement is divided into two mechanisms: condensing gas drive and vaporizing gas drive. A condensing gas drive requires an injection of an enriched gas that contains heavy hydrocarbon components. The enriched gas transfers the heavy components to the oil. Once the oil reaches the enrichment, it would be miscible with purely injected gas. Vaporizing gas drive requires a lean gas to be injected into the reservoir. As the lean gas flow through the reservoir, it vaporizes through liquefiable petroleum gas (LPG) components from the oil. When displacing gas

front vaporized adequate hydrocarbon amounts, it will be miscible with virgin reservoir oil. The lean gases used in this process are methane, nitrogen, or flue gas. When CO₂ is injected, a similar mechanism takes place. CO₂ usually is liquid or critical fluid as reservoir conditions that allow CO₂ to extract heavier hydrocarbons from the oil, making the displacement front miscible at lower miscibility pressure. In both MCM mechanisms, hydrocarbon transfer between reservoir oil and flooding fluid is required [10].

2.5 Foam Generation

Foam has three characteristics that led to its use in the enhanced oil recovery techniques (1) Foam works as a diverting agent to achieve uniform flooding displacement and enhance sweep efficiency; (2) Foam can reduce gas mobility to achieve a favorable mobility ratio between the injected gas and the reservoir fluid; and (3) foam can reduce the override problems due to the density difference in gas flooding [11]. Foam is generated mainly through three methods; (1) Pre-generated foam is generated before injection into porous media. It is formed either inside the tubing before reaching or at the surface; (2) Co-injection foam is generated while injecting both gas and surfactant solution together; and (3) Surfactant-Alternating-Gas (SAG) foam is a foam formed by alternating surfactant and gas injection [12], [13]. In all mentioned foam generation methods, the surfactant is the main component to form the foam.

2.6 Foam Injection in Fractured Carbonate Cores

Designing a representative fractured core system is important to simulate the fractured reservoir system. The majority of foam flooding experiments in fractured cores consider a horizontal fracture along the length of the core, as shown in Figure 2. In Figure 2, A Darvish

et al. tried to simulate fractures by designing a core holder with a 2 mm gap around the core [14]. The main objective is diverting injected fluids from the fracture into the matrix [13]. This fracture configuration does not represent the fractured reservoir's nature and does not account for the gravity segregation. Fractured reservoirs usually have a fracture network consisting of mainly vertical fractures and little horizontal fractures [15]. Haugen et al. studied the effect of in-situ foam generation and pre-generated foam injection on the oil recovery factor of artificially fractured cores. Edwards's limestone core sample was artificially fractured to generate horizontal fracture, and the core is shown on the right side of Figure 3. Edward's limestone samples were assembled to build a block with a fracture network, as shown on the left side of Figure 3. The rock porosity and permeability are 19% and 6 mD, respectively. An Alpha-Olefin Sulfonate surfactant (AOS) was used as a foaming agent. The flooding experiments were conducted at ambient conditions (1 atm and 23°C). The in-situ foam injection recovered 0.2% and 0% of the OOIP (original oil in place) of the core and block, respectively. However, pre-generated foam injection recovered 63% and 75% of the OOIP of the core and block, respectively. The foam quality was 91% in both experiments. The pre-generated foam injection was 33 mL/h and 66 mL/h for the core and the block. The total pre-generated foam volume is 139 PV in the fractured core. Figure 4 shows that the first 30 PVs recovered 50% of OOIP, and the remaining 109 PVs recovered 13% of OOIP. Figure 5 shows that the oil recovery rate is changing, which means that the foam is diverting the injected fluid from the fracture to each piece of the fractured block. The first 12 PVs of the pre-generated foam produced 25% of OOIP. The following 12, 17, 72, and 81 PVs produced 25%, 15%, 25% and 10%, respectively. In this study, the experimental conditions were at ambient conditions that do not represent the reservoir

conditions where temperature and pressure are higher. In addition, the number of injected PVs is high, where it cannot be injected in the reservoir. These experiments do not show the behavior of the injected fluid inside the core [19].

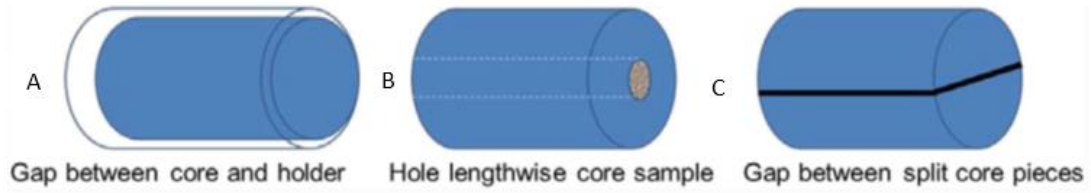


Figure 2: Schematic of fractured core systems (Dong et al., 2017)

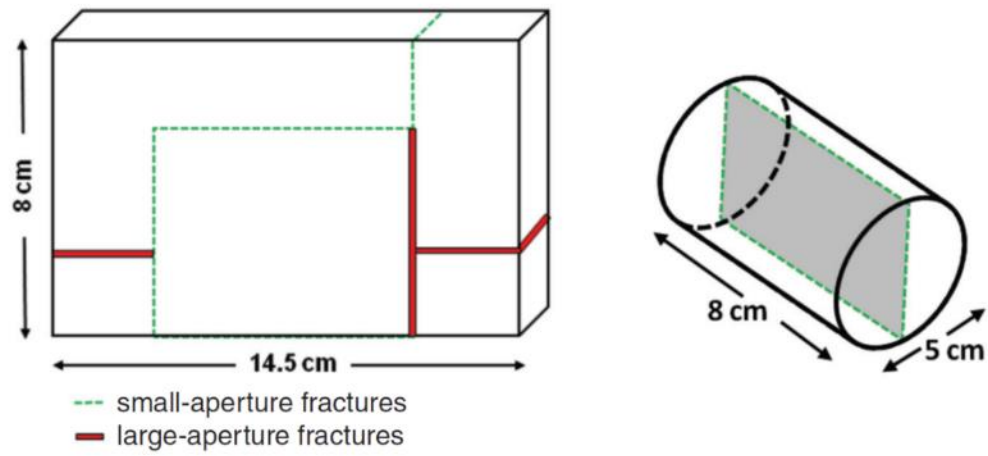


Figure 3: Schematics of the fractured core and block (Hciugen et al., 2017)

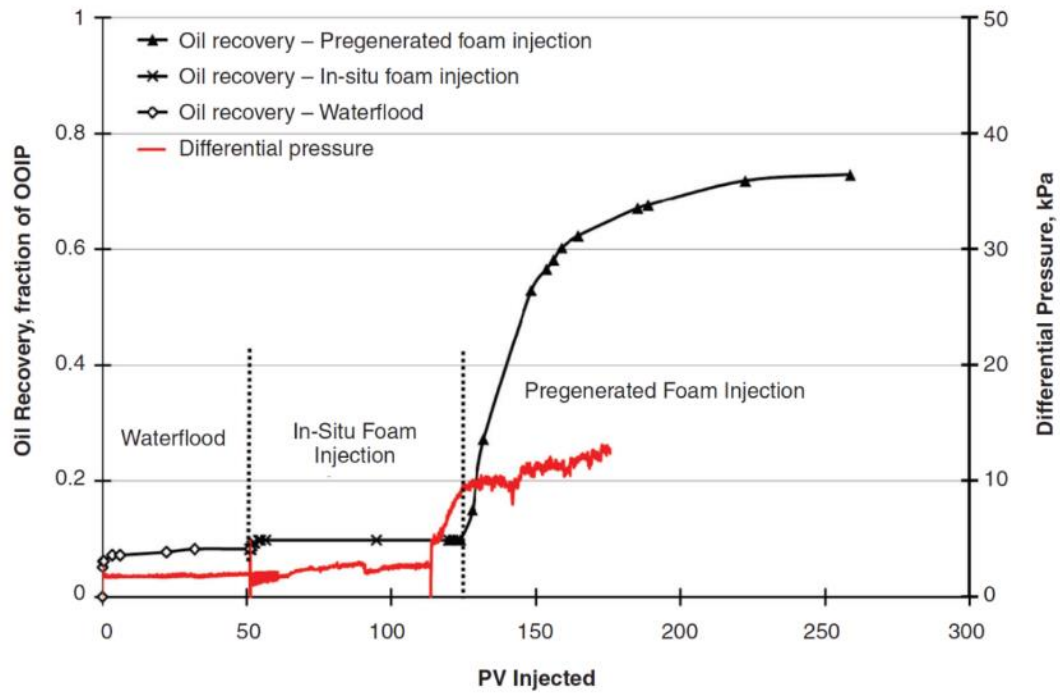


Figure 4: Differential pressure & recovery of limestone core (Hciugen et al., 2017)

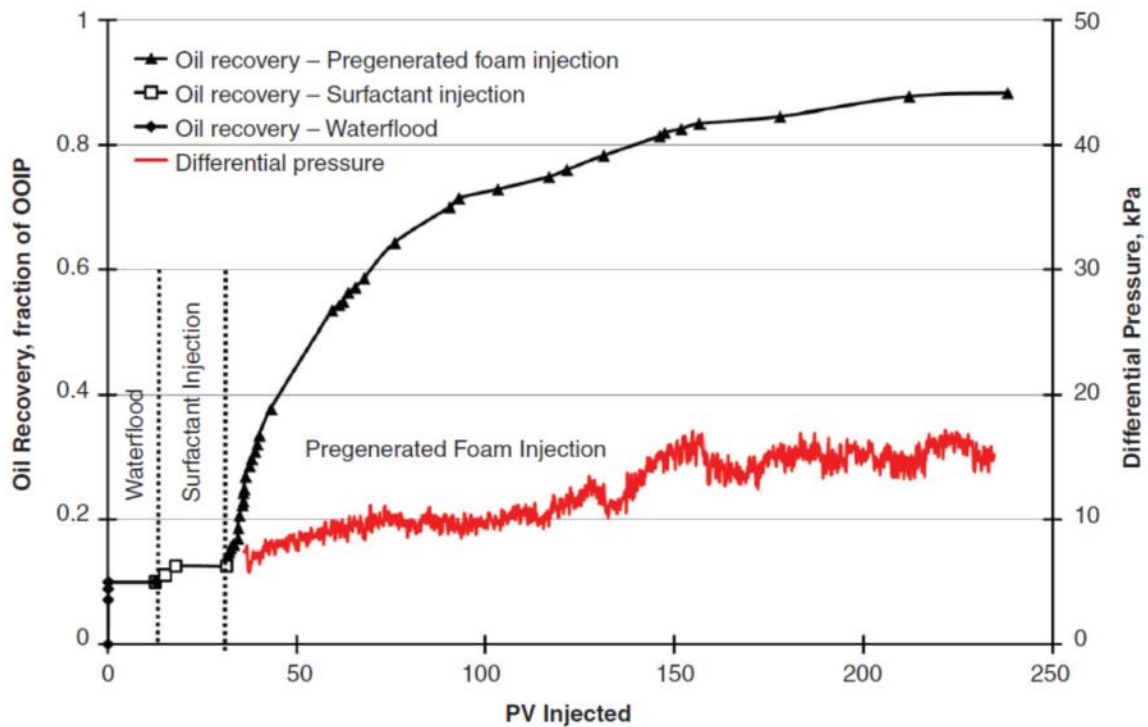


Figure 5: Differential pressure & recovery of limestone block (Hciugen et al., 2017)

Langlo's study showed a recovery factor of 90% on a fractured limestone rock flooded by foam CO₂. Figure 7 shows that the maximum recovery factor was achieved after injecting three pore volumes. Figure 6 showed the fractured core with the plastic spacer to simulate the fracture. As shown in Figure 6, the plastic spacer is divided into two squares, which means that the fracture is isolated, and the injected fluid has to flow through rock only. The fracture configuration in this work does not represent the reservoir's nature.



Figure 6: Fractured Limestone Core (Langlo, 2013)

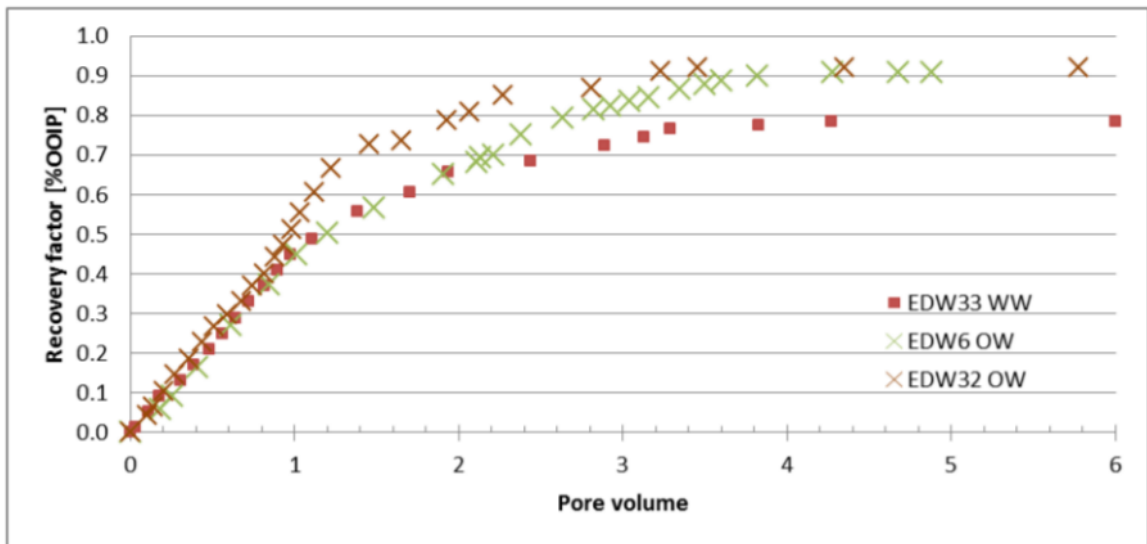


Figure 7: Recovery Factor of Three Fractured Limestone Cores (Langlo, 2013)

2.7 Foam Quality

Injecting foam with SC-CO₂ overcomes the frontal instability issue caused by unfavorable mobility ratios and gravity override [21]. Using foam with SC-CO₂ increases the viscosity and decreases the foam velocity. These two effects control the mobility of the foam. The foam quality dominates foam viscosity. The foam quality is defined as the ratio of gas to liquid [22], and is empirically correlated to the foam viscosity, where μ_f is foam viscosity, μ_g is gas viscosity, and Γ is foam quality. Foam viscosity is directly proportional to foam quality, as shown in equation 1. However, the optimal foam quality is 75% to generate stable foam [23].

$$\mu_f = \mu_g(1 + 3.6\Gamma) \quad (1)$$

Two types of foaming agents to generate high viscous foam were studied [21]. Table 1 shows one foaming agent's components, Alpha Olefin Sulfonate (AOS). The foaming agent's apparent viscosity was measured at different rates and 212°F, as shown in Figure 8. The apparent viscosity was measured for three different foam qualities 50/50, 70/30, and 80/20. The highest foam viscosity was foam at 70/30 foam quality.

Table 1: Foaming agent components

Alpha Olefin Sulfonate (wt%)	Isopropyl alcohol (wt%)	Citrus Terpenes (wt%)
10-30	1-5	1-5

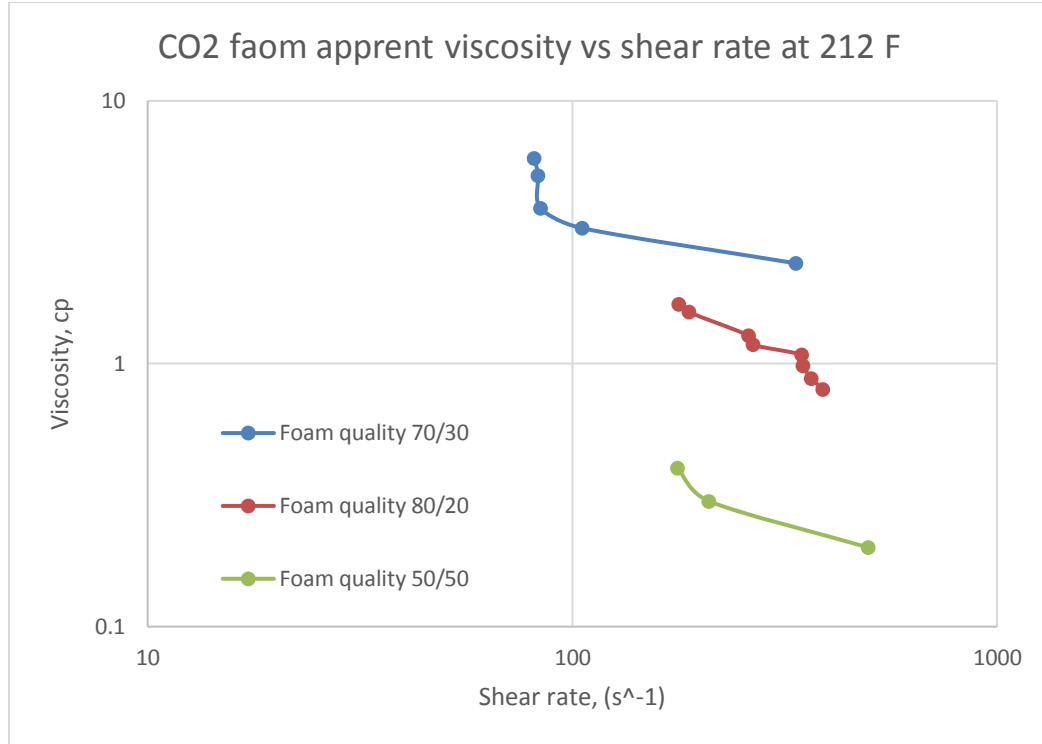


Figure 8: Apparent viscosity vs shear rate at 212 °F

2.7 Determination of multi-phase saturations

Several methods are proposed to estimate three-phase saturations using CT scan imaging. These techniques can be categorized into four major groups: one immobile phase method, matched CT-fluids methods, linear interpolation methods, and dual-energy scan methods.

2.7.1 One immobile phase

This technique assumes that one phase is immobile, and its value is constant throughout the core. With this assumption, the CT number of the immobile phase is included in the rock CT number in equation 4. Thus, it is assumed that only changes in the remaining phases' saturation alter the CT values. The mobile phases' saturation can be obtained by implementing the two-phase CT saturation methods given by equations 2 to 4.

$$CT_{owr} = (1 - \phi)\mu_r + \phi S_o \mu_o + \phi S_w \mu_w \quad (2)$$

$$1 = S_o + S_w \quad (3)$$

$$S_o = \frac{CT_{wr} - CT_{owr}}{CT_{wr} - CT_{or}} \quad (4)$$

2.7.2 Linear regression

In this method, a two-phase flood is conducted until irreducible water saturation (S_{wirr}) is achieved, and a scan (CT_{oirr}), is taken at this saturation. A three-phase flow is initiated, and the water and oil saturations are obtained using equations 5 and 6, assuming that the saturations are linearly related to CT numbers. The remaining saturation can be obtained by material balance. This method was used by Siddiqui et al. (1996) to obtain three-phase saturations of water, benzyl alcohol, and decane.

$$S_w = S_{wirr} + \left(\frac{CT - CT_{oirr}}{CT_{wr} - CT_{oirr}} \right) (1 - S_{wirr}) \quad (5)$$

$$S_o = \left(\frac{CT_{wr} - CT}{CT_{wr} - CT_{oirr}} \right) (1 - S_{wirr}) \quad (6)$$

CHAPTER 3

PROBLEM STATEMENT AND RESEARCH OBJECTIVE

3.1 Problem Statement

Since the 1950s, different gases were used for crude displacing purposes [24][25]. CO₂ reduces residual oil saturation, which leads to producing substantial amounts of oil that were not producible by other tertiary recovery means. A significant failing of this gas-driven oil recovery method is gas channeling and phase segregation due to great gas mobility and high-density differences between oil and injected gas [26]. Even though CO₂ is a supercritical fluid at reservoir conditions, its density is still lower than oil density, and its mobility greater than oil mobility. The foam was used to solve the mobility issue, increasing the recovery factor up to 90%, and reducing the gas mobility ratio at the laboratory scale. However, this increase in the recovery factor is not usually achieved at the reservoir due to the fracture system's complexity. This work intends to examine the foam sweep efficiency using a new fractured system configuration at high pressure and high-temperature conditions.

3.2 Objectives

This thesis aims to investigate foam SC-CO₂ injection for enhanced oil recovery in fractured limestone cores through experimental laboratory work with several flooding modes. The injection modes are continuous SC-CO₂ with surfactant, surfactant alternating gas (SAG), and seawater flooding, followed by the two former modes. This thesis

introduces a new core composite model with multiple horizontal and vertical fractures. This work investigates the effect of foam quality (using AOS surfactant) on the sweep efficiency, SC-CO₂ mobility, and gravity segregation at high-pressure and high-temperature conditions (3200 psi and 90 °C) using fractured limestone cores. In addition, CT scans and NMR are integrated to estimate the residual oil saturation and the ultimate recovery of each plug of the composite core.

CHAPTER 4

Methodology

4.1 Materials

4.1.1 Salts

Formation and sea water were prepared synthetically using the following mineral salts: sodium bicarbonate, magnesium chloride, sodium chloride, sodium sulfate, and calcium chloride.

4.1.2 Core samples

Pink desert and Indiana limestone cores were used to conduct all core-flooding experiments. The diameter of core samples was 1.5 inches. The core plug's length was 6 inches for the whole core and is 2 inches for the composite model.

4.1.3 Oil and gas

In this thesis, light dead crude oil was used to saturate all core samples. The density of the oil was 0.85 g/ml or 35 API at 24°C. Carbon dioxide was used in core-flooding experiments as injection fluid and was injected at the supercritical phase.

4.1.4 Surfactant and chemicals

Alpha Olefin Sulfonate was used as a foaming agent, AOS concentration was 35% pure AOS, and the remaining 65% was water. The injected surfactant solution consisted of 0.012% of AOS surfactant by weight and 99.988% seawater.

4.2 Core-flooding experimental procedure

4.2.1 Brine preparation

Formation and seawater were prepared for the experiments. The cores were saturated with formation water and were flooded with seawater and foaming agents. Table 2 shows the composition of the formation and seawater. Salts were added together to distilled water. Then, they were stirred for two hours to ensure that all salts were dissolved into the water. After that, the brine solution was filtered by membrane filtered paper. The brine salinity of the formation and seawater is 254 and 68 total dissolved salts (TDS).

Table 2: Composition of formation and seawater

Salts	Formation Water (g/liter)	Seawater (g/liter)
Sodium chloride (NaCl)	157.18	41.17
Calcium chloride ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$)	85.62	2.39
Magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$)	10.60	17.64
Sodium sulphate (Na_2SO_4)	0.37	6.34
Sodium bicarbonate NaHCO_3	0.11	0.17
Total dissolved salts (TDS)	253.88	67.71

4.2.2 Core drying

The cores were dried by placing them in an oven at a temperature of 50°C for 8 hours to evaporate trapped moisture. After that, the dry weight of the core was measured. This step is important to determine the dry weight of the rock grain. The core sample was saturated

with formation water in the later stage, and wet weight was measured to estimate the core pore volume and porosity.

4.2.3 Core water saturation

The core plug was placed in a high-pressure cell to saturate the core with the formation water. Firstly, the air from the core in the high-pressure cell was vacuumed using the vacuum pump for around ten hours. Then, the formation of brine was injected into the cell until the core was completely immersed in the brine. After that, the cell pressure was increased to 2500 psi. Then, the core was kept in the cell for 18 hours to let the formation water be imbibed into the pores. The cell pressure dropped slightly to 1400 psi, which indicates that the formation of water was imbibed into all pores. After saturation, the core sample wet weight was measured to estimate the core pore volume and porosity.

4.2.4 Centrifuge oil saturation

All core plugs were placed in a centrifuge machine and were centrifuged at different rotating speeds. The rotating speed started from 1,000 rpm, then increased by 1,000 rpm increment each hour until 10,000 rpm. After that, the core plugs were rotating at 10,000 rpm for 24 hours to ensure that the irreducible water saturation is reached. After finishing the centrifuge process, the amount of the produced water was measured to calculate the initial water saturation.

4.2.5 Composite physical model preparation

The composite core physical model consists of four parts: two 2-inch cylindrical plugs and two 2-inch hemi-cylindrical plugs as shown in Figure 9. The core samples were cut by disk saw with required dimensions. Then, core samples ends were polished by sanding paper in

order to have smooth end-faces. After saturating all core plugs with formation water, they were reassembled and wrapped with Teflon tape.

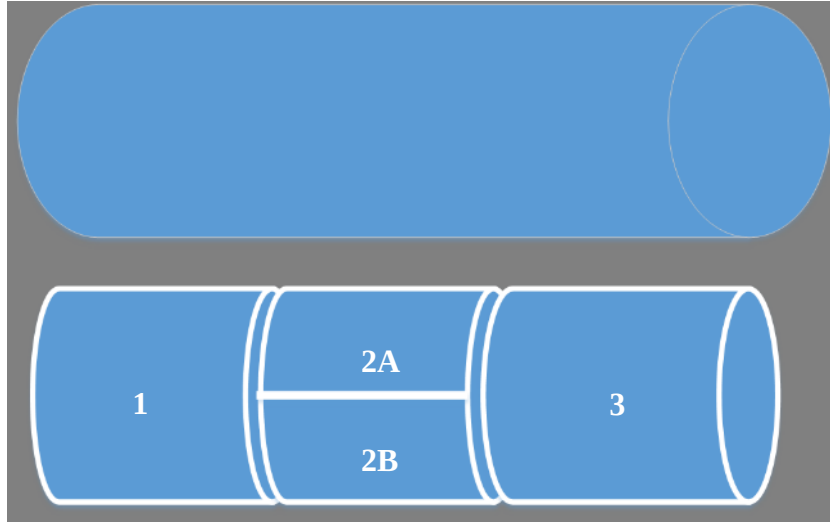


Figure 9: Core Fractured Configuration



Figure 10: Pink desert limestone composite core

4.2.6 Experimental plan

Table 3 shows the implemented experimental core-flooding plan. The first experiment was the baseline to assess the effect of the fracture model on the recovery factor. The remaining experiments were intended to investigate the effect of foam quality and injection modes.

Table 3: Experimental core-flooding plan

#	Secondary recovery	Core physical model	Foam quality (%)	CO2 Rate (cc/min)	Surfactant Rate (cc/min)
1	No seawater injection	Hole core	70	0.7	0.3
2		Fracture composite model	80	0.8	0.2
3			SAG	1 per cycle	1 per cycle
4	Seawater injection		70	0.7	0.3
5			80	0.8	0.2
6			SAG	1 per cycle	1 per cycle

4.2.7 CT scan imaging

Computerized tomography (CT) scan was used to scan all the cores at three phases: (1) Cores saturated with formation water only; (2) Cores saturated with oil and formation water at initial water saturation; and (3) After core-flooding experiments to determine the saturation profile in each part of the fracture system. This step was essential to evaluate the foam's efficiency, which diverts the injected fluid from the fracture to the matrix, the efficiency of SC-CO₂, and the reduced residual oil saturation (S_{or}).

4.2.8 NMR T2 Diffusion

Nuclear magnetic resonance (NMR) measurements were conducted on the two hemicylindrical cores to compare residual oil saturation. T2 diffusion measurement was performed to determine which core had more oil.

4.3 Experimental apparatus

4.3.1 Core-flooding system

The core-flooding system was comprised of five parts: a core holder, four floating piston vessels, three pumps, an oven, and the data-acquisition. The setup schematic is shown in Figure 11. The core was designed to accommodate the core plugs inside the core sleeve while maintaining the ability to apply confining pressure on the core. The core holder and three floating piston vessels containing the seawater, oil, and surfactant solution were placed inside the oven. The floating piston vessels were all connected to one pump since these fluids were not injected together. The CO₂ was placed outside the oven because the CO₂ pressure can exceed the vessel's pressure limitation at high temperature.

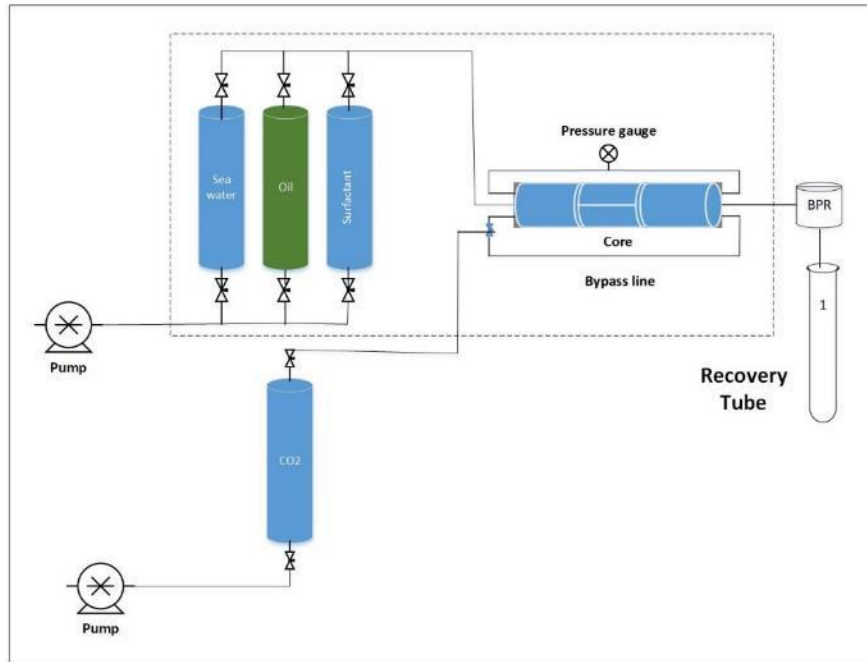


Figure 11: Schematics of core-flooding system

CHAPTER 5

RESULTS AND DISCUSSION

This section demonstrates, analyzes, and discusses the outcomes of six core-flooding experiments performed in this study. Three flooding modes were investigated in this study as below: continuous CO₂ flooding, continuous co-injection of CO₂, and surfactant-alternating gas injection. The flooding experiments were divided into two sets. Each set consisted of three cores. In the first set, each flooding mode was tested on one core individually. However, the second set was flooded by seawater before testing each flooding mode. All cores were scanned by medical CT scan at three different stages; at 100% water saturation, initial water saturation, and post-flooding. In this chapter, the recovery performance, differential pressure, CT scan images, and NMR scans are presented and discussed for each experiment.

5.1 Cores petro-physical properties

The core samples were prepared according to the preparation steps in the methodology chapter. They were dried and weighed before being saturated with formation brine. After that, the cores were weighed again to measure the porosity by material balance. Next, the core samples were placed into the core holder in the flooding system to measure the brine permeability using the Darcy equation. The samples were then placed in the centrifugal machine to displace the formation brine with oil until reaching the initial water saturation, S_{wi} . At 100% water saturation and at S_{wi} , the core was scanned by the medical CT scanner. Table 4 shows the petrophysical properties of all cores.

Table 4: .Cores petro-physical properties

Core ID	Sam No.	Length, in	Porosity, %	Permeability, md	Pore volume, cc	Initial water saturation, %
R1	R1	6	21.2	60.9	37.5	28%
C1	C11	2	20.7	75.2	37.8	16.3%
	C12A		23.2			4.0%
	C12B		20.3			3.0%
	C13		22.8			23.1%
C2	C21		19.9	72.4	34.4	7.8%
	C22A		20.7			5.1%
	C22B		20.8			5.6%
	C23		19.6			10.1%
C3	C31		23.0	79.1	38.2	25.0%
	C32A		22.8			4.1%
	C32B		21.4			9.7%
	C33		21.2			16.3%
C4	C41		18.2	126.3	29.5	15.3%
	C42A		18.5			7.1%
	C42B		18.3			9.3%
	C43		18.1			11.3%
C5	C51		17.5	115.3	28.5	13.6%
	C52A		17.6			5.2%
	C52B		17.4			10.5%
	C53		17.5			9.9%

5.2 Tertiary recovery performance

5.2.1 Ex1 CO₂ flooding with foam (70/30)

In this experiment, CO₂ and AOS surfactant were injected simultaneously into the core to form foam with a foam quality of 70%. The CO₂ injection rate was 0.7 cc/min, and the AOS surfactant solution rate was 0.3 cc/min, which results in a foam injection rate of 1.0 cc/min. The oil production started after injecting 0.3 PV of the foam solution, as shown in Fig. 12. The gas breakthrough started after injecting 0.45 PV. The differential pressure increased to 200 psi before the breakthrough, which can be referred to as two reasons; the gas compressibility and in-situ CO₂ emulsion. The differential pressure started dropping

dramatically after the breakthrough due to the reduction in the viscous forces. The ultimate recovery was 86%, which was achieved after injecting 0.9 PV of CO₂ and surfactant solution. The recovery did not increase even after flooding the core with an additional 2.3 PV. This recovery factor is considered relatively high. However, this experiment's results will be compared to the remaining experiment to investigate the effect of the different flooding modes of the recovery factor and the residual oil saturation. Figure 13 shows the CT scan images before and after flooding. The average CT number had been reduced from 2241 to 2079. This reduction indicates the changes in the overall density of the core. The estimation of residual oil saturation using the CT number Siddiqui method was 10%, which matches the recovery performance curve.

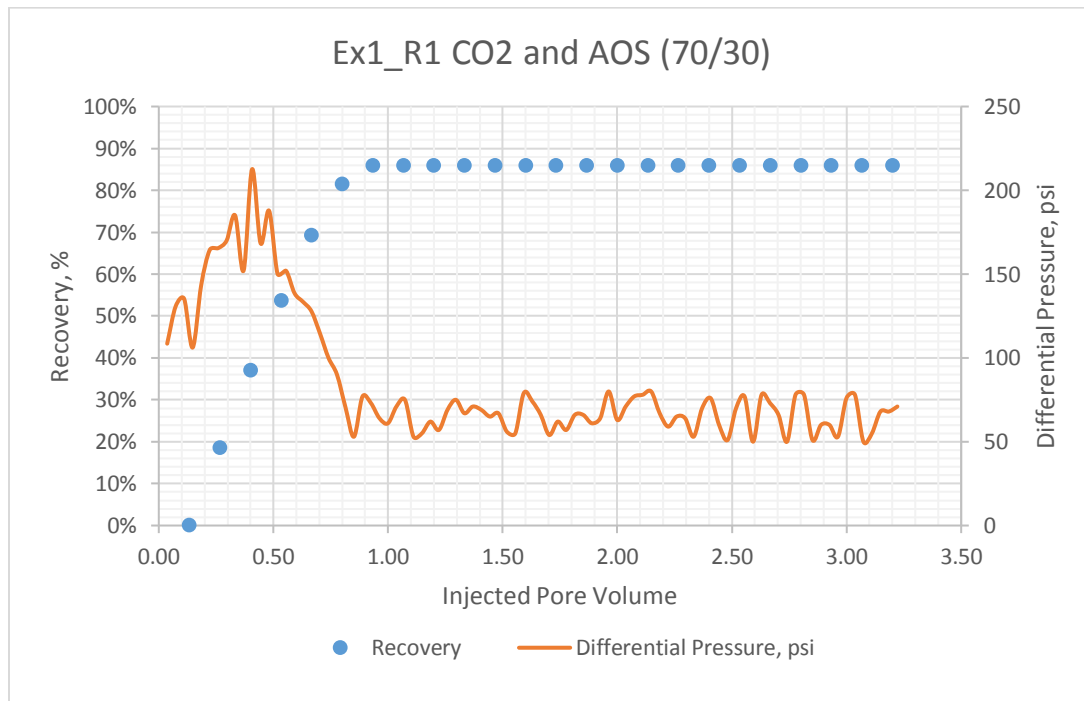


Figure 12: Ex1_R1 Recovery and differential pressure profile

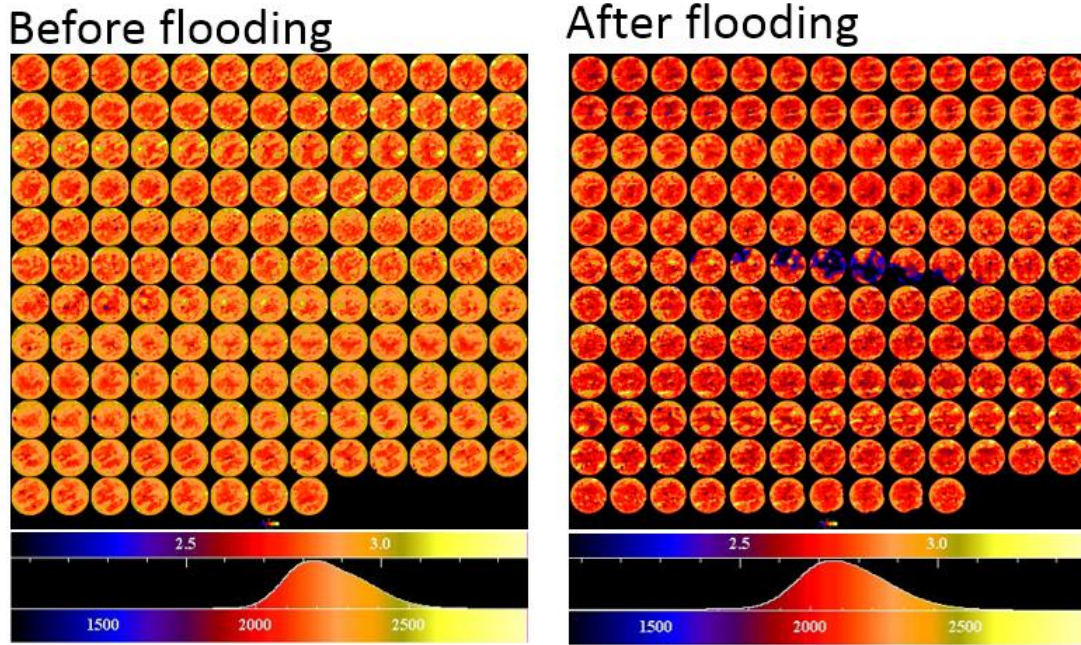


Figure 13: Core R1 CT scan images before and after flooding

5.2.2 Ex2 CO₂ flooding with foam (80/20)

CO₂ and AOS surfactant were injected simultaneously into the core composite to form foam with a foam quality of 80%. The CO₂ injection rate was 0.8 cc/min, and the AOS surfactant solution rate was 0.2cc/min, which results in a foam injection rate of 1.0 cc/min. The oil production started after injecting 0.15 PV of the foam solution, as shown in Figure 14. The gas breakthrough started after injecting 0.5 PV of the AOS surfactant solution. The differential pressure increased to 80 psi before the breakthrough due to the in-situ CO₂ foam emulsion to block the high permeability zones. The differential pressure started dropping after the breakthrough. However, the differential pressure was decreasing slightly and fluctuating. The fluctuation is because of the foam trying to block the high permeability zones. The ultimate recovery was 74%, which was achieved after injecting 2 PV of CO₂ and surfactant solution. However, most of the oil production was recovered by the first PV. Figures 15, 16, and 17 show the CT scan images before and after flooding. Average CT

number reduced from 2179 to 2055, from 2170 to 2051, and from 2171 to 2023, respectively, for each plug in the composite core. This reduction indicates the changes in the overall density of the core. The estimated residual oil saturations using CT numbers were 29.9%, 34.2%, and 22.3%, which match the overall recovery factor. NMR T2 diffusion shows that gravity override was not resolved when the lower core has more oil than the upper one, as shown in Figures 18 and 19.

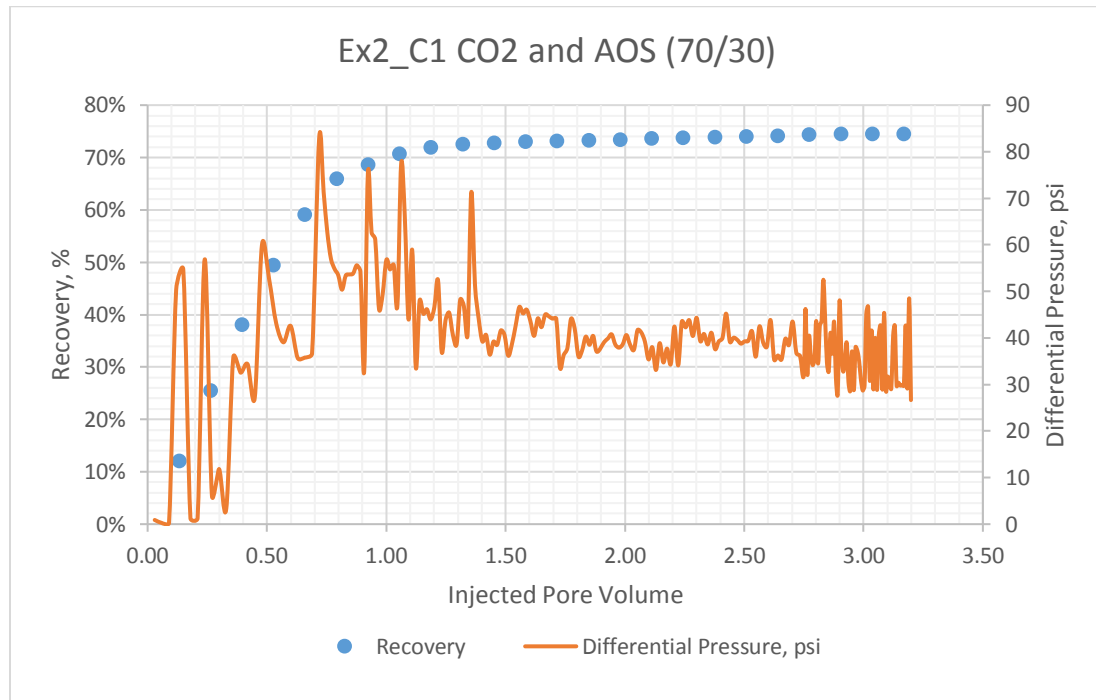
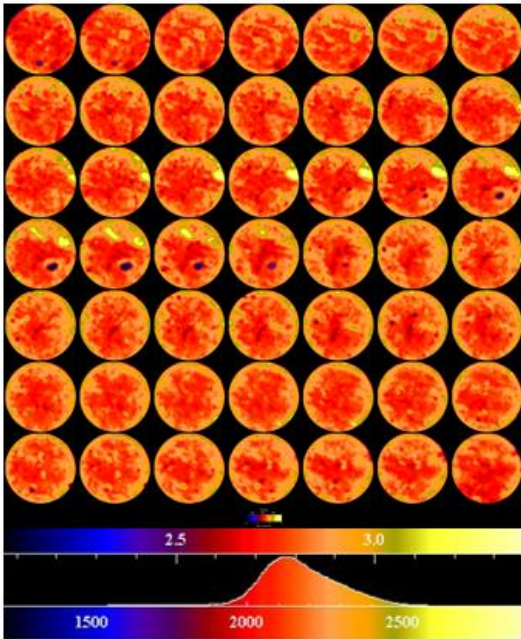


Figure 14: Ex2_C1 Recovery and differential pressure profile

Before flooding



After flooding

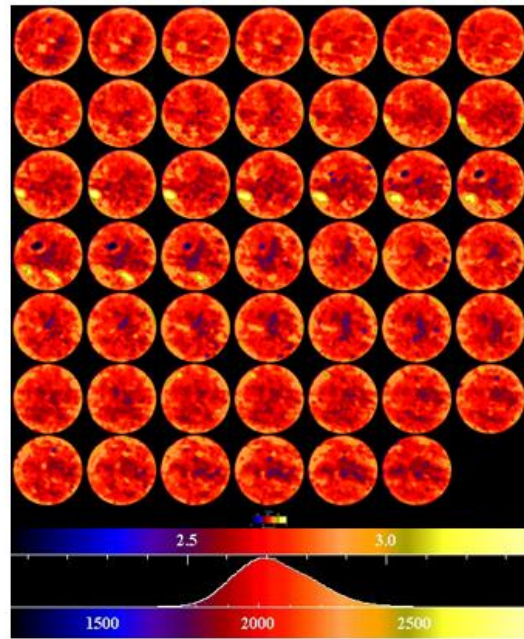
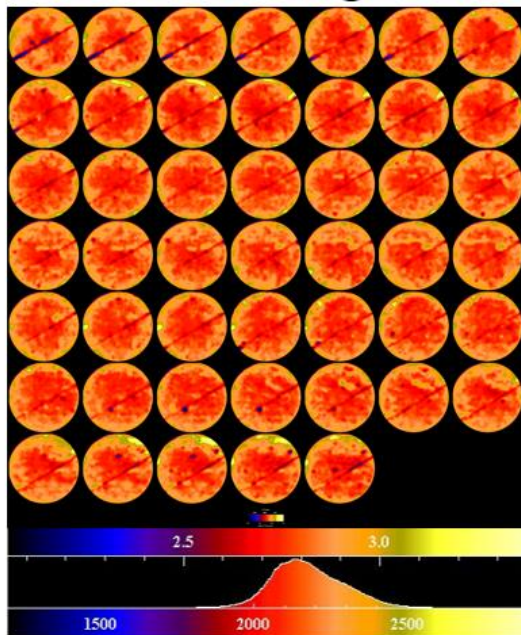


Figure 15: Core C11 CT scan images before and after flooding

Before flooding



After flooding

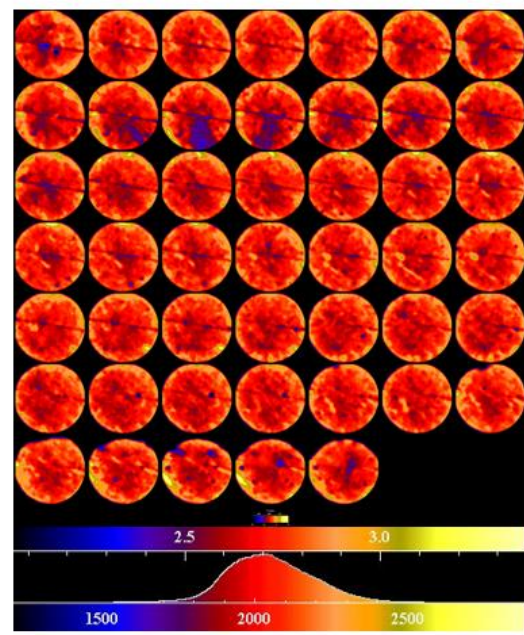
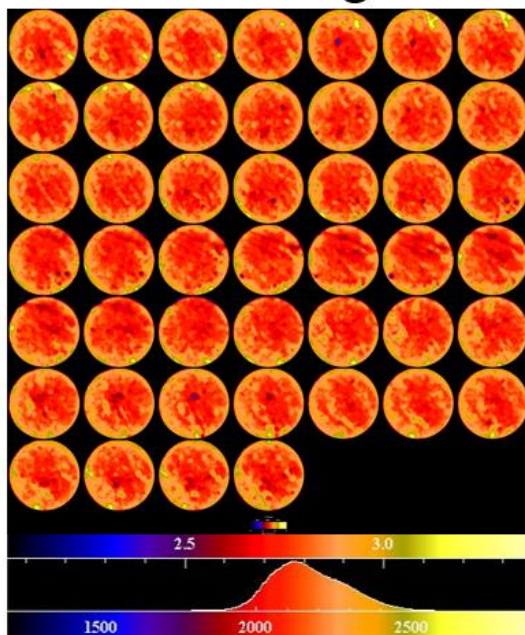


Figure 16: Core C12 CT scan images before and after flooding

Before flooding



After flooding

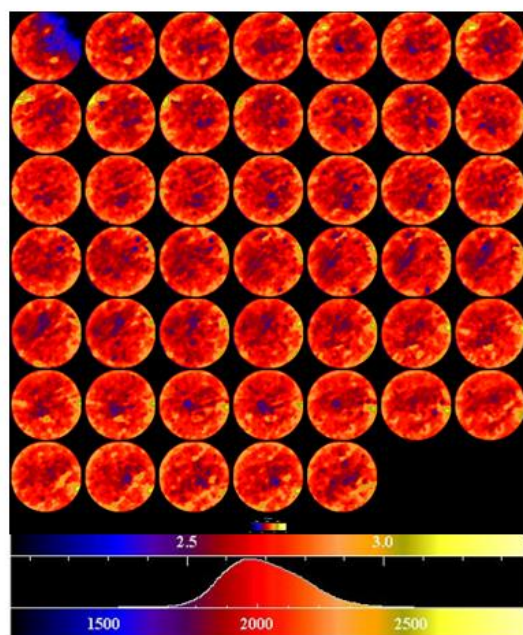


Figure 17: Core C13 CT scan images before and after flooding

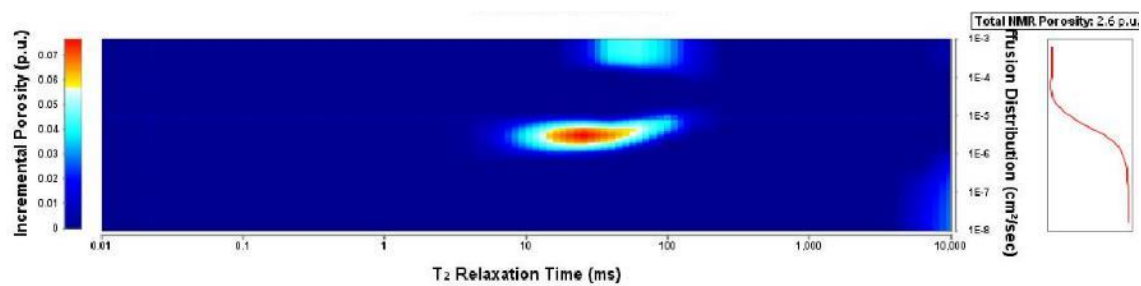


Figure 18: C12A NMR T2 diffusion

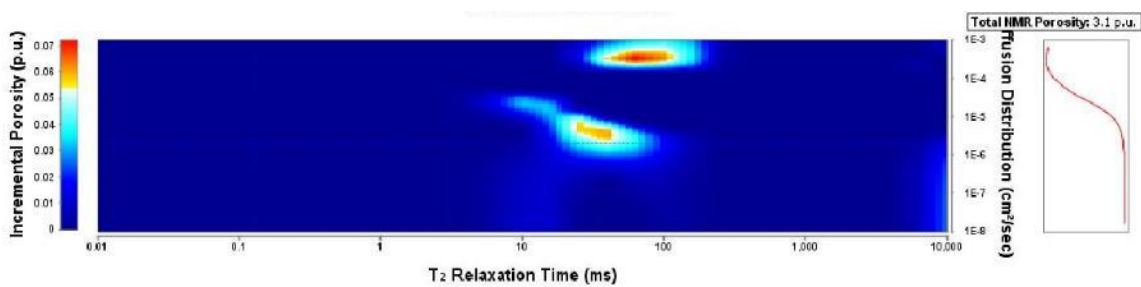


Figure 19: C12B NMR T2 diffusion

5.2.3 Ex3 Surfactant-alternating gas (SAG) injection

In this experiment, surfactant-alternating gas (SAG) injection mode was investigated. Seven equal cycles of SAG flooding were injected to displace the oil inside the composite core at a pore pressure of 3200 psig. In this flooding mode, the slug size was 0.3 PV of each fluid at an injection rate of 1.0 cc/min. Oil production started after injecting 0.45 PV, as shown in Figure 20. Figure 20 shows the differential pressure profile. The differential pressure sharply increased while injecting the SC-CO₂ slug, which indicates that the previous surfactant slug has increased the viscous forces to divert the flow from the fracture to the matrix. The ultimate recovery was 78%, which was achieved after injecting 3 PV. Comparing the in-situ co-injection results to the SAG injection, it seems that in-situ co-injection is more efficient than the SAG injection because most of the recovered oil was produced by the first PV injected fluid. Figures 21, 22, and 23 show the CT scan images before and after flooding. Average CT number reduced from 2247 to 2117, from 2229 to 2096, and from 2210 to 2094, respectively, for each plug in the composite core. This reduction indicates the changes in the overall density of the core. The estimated residual oil saturations using CT numbers were 26%, 22%, and 18%. However, the estimated overall recovery by the CT method 73%, which is 5% lower than the measured recovery. NMR T2 diffusion shows that the oil saturation is almost uniform in the two hemi-cylindrical plugs, as shown in Figures 24 and 25.

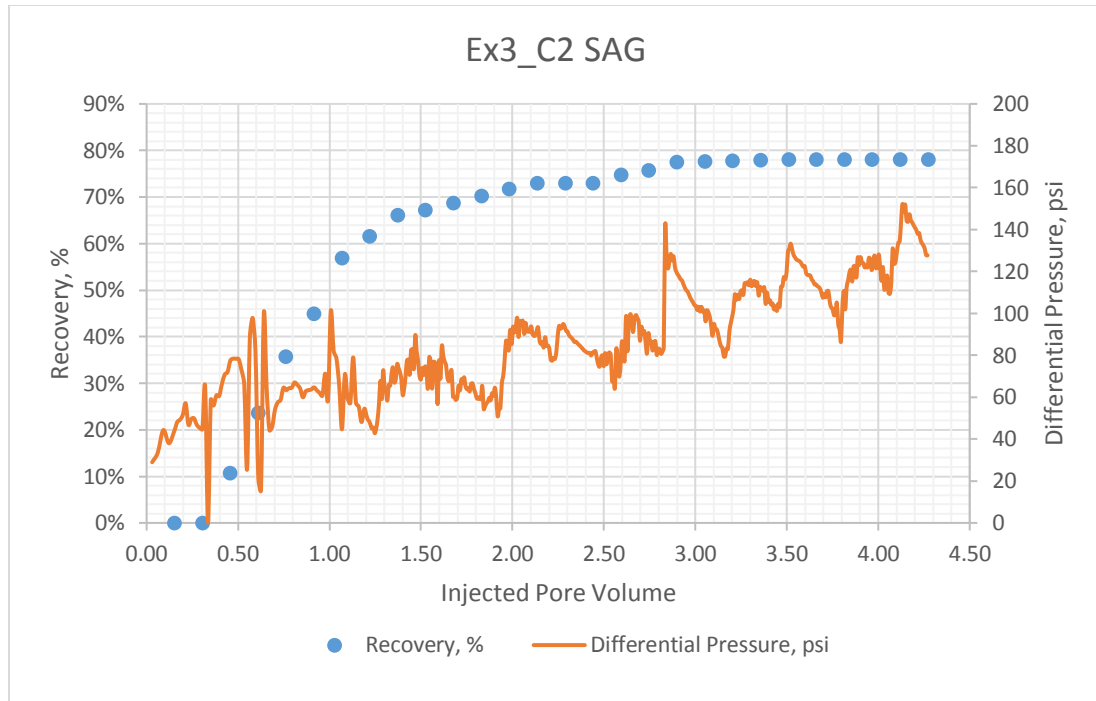


Figure 20: Ex3_C2 Recovery and differential pressure profile

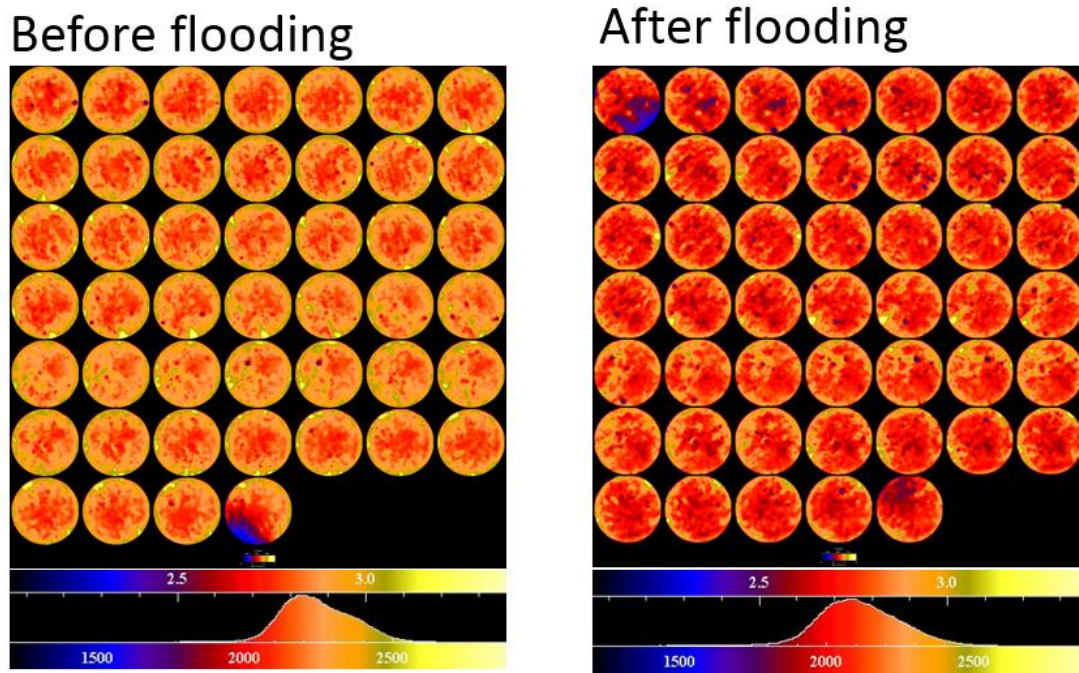
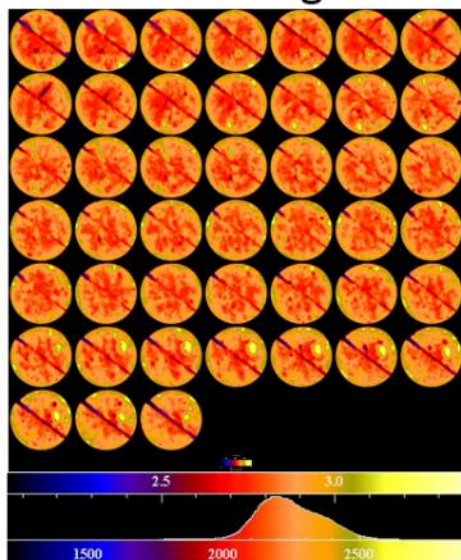


Figure 21: Core C21 CT scan images before and after flooding

Before flooding



After flooding

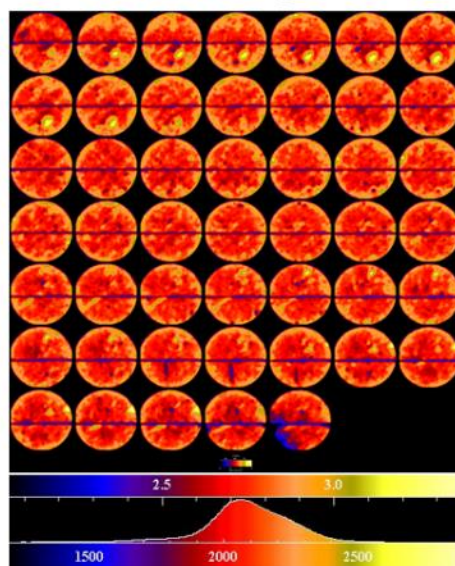
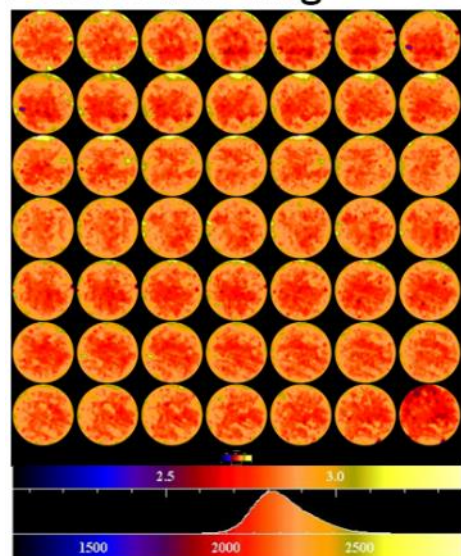


Figure 22: Core C22 CT scan images before and after flooding

Before flooding



After flooding

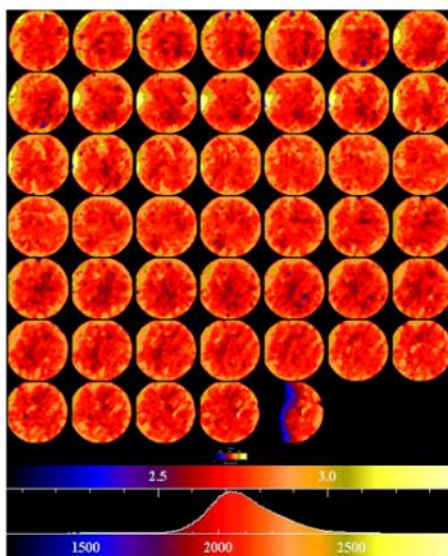


Figure 23: Core C23 CT scan images before and after flooding

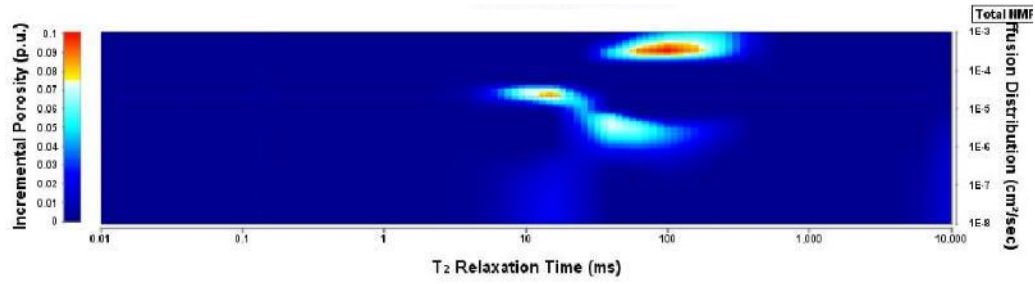


Figure 24: C22A NMR T2 diffusion

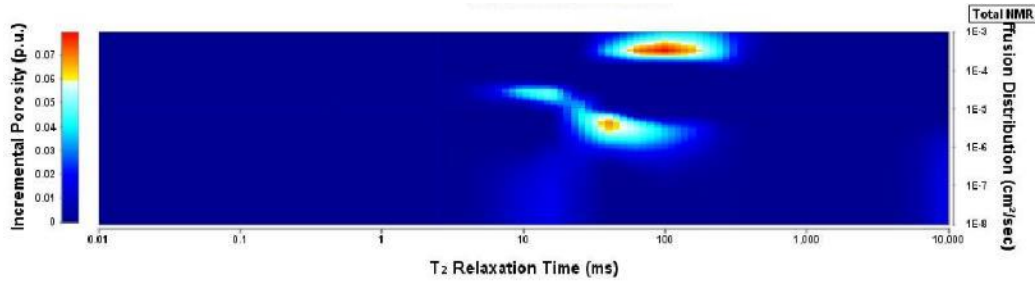


Figure 25: C22B NMR T2 diffusion

5.3 Secondary and tertiary recovery performance

5.3.1 Ex4 Waterflooding, CO₂ and AOS (70/30)

In the experiment, the core was flooded by 3 PV of seawater with an injection rate of 1 cc/min. The seawater flooding recovered 34% of the OOIP. The oil recovery reached its maximum after injecting 0.85 PV and did not increase after flooding the core with an additional 2 PV. After that, CO₂ and AOS surfactant were injected simultaneously into the core composite to form foam with a foam quality of 70%. CO₂ injection rate was 0.7cc/min, and the AOS surfactant solution rate was 0.3 cc/min, which results in a foam injection rate of 1.0 cc/min. The oil production started after injecting 0.2 PV of the foam solution, as shown in Figure 26. The gas breakthrough started after injecting 0.3 PV. The differential pressure increased to 230 psi before the breakthrough due to the in-situ CO₂ foam emulsion blocking the high permeability zones. The differential pressure started dropping after the

breakthrough. However, the differential pressure was decreasing slightly and fluctuating. The fluctuation is because of the foam trying to block the high permeability zones. The ultimate recovery was 70%, which was achieved after injecting 3.4 PV of the CO₂ and surfactant solution. Figures 27, 28, and 29 show CT scan images before and after flooding. Average CT numbers reduced from 2189 to 2050, from 2228 to 2117, and from 2226 to 2108, respectively, for each plug in the composite core. This reduction indicates the changes in the overall density of the core. The estimated residual oil saturations using CT numbers were 22.5%, 35.8%, and 24.8%, which match the overall recovery factor. NMR T2 diffusion did not indicate oil saturation, as shown in Figures 30 and 31.

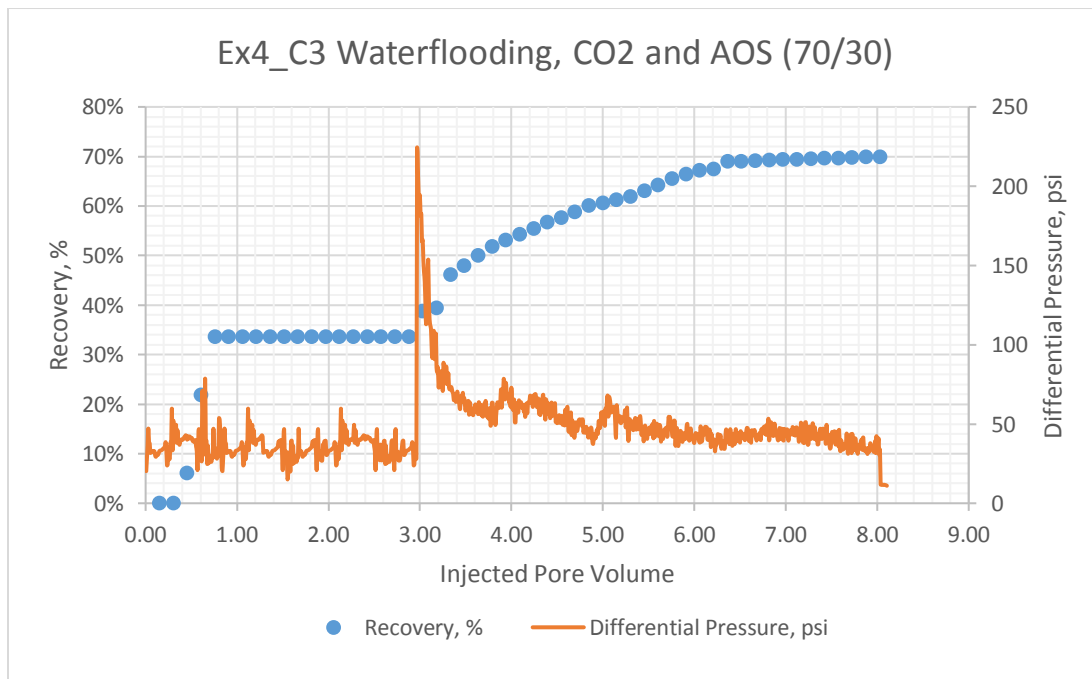
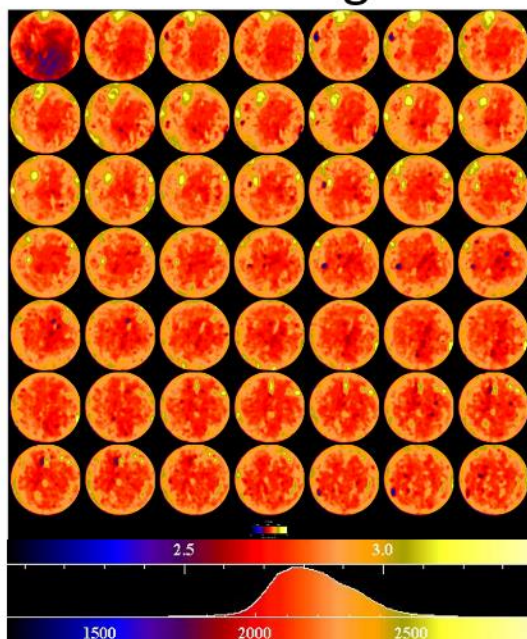


Figure 26: Ex4_C3 Recovery and differential pressure profile

Before flooding



After flooding

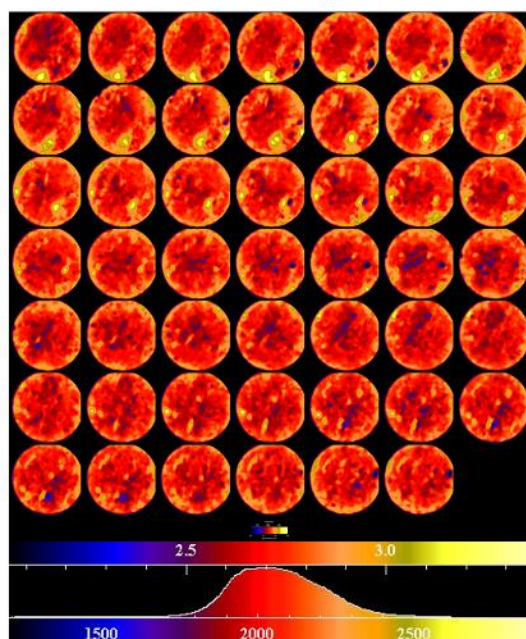
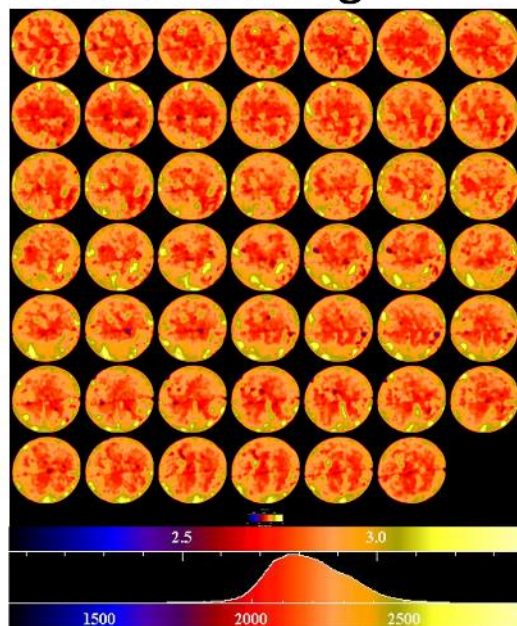


Figure 27: Core C31 CT scan images before and after flooding

Before flooding



After flooding

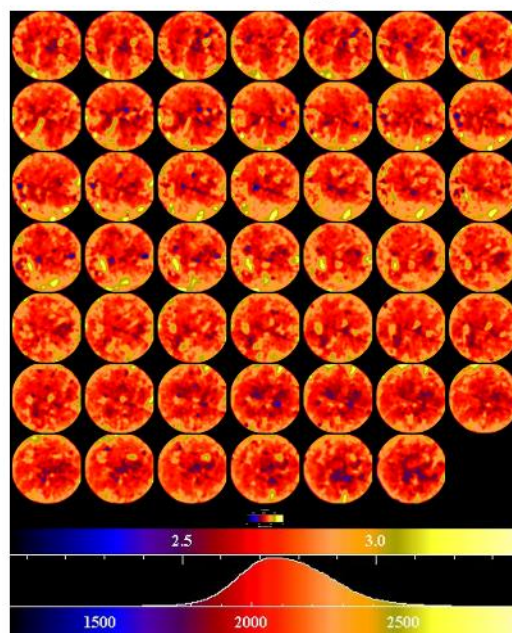
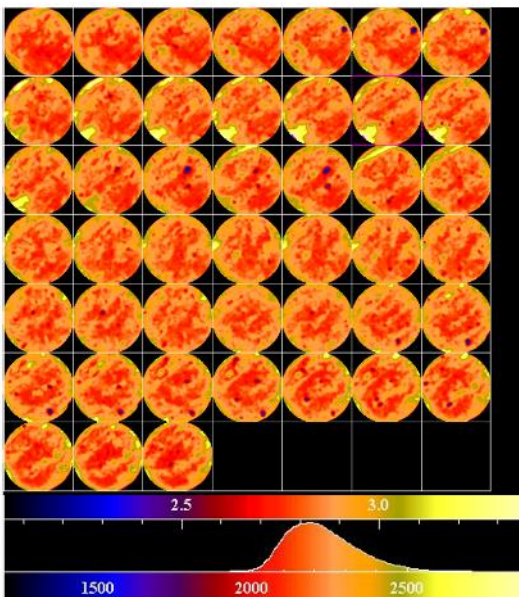


Figure 28: Core C32 CT scan images before and after flooding

Before flooding



After flooding

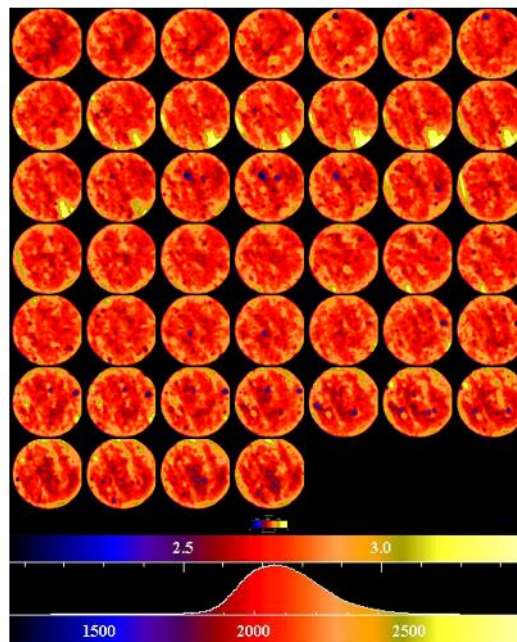


Figure 29: Core C33 CT scan images before and after flooding

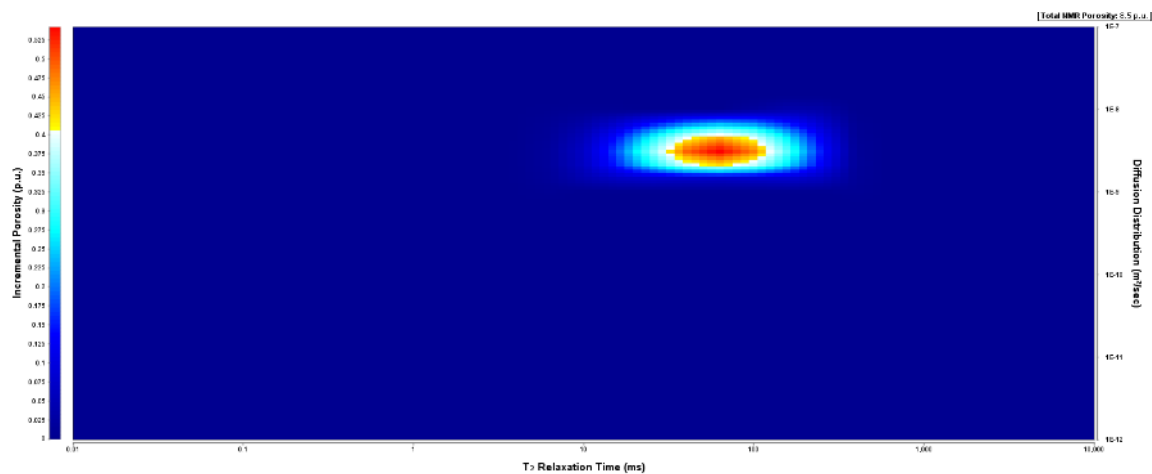


Figure 30: C32A NMR T2 diffusion

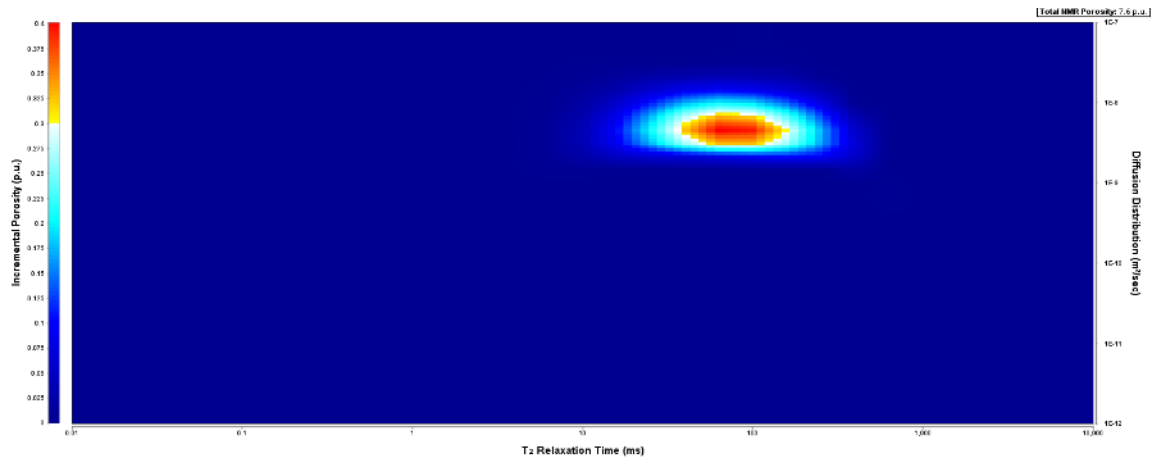


Figure 31: C32B NMR T2 diffusion

5.3.2 Ex5 Waterflooding, CO₂ and AOS (80/20)

In the experiment, the core was flooded by 3 PV seawater with an injection rate of 1.0 cc/min. The seawater flooding recovered 36% of the OOIP. The oil recovery reached its maximum after injecting 1 PV and did not increase after flooding the core with an additional 2 PV. After that, CO₂ and AOS surfactant were injected simultaneously into the core composite in order to form foam with a foam quality of 80%. The CO₂ injection rate was 0.8 cc/min, and the AOS surfactant solution rate was 0.2 cc/min, which results in a foam injection rate of 1.0 cc/min. The oil production started after injecting 0.2 PV of the foam solution, as shown in Figure 32.

The gas breakthrough started after injecting 0.15 PV. The differential pressure increased to 70 psi before the breakthrough due to the in-situ CO₂ foam emulsion blocking the high permeability zones. The differential pressure started dropping after the breakthrough. However, the differential pressure was decreasing slightly and fluctuating. The fluctuation is because of the foam trying to block the high permeability zones. The ultimate recovery was 67%, which was achieved after injecting 2 PV of CO₂ and surfactant solution. The

injection rate was increased 2.0 cc/min while maintaining the same foam quality. However, the recovery did not improve. Figures 33, 34, and 35 show the CT scan images before and after flooding. Average CT number reduced from 2242 to 2154, from 2225 to 2100, and from 2246 to 2151, respectively, for each plug in the composite core. This reduction indicates the changes in the overall density of the core. The estimated residual oil saturations using CT numbers were 30%, 26.5%, and 30%, which did not match the overall recovery factor. This is due to the fracture in the middle core plug, as shown in Figure 34. NMR T2 diffusion shows that the oil saturation is almost uniform in the two hemicylindrical plugs shown in Figures 36 and 37.

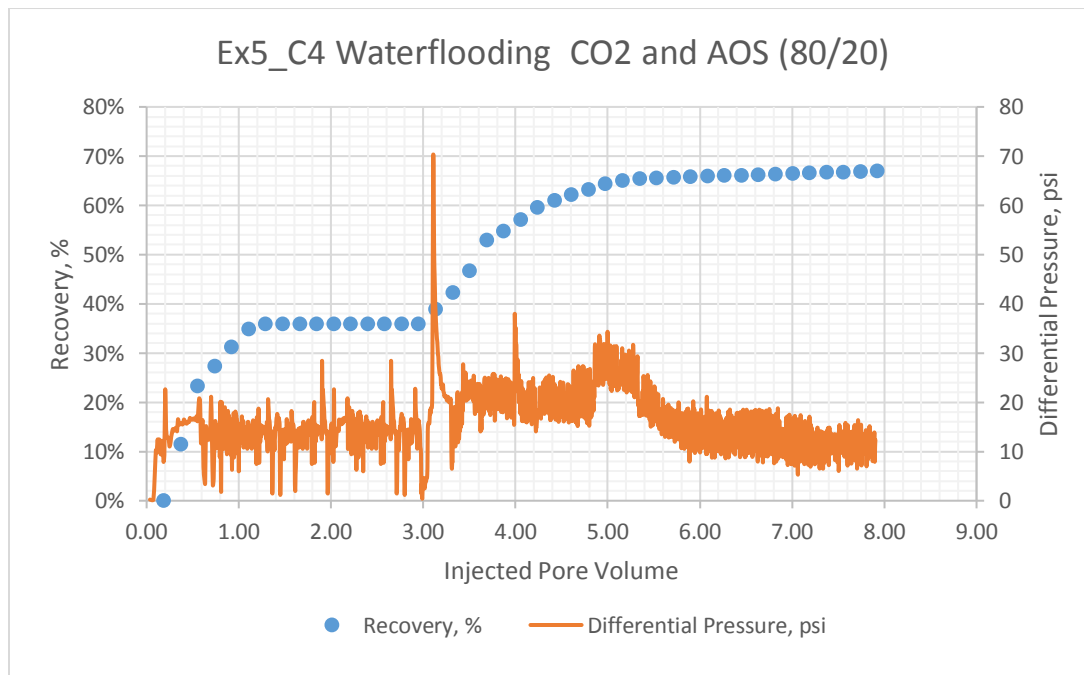
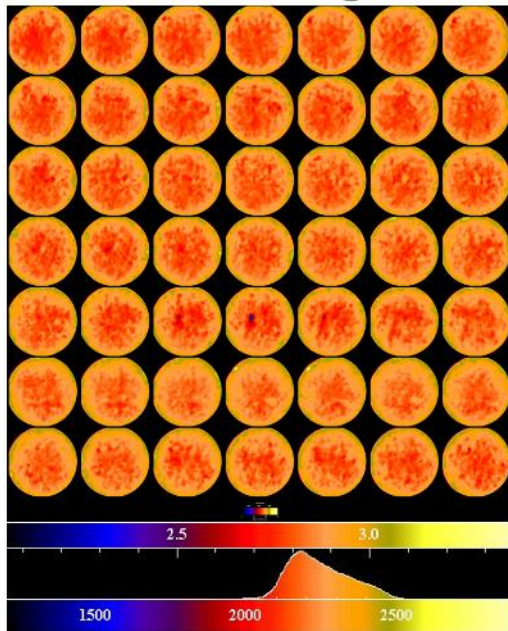


Figure 32: Ex5_C4 Recovery and differential pressure profile

Before flooding



After flooding

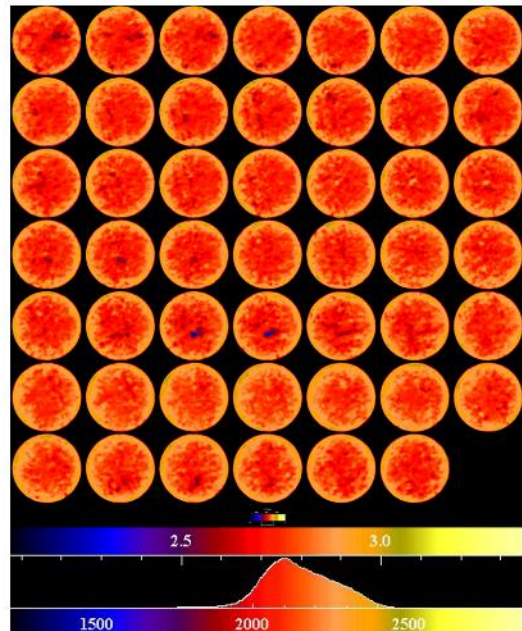
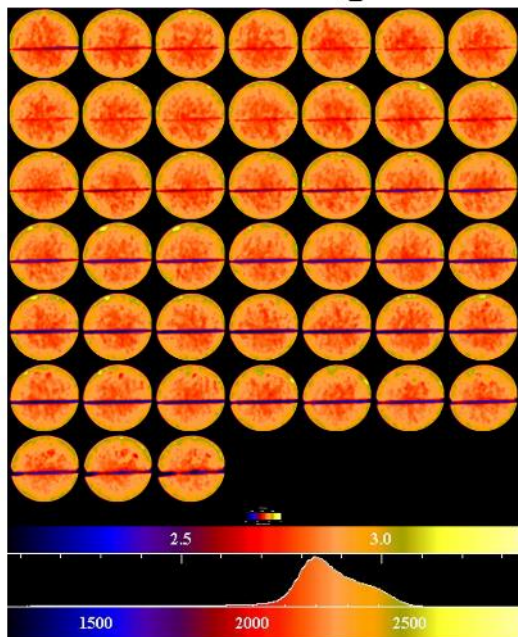


Figure 33: Core C41 CT scan images before and after flooding

Before flooding



After flooding

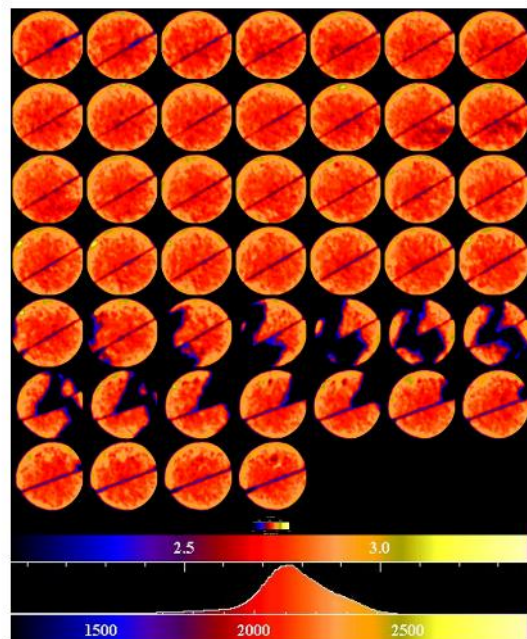
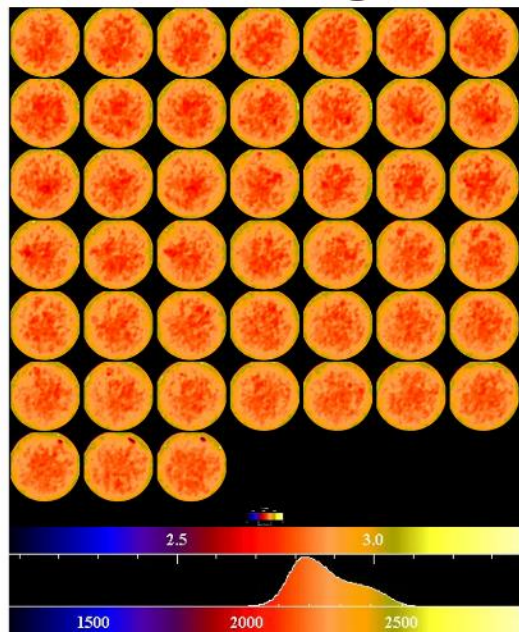


Figure 34: Core C42 CT scan images before and after flooding

Before flooding



After flooding

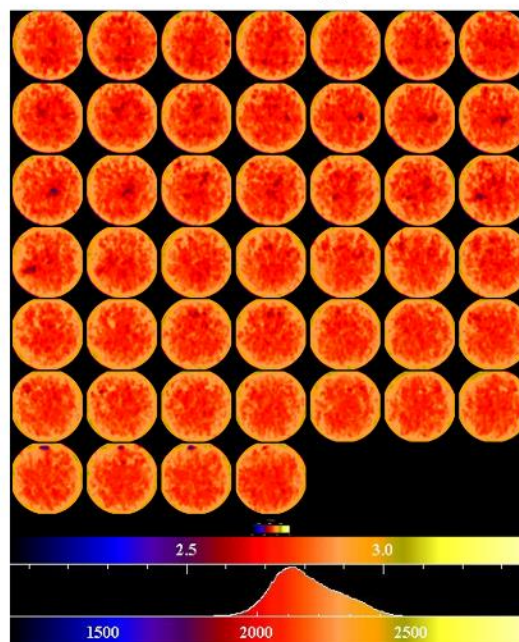


Figure 35: Core C43 CT scan images before and after flooding

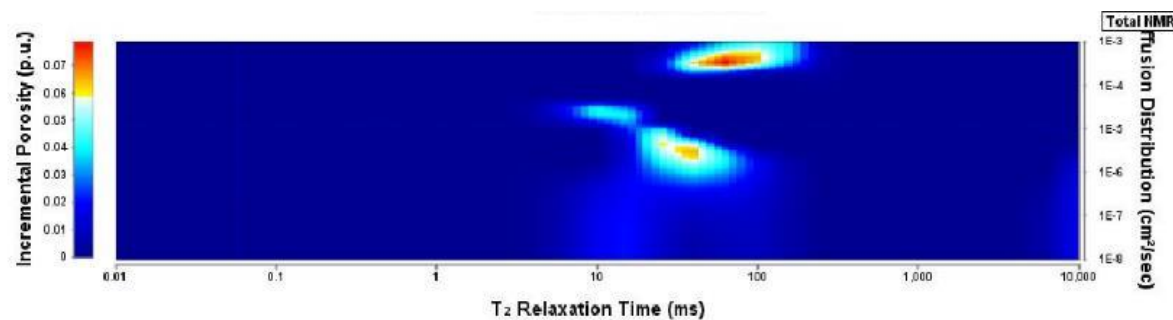


Figure 36: C42A NMR T2 diffusion

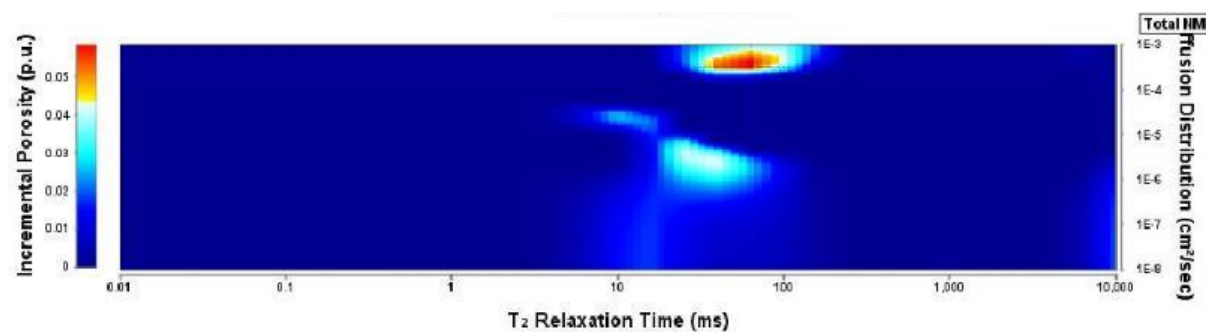


Figure 37: C42B NMR T2 diffusion

5.3.3 Ex6 Waterflooding and SAG

In the experiment, the core was flooded by 3 PV seawater with an injection rate of 1.0 cc/min. The seawater flooding recovered 37% of the OOIP. The oil recovery reached its maximum after injecting 0.9 PV and did not increase after flooding the core with an additional 2 PV. After that, surfactant-alternating gas injection mode was investigated. Five equal cycles of SAG flooding were injected to displace the oil inside the composite core at a pore pressure of 3200 psig. In this flooding mode, the slug size was 0.3 PV of each fluid at an injection rate of 1.0 cc/min. Oil production started after injecting 0.3 PV, as shown in Figure 38. Figure 38 shows the differential pressure profile. The differential pressure sharply increased while injecting the SC-CO₂ slug, which indicates that the previous surfactant slug has increased the viscous forces to divert the flow from the fracture to the matrix. The ultimate recovery was 72%, which was achieved after injecting 1.5 PV. Figures 21, 22, and 23 show the CT scan images before and after flooding. Average CT number reduced from 2250 to 2158, from 2243 to 2134, and from 2239 to 2143, respectively, for each plug in the composite core. This reduction indicates the changes in the overall density of the core. The estimated residual oil saturations using CT numbers were 26%, 25.2%, and 23%. The CT method's estimated overall recovery is 74%, which is 2% higher than the measured recovery. NMR T2 diffusion shows that the oil saturation is almost uniform in the two hemi-cylindrical plugs, as shown in Figures 42 and 43.

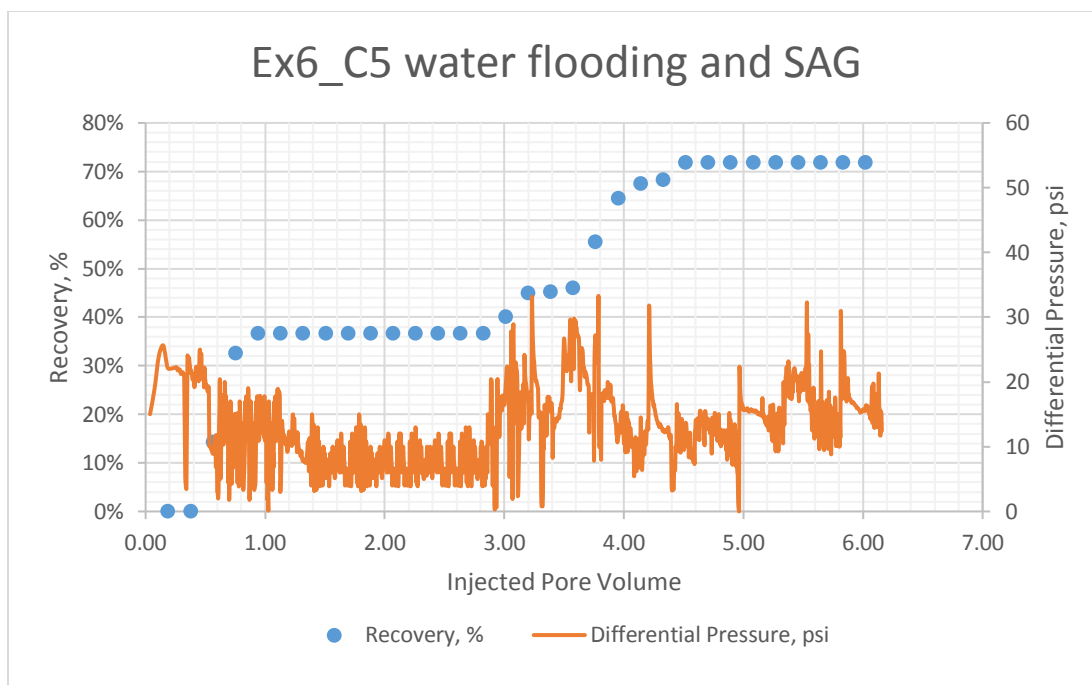
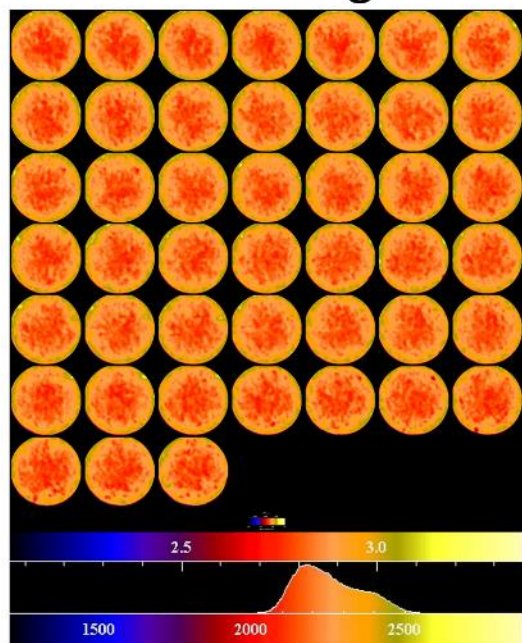


Figure 38: Ex6_C5 Recovery and differential pressure profile

Before flooding



After flooding

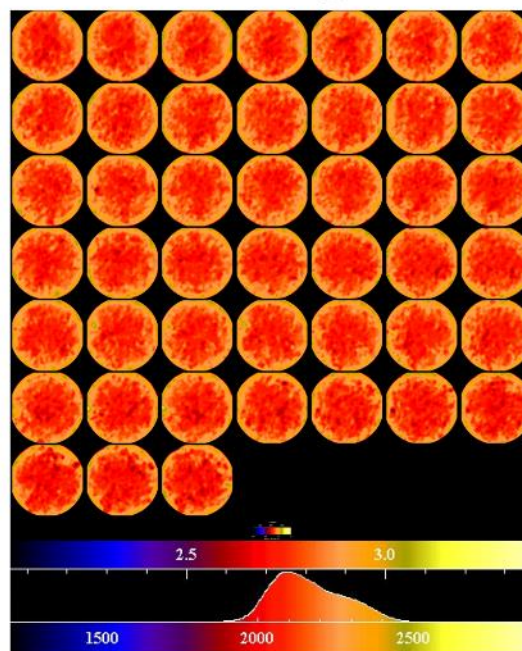
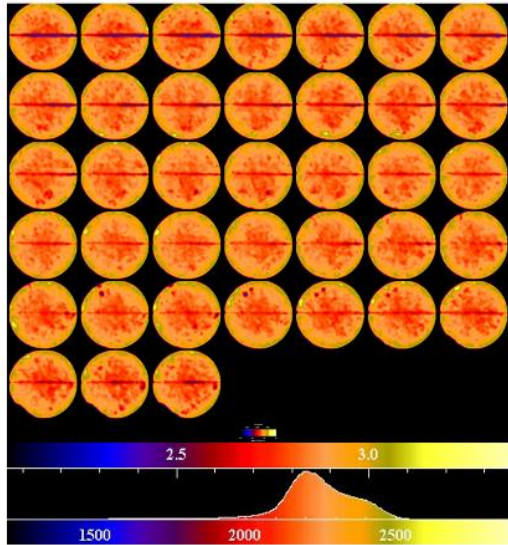


Figure 39: Core C51 CT scan images before and after flooding

Before flooding



After flooding

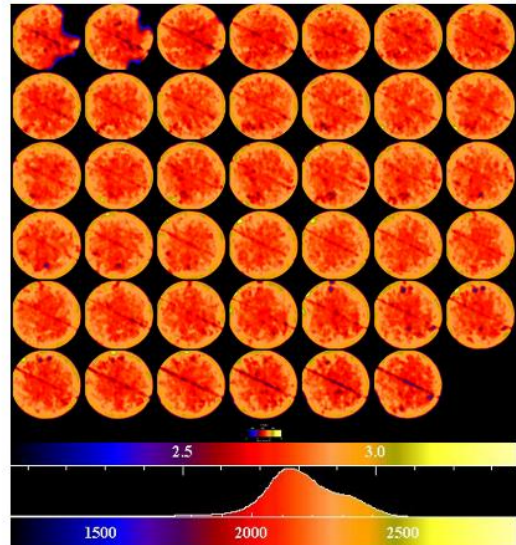
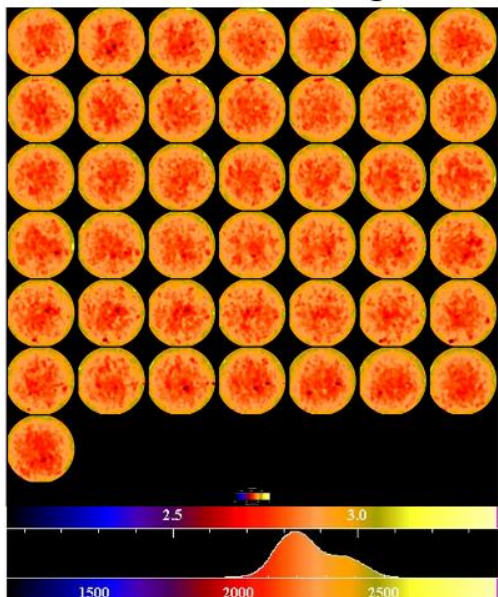


Figure 40: Core C52 CT scan images before and after flooding

Before flooding



After flooding

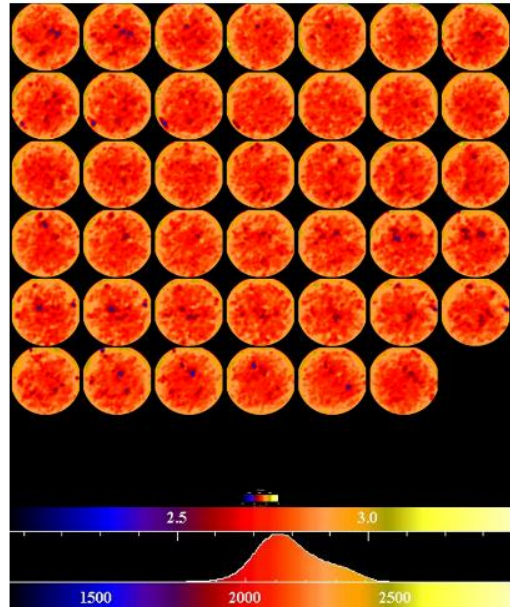


Figure 41: Core C53 CT scan images before and after flooding

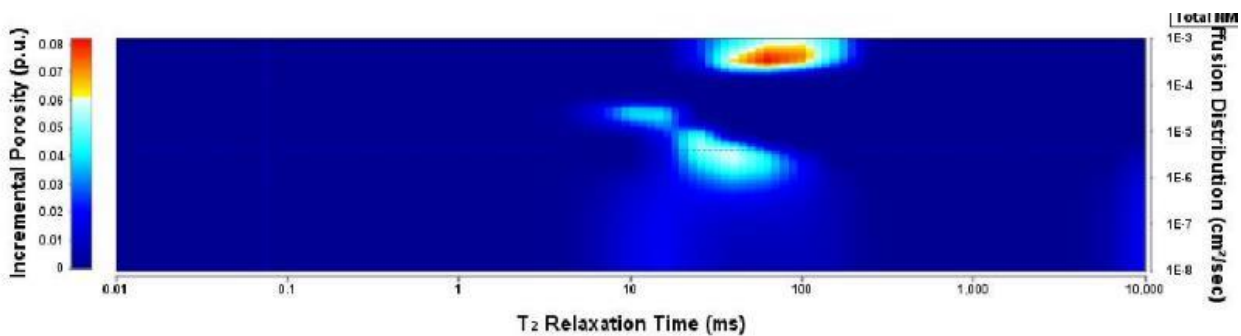


Figure 42: C52A NMR T2 diffusion

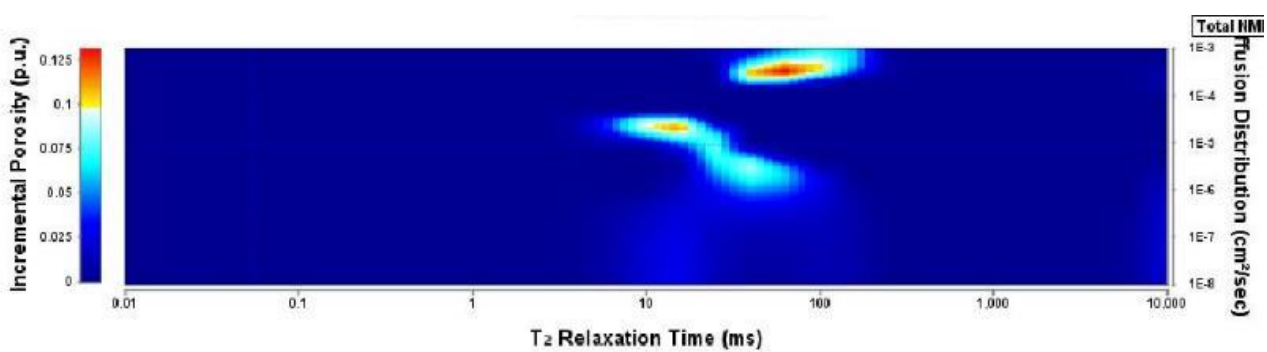


Figure 43: C52B NMR T2 diffusion

Table 5: Summary of CT number, Swi, Sor, and recovery factor

Core ID	Sam No.	CT 100	CT Swi	CT post	Swi	Sor	CT Recovery	Measured Recovery
R1	R1	2270	2241	2079	0.28	0.11	85%	86%
C1	C11	2250	2180	2055	0.16	0.30	74%	74%
	C12A	2235	2170	2052	0.04	0.34		
	C12B				0.03			
	C13	2231	2171	2024	0.23	0.22		
C2	C21	2299	2247	2117	0.08	0.26	73%	78%
	C22A	2268	2229	2096	0.05	0.22		
	C22B				0.06			
	C23	2238	2210	2094	0.10	0.18		
C3	C31	2249	2189	2050	0.25	0.23	72%	70%
	C32A	2298	2228	2117	0.04	0.36		
	C32B				0.10			
	C33	2276	2226	2108	0.16	0.25		
C4	C41	2290	2242	2155	0.15	0.30	72%	67%
	C42A	2275	2225	2101	0.07	0.27		
	C42B				0.09			
	C43	2295	2246	2151	0.11	0.30		
C5	C51	2290	2250	2159	0.14	0.26	74%	72%
	C52A	2285	2243	2134	0.05	0.25		
	C52B				0.11			
	C53	2275	2239	2143	0.10	0.24		

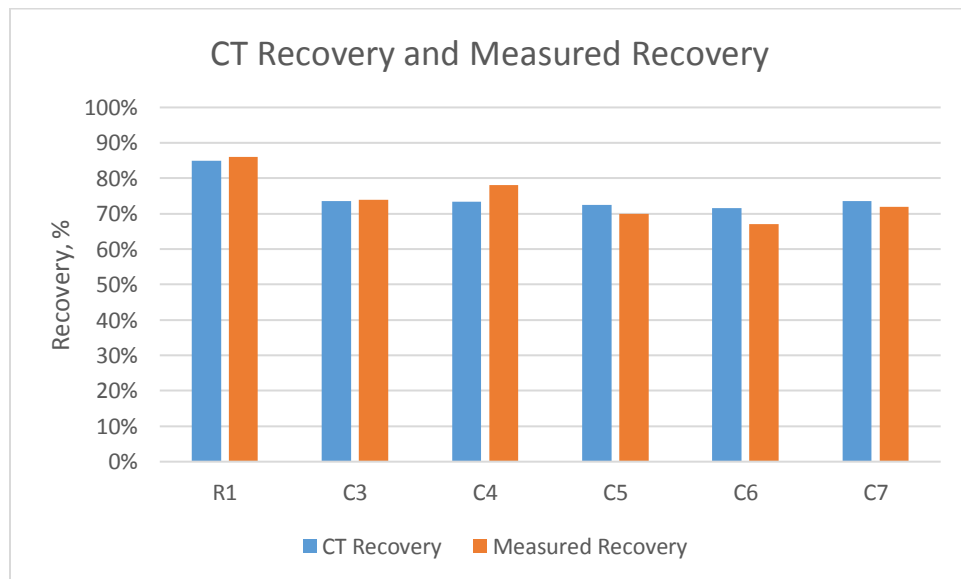


Figure 44: CT recovery estimating and measure recovery

CHAPTER 6

CONCLUSIONS AND RECCOMENDATIONS

This thesis aims to investigate foam SC-CO₂ injection for enhanced oil recovery in fractured limestone cores through flooding schemes. This thesis introduces a new core composite model with multiple horizontal and vertical fractures. This work investigates the effect of foam quality (using AOS surfactant) on the sweep efficiency, SC-CO₂ mobility, and gravity segregation at high-pressure high-temperature conditions (3200 psi and 90 C). In addition, CT scan and NMR are integrated to estimate the residual oil saturation the ultimate recovery of each plug of the composite core. In this research, a novel approach was developed to quantify oil recovery from each composite core plug. This novel approach integrates X-ray CT scans and NMR diffusion, which estimates the individual oil recoveries that added to the total measured recovery, as shown in Table 5 and Figure 44. This approach was able to detect the density override in the composite core. The new approach facilitates the composite core flooding analysis by allowing researchers to have more details about the residual oil distribution. Six core flooding experiments were conducted that investigated several EOR schemes involving secondary recovery, CO₂ foam flooding, and surfactant-alternating gas (SAG). The AOS surfactant was used to generate the foam. Three experiments investigated the CO₂ foam flooding with different foam qualities and SAG without waterflooding. The results showed that the CO₂ foam flooding reached the ultimate recovery faster than the SAG flooding. However, SAG showed the ability to overcome the gravity override issue. The other three experiments were flooding

by seawater and then investigated the same flooding schemes. The results showed that the recovery of CO₂ foam flooding with 80% foam quality was faster than the recovery of CO₂ foam flooding with 70% foam quality. The outcomes of this research may help further to understand the behavior of foam inside the core. For future work, many recommendations can be suggested:

- To change the flooding strategy from the co-injection mode to pre-generated foam mode.
- To investigate the phase behavior of the injected gases and surfactant when coming in contact with the oil in place. The study of phase behavior is useful to understand better the interactions that occur on a molecular level.
- To introduce the technique of visualized testing during the core flooding is thought to be helpful in tracing the flood fronts and the contacted region inside the core by the foam, such as in-situ CT scan.
- Finally, evaluating different surfactants with multiple concentrations.

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