Critical rate analysis for CO2 injection in depleted gas field, Sarawak Basin, offshore East Malaysia

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ABSTRACT
This study aimed to address the challenges and strategies to determine the critical rate of CO2 injection into a carbonate depleted gas field. In this research, the critical rate is the maximum allowable injection rate before formation damage initiation. The cause of formation damage could be due to in-situ mobilization or trapping of migratory fines resulting in plugging the flow path. This study performed a thorough investigation of a different rock-fluid system to evaluate the safe injection limit, as the critical rate is different for each rock-fluid system. The geochemical effect of CO2 injection toward carbonate formation was also investigated in this research. Other than that, the porosity and permeability changes due to CO2-brine-rock multiphase flow characteristics were considered to understand the feasibility of CO2 sequestration into carbonate formation. This research discussed experimental design to mimic the CO2 injection scenario of CO2 into carbonate depleted gas field. Therefore, several core flooding experiments were conducted under reservoir conditions using representative native cores, CO2, and synthetic formation brine. Abrupt changes in differential pressure ($D_P$), analysis of effluent collected after CO2 multi-rate flow, and pH reading are the key indicators to consider that the condition has reached a critical rate. The experimental result demonstrated the existence of fines migration, scale formation, and salt precipitation after the core was subjected to supercritical CO2 multi-rate flow. Considering these issues and challenges associated with injectivity, this study recommended a maximum injection rate prior to field scale injection.

KEYWORDS
Core flooding; carbonate; dissolution; precipitation; CO2 injection

Introduction
CO2 is an efficient injection fluid to enhance oil recovery and geological storage exercise. The injected CO2 is preferred to be in a supercritical state at which it has properties of both a gas and a liquid [1]. Under these conditions, CO2 possesses the solvating power of a liquid and the diffusivity of gas, leading to higher CO2 volume for sequestration.

$CO_2$'s temperature must be $\geq 30.98^\circ{C}$ with pressure $\geq 1070$ psi [2, 3] to be in supercritical forms which will ensure the maximum capacity of stored CO2 needs to be contained under a layer of dense, impenetrable rock above it, known as caprock, to prevent CO2 movement upward. Besides, the sequestration sites require sufficient storage capacity to hold the amount of CO2 planned to be injected over a project's life.

Geological storage
There are several potential geological CO2 storage sites. Geological storage of CO2 can be undertaken in a variety of geological settings such as basins, oil fields, depleted gas fields, deep coal seams, and saline aquifer, [4–8]. It was mentioned that saline aquifers have the highest storage capacity among all other types of underground storage reservoirs [9].

Depleted oil and gas reservoir
This storage location is where thousands of oil and gas fields are approaching the ends of their economically productive lives due to intensive petroleum exploitation. The attractive feature for depleted oil and gas reservoirs as a CO2 sequestration site would be a proven geological structure of porous rocks with impermeable cap rock, traps,
and known to have held hydrocarbon for millions of years. Therefore, these fields could be adapted easily for storage of CO₂. Other than small exploration costs, there is potential to re-apply existing equipment for transportation and CO₂ injection.

Nevertheless, it was mentioned by Davison et al. [10] that there would need to be some changes in current practice to make use of depleted oil and gas reservoirs for CO₂ storage.

Deep saline reservoirs
A saline aquifer refers to sedimentary rock types saturated with saline water and unsuitable for supplying potable water. The aquifers that would be used for CO₂ storage must be deep underground, and the technique of CO₂ injection for this reservoir is similar to those for depleted fields.

While not as nearly as common as injecting CO₂ for EOR, this storage location is being practiced in Sleipner, Norway, North Sea. It was started in 1996 when the CO₂ is directly injected into the deep saline aquifer of 1 km deep under the seabed in the Utsira sand formation. Located in the North Sea at about 200 km off the Norwegian coast, the natural gas field mainly made of methane, which contains 4–10% of CO₂. Another case study for CO₂ storage in the deep saline aquifer is located in the natural gas field in Salah (Algeria), which is estimated to hold a storage capacity of 17 million tons of CO₂.

Coal-bed methane
In this case, injected CO₂ binds to the coal and is sequestered permanently. The CO₂ is injected into coals to exchange with methane. However, this storage location is in the research phase, with no operational projects.

Trapping mechanism
Theoretically, injected CO₂ will be stored by different types of trapping mechanisms such as hydrodynamic/structural/stratigraphic trapping, residual or capillary trapping, solubility trapping, and mineral trapping.

Hydrodynamic/structural/stratigraphic trapping
This trapping mechanism refers to supercritical CO₂ trapped under low permeability caprock. The density of supercritical CO₂ being less than water tended to rise buoyantly until it reaches the caprock. This is also known as viscous fingering. Since the caprock has a greater capillary entry pressure than the buoyancy force, supercritical CO₂ will accumulate in such a structural and stratigraphic feature. Typically, this trapping mechanism may take effect after 20–40 years of injection period, depending on rock type.

The trapping efficiency is determined by the structure of the sedimentary basins, which have an intricate plumbing system by high and low permeability strata that control the flow of fluids through the basin [1]. Anticlinal folds or seal fault block are the most common structural traps found in reservoirs that have held oil and gas for millions of years.

 Injected CO₂ may take millions of years to travel by buoyancy forces up-dip to reach the surface before it leaks back into the atmosphere. This trapping mechanism highly depends on the sealing capacity of caprock, making it a big challenge for site selection [1, 8].

Residual/capillary trapping
As the supercritical CO₂ is injected into the formations, it first displaces brine in a co-current fashion until the injection is stopped. Then, CO₂ will migrate up towards, and brines flows downwards due to density differences and replace CO₂ as a wetting phase. Thus, CO₂ trapped in a small cluster of pores while being saturated by brine. The capillary forces and relative permeability effects will contribute to converting the CO₂ injected into an immobile phase. This is the same concept on how the hydrocarbon was held for millions of years. Typically, this trapping mechanism may take effect hundreds of years post-injection depending on rock type.

Solubility trapping
Solubility trapping is where CO₂ dissolves in brine, just like sugar dissolves in tea. Dissolved CO₂ in brine is denser than brine; thus will sink to the bottom of formation over time, trapping the CO₂ even more securely. The dissolution would increase the density of brine up to approximately 1% compared with the original brine injection depending on rock type.

Mineral trapping
Mineral trapping refers to dissolve CO₂ from the solubility trapping mechanism change into a stable mineral phase via reactions with mineral and organic matter in the formation. For example, the
creation of carbonates as a result of aluminosilicate minerals can be an essential trapping mechanism though the timeline is on the order of 100’s to 1000s of years [11].

**Challenges during CO₂ injections**

**Salt precipitation**

Injection of CO₂ into saline aquifers will cause carbonation of contacted in situ brines, disturbing the chemical equilibrium that had been established over geological time frames between the brine and host rock [12]. As NaCl is the most abundant compound in a saline aquifer, saline formation water will be evaporated when CO₂ is injected into the formation, which gives rise to salt precipitation as it reaches the solubility limit. Recent studies have proven that salt precipitation occurred due to CO₂ injection [13–16].

The precipitated solids reduce the pore space available to the fluids and may block the pore throats, which subsequently hinder any further carbon dioxide injection [13]. This condition will lead to permeability impairment associated with the multiphase flow and chemical reaction between CO₂, brine, and rock. The probability of water trapped in the pore throat in low-permeability water-wet formation is high and will cause permeability reduction [17]. The capillary forces exerted between CO₂ and brine increases the severity of formation damage. Nevertheless, more damage is observed in heterogeneous rocks compared with a homogenous rock because its higher permeability value initiates more substantial precipitation reaction. CO₂ dissolves in the formation of brine, forming carbonic acid that dissolves carbonate mineral [18]:

\[ H_2O + CO_2 \rightarrow H_2CO_3 \] (1)

\[ CaCO_3 + H_2CO_3 \rightarrow Ca^{2+} + 2HCO_3^- \] (2)

\[ CaMg(CO_3)_2 + H_2CO_3 \rightarrow Ca^{2+} + Mg^{2+} + 2HCO_3^- \] (3)

**Scaling effect**

Another issue attributed to CO₂ injection is the potential for scaling caused by incompatibility between CO₂ and the formation of water chemistry. If this problem is not addressed at an early stage, it could severely impact injection operations at the field scale level. CaCO₃ is the most likely scale for carbonate reservoirs with other potential scales such as BaSO₄, SrSO₄, FeCO₃, and FeS. This work also investigated formation damage due to scaling and dissolution. Krumhansl et al. [19] and Egermann et al., [20] stated that brine formation might lead to calcium sulfate precipitation governed by Equation 4.

\[ Ca^{2+} + SO_4^{2-} + xH_2O \rightarrow CaSO_4 \times xH_2O \] (4)

Where \( x \) is representing 0 for anhydrite, 0.5 for hemihydrate, and 2 for gypsum. The high fluid flow rate in the porous media was the main reason for fine migration in individual reservoirs [21]. According to Wojtanowicz et al. [22] and Nguyen et al. [23], the critical flow rate/velocity may suspend fines or force them to move and deposit or bridge in the pore space and/or throat, which subsequently result in pore plugging.

Meanwhile, Miranda and Underdown [21] proposed a unique method for determining critical rate by injecting fluid at a very low injection rate called baseline permeability. The rate is increased in a step-wise manner. After that, the rate is returned to the initial rate (baseline permeability) after each incremental stage. Experimentally derived flow rate and permeability data are converted to the bottom hole and wellhead production rates using completion data and well geometry [21]. In-depth knowledge of flow during CO₂ injection is paramount to evaluate the feasibility of CO₂ sequestration in depleted carbonate field in Malaysia. To simulate the condition, coreflood testing using a multi-rate water-alternating-gas CO₂ (WAG) injection will be introduced to determine the critical rate for the investigated zone.

**Storage development plan (SDP)**

The first step in implementing a detail Storage Development Plan (SDP) for CO₂ sequestration in full-field scale is to conduct an SDP screening study that examines the field history data in terms of production, injection, pressure profile, and drives mechanism. Primary reservoir engineering data such as fluid and rock properties must be taken into account prior to CO₂ injection during the pilot phase.

Integration between full-scale projects and research is needed to gain knowledge and experience required for injectivity study. Typically, laboratory coreflooding experiments are designed and conducted to instigate further the efficiency of the CO₂ injection of the selected field. The assumption used for the laboratory coreflooding tests is that the reaction observed in laboratory analysis will be dominant in the long-term process.
The experimental study is essential to understand what would happen at a microscopic level, hence, could significantly reduce the cost of injectivity study. If successful results are obtained from these coreflooding experiments, the study plan may proceed further in the field scale application but to be conducted in stages to minimize uncertainties such as pilot project and finally full-scale level if the project is found to be economically robust [24].

Methods

Experimental apparatus

The maximum allowable pressure and temperatures for the core-flooding apparatus are 15,000 psi and 200 °C, respectively. This equipment capable of performing a wide range of core-flooding related research within one stand-alone integrated system. The system is applicable for all main multiphase flow experimental studies such as EOR related research, formation damage analysis, and CO2 injectivity study. The most significant wetted metal parts of the apparatus are made of highly resistant material such as Hastelloy and stainless steel to cater to the corrosive effect of CO2 and H2S. This feature enables the equipment to be rust free and corrosion resistant even under high-temperature environments with high chloride concentration.

The separation and collection system

The separation and collection system comprised of a pump, sensors, and separator. The effluent is passing through back pressure regulator (BPR) into separator due to gravity inside the specially designed separator (Figure 1). After the BPR, the system is at ambient condition. Material balance calculation is taken into account to back-convert the produced volumes to reservoir conditions. Furthermore, for the experiments carried out during this research, the produced CO2 is sucked by a wet test meter while the effluent is collected in an air-tight jar.

The data logging and monitoring system

All components of the apparatus are connected to a computer with appropriate data logging and monitoring software installed. The whole system, including the sensors, the PID controllers, the injection and collections pumps, and the pneumatic valves, are monitored and controlled using this computer [25]. The data logging was conducted with time-steps as short as one second.

The material

Fluids

Three different types of fluids were used during various stages of the experimental work. These fluids included dead brine (synthetic brine with no dissolved gas content), supercritical CO2, and distilled water. The CO2 gas applied was of a bottled high-purity grade (99.99%) carbon dioxide. The synthetic brine was prepared in the preparation room using distilled water and sodium chloride (NaCl).

Core samples

Native carbonate core samples from Field A were used in this experimental work. They were chosen from several varieties to cover a range of carbonate types present in such geological structures. The core samples could be divided into three different groups based on their porosity and permeability range.

Experimental procedure

All the core plugs used for this experiment followed a standard operating procedure that has been outlined in the literature. Apart from the
coreflooding experiments which constituted the core of this research, there was a variety of other experimental and preparation work carried out as well, which included [25]. Briefly, core cleaning and drying was done after core sample selection at warehouse to remove any possible hydrocarbon, salt, and drilling mud residues. The porosity, permeability, grain density was then measured, including water permeability. Sample saturation in dead brine under vacuum condition was done to restore their original wettability.

This article practiced the application of WAG injection of CO$_2$ concept rather than simultaneous water and CO$_2$ injection schemes. A higher percentage of CO$_2$ were stored by residual trapping under WAG injection based on a study conducted by Juanes et al. [26] and Pentland et al. [27]. The first step to prepare coreflooding experiment was to measure the dry weight, length, and diameter of the carbonate core plug. The sample was immersed in synthetic brine at ambient condition and vacuumed until no bubble was released from synthetic brine. The selected core plug was then pressurized for 36 h at 1,000 psi in accumulator to remove the gas trapped in the pore space. After the measurement of wet weight of the saturated core plug, we calculated the PV based on Eq. (5), where $r$ and $L$ is the core plug radius and length in cm, respectively. In this calculation, water density is assumed to be 1 g/cc.

Pore Volume,

\[
PV \ (cc) = \frac{\text{Saturated weight (g)} - \text{Dry weight (g)}}{\text{Brine density (g/cc)}}
\]  

(5)

Bulk Volume,

\[
BV \ (cc) = \pi \times r^2 \times L
\]  

(6)

Porosity,

\[
\theta \ (%) = \frac{PV}{BV} \times 100
\]  

(7)

Water permeability ($K_w$) must be established prior to WAG injection of CO$_2$. The chosen brine rates for this purpose are at 0.5 ml/min, 1.0 ml/min and 1.5 ml/min. After $K_b$ was obtained, it was advisable to use $\Delta P$ corresponding to flow rate of 1.0 ml/min as a baseline. CO$_2$ was flowed from top to bottom of core holder at identified flow rate and constant volume until it reached a stabilized $\Delta P$. The core was then saturated back by injecting the brine from bottom to top of core holder at constant flow rate of 1 ml/min to restore initial reservoir condition of the core. This process was repeated until CO$_2$ flow rate unable to reach stabilized $\Delta P$. The effluent and gas produced at each step were collected for material balance calculation and interpretation.

Saeedi [25] suggested to place the core-holder containing sample in vertical position where CO$_2$ is injected from top to bottom while brine is injected from bottom to top to eliminate the effect of gravity segregation (underrun or override of the injected fluids). Core holder and brine accumulator were located inside an oven at in-situ reservoir pressure and temperature. CO$_2$ accumulator was placed outside the oven at ambient condition to prevent CO$_2$ expansion for safety purpose. Corrected flow rate injection was incorporated using Charles Law equation to reduce the
experimental error produced during laboratory analysis (Figure 2).

Result and discussion

Lithofacies, mineralogy and petrography

Thirty feet of core samples were selected from mouldic limestones intervals in Field A. The faunal assemblages indicate a range of environment deposition from protected/reefoid to deeper marine. The samples are classified as highly porous boundstone samples made of coral, algal, and calcareous skeletal particles cemented by sparry calcite according to Dunham [28]. Minor recrystallization was observed in the samples.

X-ray diffraction analysis (XRD) were conducted to analyze the mineral composition in the carbonate samples (Table 1). Results show that calcite is the main mineral with average of 80 wt%, followed by total clay with 7 wt% and other accessory mineral such as quartz, K-Feldspar, plagioclase, dolomite, siderite and pyrite. Total clay consists of mostly by illite, mixed-layer I/S, kaolinite, chlorites, and smectite.

Only three samples were selected for injectivity study, namely sample no 1, sample 2, and sample 3. Pre and post petrography analyses was conducted to evaluate the changes in rock texture and mineral composition due to the CO2 injection. Table 2 shows the comparison of petrographic properties between pre and post CO2 injection for the three analyzed samples. No significant changes observed in texture and morphology in all analyzed samples. The porosity changes shown to be very minimal.

**Porosity and permeability**

Routine core analysis was conducted on all samples to determine the porosity, permeability and grain density. Properties of each core sample for both pre and post injection were tabulated in Table 3. The net confining stress (NCS) stress applied in the measurement were 800 psi to represent the ambient condition and 1200 psi for in situ NCS. No major differences between data obtained from this two NCS which indicated the carbonate samples is less stress sensitive. Nitrogen was used as a conduit through a porosimeter and validated using the calculation in Equation 7. Porosity range for this set of samples is from 26% up to 31% and the permeability (K klink) ranges is in between 55 and 1800 mD. The grain density 2.71 g/cc obtained in all samples which reflect the high calcite content.

**Pre and post measurements**

Pre and post RCA measurement were also conducted on the three samples to determine the potential changes caused by the CO2 injection. Table 4 shows the comparison RCA result between pre and post CO2 injection.

**Water analysis post CO2 injection**

Synthetic brine composition was formulated based on a brine sample from the B-3 Field due to the unavailability of brine composition from A Field. A and B fields shared common aquifer support; hence brine composition from B Field was appropriate representative to be used for A field. The brine sample from the B Field was collected at surface separator during the production testing.

The authors decided to filter the brine to remove cloudy appearance. Brine salinity reading for the B-3 field was 21223 ppm, based on the total NaCl constituent in the synthetic brine, while the total dissolved solid was 22789 ppm. Tables 5 and 6 present the composition of effluent collected at each incremental flow rate used in this experiment. From the cation data, Sample 1 and Sample 3 show similar trend where Ca2+ increase up to 400 mg/l which suggest a potential of calcite dissolution as results from the reaction with injected CO2 and brine. However, there no evolution of Ca2+ ion in brine from core flooding of sample 2. This is not expected since there is reduction of porosity in the sample 2, which indicates

| Table 1. Mineral composition. |
|-----------------------------|
| No | Quartz | Plagioclase | K Feldspar | Calcite | Dolomite | Siderite | Pyrite | Total Clay | TOTAL |
|----|--------|-------------|------------|---------|----------|---------|-------|------------|-------|
| 1  | 0.3    | 2.1         | 2.1        | 80.1    | 7.3      | 0.9     | 1.4   | 5.8        | 100   |
| 2  | 0.4    | 2.3         | 3.1        | 84.9    | 1.3      | 0.8     | 1.8   | 5.4        | 100   |
| 3  | 0.5    | 2.2         | 2.2        | 81.7    | 3.4      | 0.9     | 1.6   | 7.5        | 100   |
| 4  | 0.4    | 2.5         | 2.8        | 80.0    | 3.0      | 1.0     | 2.1   | 8.1        | 100   |
| 5  | 0.5    | 2.5         | 2.8        | 80.5    | 4.3      | 1.0     | 1.7   | 6.7        | 100   |
| 6  | 0.4    | 2.6         | 2.3        | 81.7    | 3.8      | 0.9     | 1.6   | 6.6        | 100   |
| 7  | 0.7    | 2.7         | 2.9        | 77.2    | 5.2      | 1.0     | 2.1   | 8.2        | 100   |
| 8  | 0.4    | 2.4         | 3.2        | 81.5    | 3.5      | 1.0     | 1.7   | 6.2        | 100   |
| 9  | 0.4    | 2.5         | 2.6        | 78.2    | 5.9      | 0.9     | 1.8   | 7.7        | 100   |
minimal dissolution. Therefore, data in sample 2 needs to be carefully used as there is potential for measurement error. In anion composition, the bicarbonate seems to be increased with time. This suggests more CO2 dissociate as more cycle of CO2 injection take place.

Figures 3 and 4 indicate cloudy solutions for sample 1 and 2 post-CO2 injection as an indication of geochemical reaction between carbonate cores plugs and injected supercritical CO2.

| Table 2. Comparison of petrographic analysis on pre-post CO2 injection for the analyzed samples. |
|---------------------------------------------|---------------------------------------------|---------------------------------------------|
| Sample | Carbonate classification; Dunham [28] | Sample condition | Pre-CO2 injection | Post-CO2 injection | Pre-CO2 injection | Post-CO2 injection | Pre-CO2 injection | Post-CO2 injection |
| % Calc skeletal grains | 10.5 | 3.2 | 15.0 | 7 | 5.0 | 2.5 |
| % Calcite spar (>10 m) | 19.2 | 29 | 36 | 33 | 25 | 37.5 |
| % Microspar (>10 m) | 45.4 | 43.9 | 25 | 34 | 50.1 | 38.7 |
| % Dolomite | 0.8 | 1 | 0.85 | 1 | 0.4 | 0.66 |
| % Clay | 0.1 | 0.1 | 0 | 0 | 0 | 0 |
| % Modal porosity | 26 | 25 | 23 | 21 | 21 | 22.4 |
| Carbonate grain types | coral, foraminifera and red algae | coral, foraminifera and red algae | coral, foraminifera and red algae |
| Authigenic cements types | minor dolomite, sparry calcite | minor dolomite, sparry calcite | minor dolomite, sparry calcite |
| Clay Dominant pore types | non observed | non observed | non observed | Intercrystalline, Dissolution |
| Pre-post comparison | No significant change in texture | No significant change in texture | No significant change in texture |
| or morphology | or morphology | or morphology |

| Table 3. Routine core analysis result. |
|-----------------------------------|-----------------------------------|-----------------------------------|
| No | Grain density (g/cc) | Kair (mD) | Klinkenberg (mD) | Porosity (%) | Grain density (g/cc) | Kair (mD) | Klinkenberg (mD) | Porosity (%) |
| 1 | 2.71 | 213 | 172 | 26 | 2.71 | 208 | 158 | 26 |
| 2 | 2.71 | 123 | 81 | 29 | 2.71 | 122 | 78 | 29 |
| 3 | 2.71 | 1824 | 1769 | 30 | 2.71 | 1744 | 1691 | 30 |
| 4 | 2.71 | 56 | 43 | 31 | 2.71 | 55 | 43 | 31 |
| 5 | 2.71 | 457 | 435 | 30 | 2.71 | 416 | 395 | 30 |
| 6 | 2.72 | 244 | 115 | 30 | 2.72 | 240 | 107 | 30 |
| 7 | 2.71 | 273 | 211 | 27 | 2.71 | 271 | 204 | 27 |
| 8 | 2.71 | 2046 | 1986 | 29 | 2.71 | 1958 | 1901 | 29 |
| 9 | 2.71 | 1084 | 1045 | 28 | 2.71 | 1046 | 1008 | 27 |

| Table 4. Comparison pre-post CO2 injection (RCA). |
|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
| Sample | | | | | | | | |
| Sample condition | Pre-CO2 injection | Post-CO2 injection | Pre-CO2 injection | Post-CO2 injection | Pre-CO2 injection | Post-CO2 injection | Pre-CO2 injection | Post-CO2 injection |
| Net confining stress (psi) | 800 | 1200 | 800 | 1200 | 800 | 1200 | 800 | 1200 |
| Kair (mD) | 158 | 208 | 158 | 208 | 158 | 208 | 158 | 208 |
| Klinkenberg (mD) | 104 | 158 | 104 | 158 | 104 | 158 | 104 | 158 |
| Porosity (%) | 26 | 25 | 26 | 25 | 26 | 25 | 26 | 25 |
| Sample 2 | | | | | | | | |
| Sample condition | Pre CO2 Injection | Post-CO2 Injection | Pre CO2 Injection | Post-CO2 Injection | Pre CO2 Injection | Post-CO2 Injection | Pre CO2 Injection | Post-CO2 Injection |
| Net confining stress (psi) | 800 | 1200 | 800 | 1200 | 800 | 1200 | 800 | 1200 |
| Kair (mD) | 90 | 122 | 90 | 122 | 90 | 122 | 90 | 122 |
| Klinkenberg (mD) | 50 | 78 | 50 | 78 | 50 | 78 | 50 | 78 |
| Porosity (%) | 29 | 27 | 29 | 27 | 29 | 27 | 29 | 27 |
| Sample 3 | | | | | | | | |
| Sample condition | Pre CO2 Injection | Post-CO2 Injection | Pre CO2 Injection | Post-CO2 Injection | Pre CO2 Injection | Post-CO2 Injection | Pre CO2 Injection | Post-CO2 Injection |
| Net confining stress (psi) | 800 | 1200 | 800 | 1200 | 800 | 1200 | 800 | 1200 |
| Kair (mD) | 1233 | 1744 | 1233 | 1744 | 1233 | 1744 | 1233 | 1744 |
| Klinkenberg (mD) | 1005 | 1691 | 1005 | 1691 | 1005 | 1691 | 1005 | 1691 |
| Porosity (%) | 30 | 27 | 30 | 27 | 30 | 27 | 30 | 27 |

Table 7 shows the values of the in-situ reservoir condition parameter used during experiments that were carried out under high temperature and high pressure. The pressure and temperature values used were the same as the in-situ reservoir conditions of the A field.

A preliminary modelling study had been conducted, and the CO2 plan to be injected at a depth of 5556 ft from seabed. Net overburden pressure were calculated, and value obtained will be used
Table 5. Cation analysis of brine effluent pre and post-CO₂ injection.

| Sample 1 | Brine effluent post-CO₂ injection rate (ml/min) | Sodium, Na (mg/l) | Calcium, Ca (mg/l) | Magnesium, Mg (mg/l) | Potassium, K (mg/l) | Strontium (mg/l) | pH at 27°C |
|----------|-----------------------------------------------|-------------------|-------------------|---------------------|-------------------|-----------------|-------------|
| Initial Brine | 8748 | 0 | 0 | 3 | 0 | 8.5 |
| 4 | 8732 | 130 | 10 | 40 | 8 | 8.3 |
| 6 | 7236 | 146 | 13 | 61 | 12 | 8.3 |
| 8 | 5748 | 138 | 9 | 36 | 9 | 8.3 |
| 10 | 6916 | 330 | 13 | 64 | 12 | 8.4 |
| 12 | 8588 | 60 | 23 | 148 | 14 | 8.2 |
| 14 | 6275 | 467 | 30 | 107 | 15 | 8.2 |
| 16 | 6778 | 177 | 29 | 110 | 26 | 8.2 |

Table 6. Anion analysis of brine effluent post CO₂ injection.

| Sample 1 | Brine effluent post-CO₂ injection rate (ml/min) | Sulphate, SO₄ (mg/l) | Chloride, Cl (mg/l) | Bicarbonate, HCO₃ (mg/l) | Carbonate, CO₃ (mg/l) |
|----------|-----------------------------------------------|---------------------|-------------------|------------------------|----------------------|
| Initial Brine | 1080 | 12475 | 470 | 13 |
| 4 | 900 | 11790 | 1761 | 0 |
| 6 | 870 | 10371 | 965 | 0 |
| 8 | 660 | 8319 | 848 | 0 |
| 10 | 840 | 10224 | 939 | 0 |
| 12 | 900 | 12377 | 991 | 0 |
| 14 | 690 | 9589 | 1056 | 0 |
| 16 | 750 | 9833 | 1082 | 0 |

in the core flooding experiment (Table 7). Assumptions employed for this calculation are overburden gradient at one psi/ft and Poisson ratio equivalent to 0.3.

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| 16 | 6778 | 177 | 29 | 110 | 26 | 8.2 |

Coreflooding experiment

A total of 3 sets of cyclic-coreflooding experiments were conducted during laboratory work. The water
A permeability experiment was conducted prior to the major experiments to confirm the liability of the core plug for the analysis. To test this theory, an incremental water rate of 0.5, 1.0, and 1.5 ml/min were flowed at approximate total of 100 min or until it reaches stabilized differential pressure. Then, a decremented water rate of 1.5, 1.0, and 0.5 ml/min was injected to ensure the differential pressure at the incremental rate is equivalent to the decremented rate.

As a baseline, the brine was flowed at 1 ml/min throughout the coreflooding experiments to maintain rock integrity. The brine flow rate of 0.5 ml/min was not chosen as a baseline since it will create unnecessary chemical reaction that will affect the CO₂ injection parameter at later stage, due to longer exposure time during baseline brine injection.

Other than permeability obtained using porosimeter, the permeability of brine can be calculated during this laboratory work. To do this, the differential pressure at each stabilized flow rate of 0.5, 1.0, and 1.5 ml/min was plotted on a different graph of delta pressure versus brine flow rate. The slope of this graph is measured using the best-fit line, and the value was applied in the Darcy equation. Figure 5 shows differential pressure versus brine rate graphs, which eventually gives permeability value of 147, 76, and 1659 mD. These numbers are considered correct since these
data are not far off from value obtained in Table 4 for sample 2.

It should be understood that the samples presented in this section were all carbonates and, with slight variations, all of conventional 1.500 inch (38.1 mm) diameter but with varying lengths. It is worth noting that the relationship between core plug permeability and the differential pressure between the inlet and outlet of the core is inverse proportional to each other.

**Relationship between differential pressure and CO₂ injection**

Based on the CO₂ phase diagram, the requirement for CO₂ to be in the supercritical condition is to ensure the pressure and temperature of CO₂ must be above 1071 psi and 31 °C, respectively. Supercritical CO₂ takes up much less space and diffuses better than in gas state through the tiny pore spaces in rocks, which lead to the higher CO₂ storage volume.

Presented in Figure 6 is the comparison study between three core plug samples of different permeability. We highlight that the water permeability of sample 1, sample 2, and sample 3 are approximately 147, 120 mD, ϕ = 26%; sample 2 Kₐir = ~120 mD, ϕ = 29%; sample 3 Kₐir = ~1700 mD, ϕ = 30%.

![Figure 5. Differential pressure (Delta P) versus brine rate cross-plot for the three analyzed samples. Sample 1 Kₐir = ~200 mD, ϕ = 26%; sample 2 Kₐir = ~120 mD, ϕ = 29%; sample 3 Kₐir = ~1700 mD, ϕ = 30%.](image-url)
plugs causing the ΔP to continue to raise until the flow rate was unable to reach stabilized ΔP. Presented below is delta P vs. pore volume of sample 1, showing approximately 10–20 pore volume during CO2 injection (Figure 7). The authors only presented one plot since the frequency at which the authors recorded the delta P during the injection implementation for other core plugs are almost similar.

Since the highest permeability core plug possesses the lowest differential pressure, it will be easier for the core plug to reach its critical rate as it gets easier for the solid particle to disperse from the main body and build up at the core plug outlet. Meanwhile, for the lower permeability core plug, the integrity of the structure is firmer and stronger that makes it more difficult for these rocks to react with supercritical CO2 and travel through the coreflood system.

Plotted in Figure 8 is the resemblance pattern of concentration ratio versus pore volume for all three core plug samples. As previously mentioned, the highest permeability samples carry the lowest concentration ratio as compared to the lowest permeability core. To the best knowledge of the author, such experimental results as those presented here could be due to the fact that core plug sample 1 experienced the greatest geochemical reaction as compared to sample 3 at the same pore volume measurement.

Despite the fact that there may not be a valid universal relationship defined between the injection rate and distance travel by the solid particles to block the pathway, experimental analyses through this research has confirmed that as critical rate gets higher, the chances at well level are the supercritical CO2 will crack the formation at a shorter distance than that of lower critical rate CO2 injection, as shown in Figure 9.

It can be deduced that based on three different native core plugs that were used in this experiment, solid particles of sample 3 that consist of the highest permeability of all core plugs have the ability to allow the crack to happen further away for the wellbore.

To the best knowledge of the author, to date, the data presented in this study is the most comprehensive discussion on the critical rate analysis. In high permeability samples and samples susceptible to formation damage (e.g. fines migration, mineral dissolution/precipitation, etc.) the critical rate (represented during the experiments by the differential pressure across the samples) may decrease as the cyclic CO2-brine injection proceeds. However, it is predicted that the extent of any reduction in the injectivity would stabilize after several injection cycles.

The author concluded that for the formation that has permeability range between 100 to 1000 mD, the critical rate would be around 10 ml/min, in which the disperse particle could travel up to 0.19 m around the wellbore before the formation breaks. Meanwhile, for rock permeability of 1000 to 10000 mD, and 10 to 100 mD, the critical rate would be 8 ml/min and 12 ml/min, respectively, with the ability to travel up to around 0.2 and 0.22 m.

### Effect of CO2 injection on chemical reaction at wellbore condition

Several other phenomena have been encountered, such as scaling due to brine chemistry, mineral dissolution, and precipitation post-CO2 injection. These chemical changes would have a significant impact on the determination of the critical rate. However, the rate of chemical reaction occurs at less speed compared to physical changes. That is the reason why the critical rate is observed for all samples.

Continuous CO2 WAG experiment must be conducted without stopping to avoid an increase in the chemical reaction between fluid-rock, which

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**Figure 6.** Delta P vs. sCO2 injection rate; sample 1; \( K_{\text{air}} = \sim 200 \text{ mD}, \phi = 26\% \); sample 2; \( K_{\text{air}} = \sim 120 \text{ mD}, \phi = 29\% \); sample 3; \( K_{\text{air}} = \sim 1700 \text{ mD}, \phi = 30\% \).
would result in a serious error during the interpretation of the lab experiments. It is necessary to point out that the data plotted in Figure 10 indicated that the precipitation occurs due to the dry-out phenomenon caused by the evaporation of NaCl solution if the reader observed reduction in sodium cation effluent collected after the CO₂ injection.

These solid particles eventually blocked the pore throats and directly hindered any fluid movement inside the core plug. Reduction in ions indicates that precipitation takes place, while an increase in ions in the effluent represents the dissolution of minerals.

The limitations of this experiment in terms of geochemical analysis are:
1. The effluent was tested at an ambient condition that might not be representative of reservoir conditions.
2. The pH reading during coreflooding experiment (reservoir condition) rather than after the back-pressure regulator (ambient condition).
3. It is also recommended to utilize a bigger brine accumulator in order to cater to up to 300 pore volume of brine and/or CO₂ injection because the increase in reaction time between the core plug and brine would definitely impact the output data that is required for injectivity study.

Porosity and permeability impairment due to injection of CO₂

Figure 11 shows a comparison between the permeability and porosity calculated for the three samples, pre and post CO₂ injection at 1200 psia net confining stress. Observation on the physical characteristic of core plugs can be concluded that the permeability and porosity of the core plug sample have been destroyed due to geochemical reactions occur between the physical core plug and the acidic solution, and physical reaction between the flow rates and the core plug. The porosity and permeability have reduced from its original value after exposed to supercritical CO₂ injection regardless of net confining stress.
Conclusion

In conclusion, the recent work on the CO₂ injection into a carbonate field in offshore Malaysia demonstrated the existence of fines migration, scale formation, and salt precipitation after the core was subjected to supercritical CO₂ at multi flow-rate. Considering these issues and challenges associated with injectivity, this study recommended the optimum injection rate prior to field scale injection.

Key points of the findings can be summarized as follows:

1. The formation heterogeneity around the wellbore will not result in formation damage due to fines migration if CO₂ is injected within a safe injection limit. Injection below the critical limit by taken into account permeability rock is crucial to avoid formation damage around the wellbore. Nevertheless, looking at the geochemical perspective, if the injection rate is too slow, it will cause more chemical reactions between fluid and rock due to a longer time taken to for the CO₂ to flow within the core plug.

2. Several other phenomena have encountered, such as scaling due to brine chemistry, mineral dissolution, and precipitation from post-CO₂ injection. These chemical changes would have a significant impact on the determination of the critical rate. However, the rate of chemical reaction occurs at less speed compared to physical changes. That is the reason why the critical is observed for all samples. There are other samples that are not
representative of this report have shown that the chemical reaction could occur at a higher speed than physical changes of the core that make it difficult for the author to conclude the critical rate analysis. Having said that, the core plug will not be suitable for this study. The author also has observed that it is at utmost important to run the coreflood experiment in continuous order without stopping to avoid an increase in a chemical reaction between fluid-rock, which would result in a serious error during the interpretation of the lab data.

3. The experimental approach presented here seems to explain the critical rate observed in a heterogeneous Malaysian limestone. The experimental methodology might as well be used for prediction of critical rate for CO₂ sequestration in other areas.

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Data access statement
Due to the nature of this research, participants of this study did not agree for their data to be shared publicly, so supporting data is not available.

Disclosure statement
No potential conflict of interest was reported by the authors.

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