

# Optimization of Pore Compressibility Lab-based measurements; Case Study of Supercritical CO<sub>2</sub> Exposure and Dissolution

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**Abstract**— Injection of carbon dioxide into underground geological formations with proper structure is a common approach performed, such as, for geo-sequestration purposes. During this operation, host rock may then undergo a series of chemical and petrophysical alterations including dissolution and changes in pore structure. Subsequently, knowledge of potential variation in reservoir rock properties against these alterations is of great interest in planning for the geological storage of CO<sub>2</sub>. Amid these properties is the pore compressibility as a key factor in understanding of rock geomechanical behaviors. However, lab-based analysis of pore compressibility is a challenging procedure with ongoing issues. The present work provides a comparison between two different approaches for lab-based measurement of pore compressibility. The work compares usage of sister plugs and a single one to determine the effect of dissolution and applied pressure on pore compressibility in limestone samples exposed to supercritical CO<sub>2</sub>. For this purpose, core-flooding experiments with pore-water rich in CO<sub>2</sub> were carried out to resemble real reservoir condition while the samples were undergone pore compressibility measurements with the above-mentioned approaches. In the end, it was revealed that using a single plug will provide more precise results compared to the sister plugs, however, attention must be paid to a number of critical parameters. In fact, injection and confining pressures are potential to cause pore closure, and so change the internal structure, during pre-injection pore compressibility measurement. Moreover, in case of using sister-plug approach, it is important to screen the samples based on their pore type and internal structure,

**Keywords**— Limestone, pore volume compressibility, single and sister plugs, supercritical CO<sub>2</sub>.

## I. Introduction

Injection and storage of CO<sub>2</sub> in subsurface geological reservoirs has been proposed as an attractive option to deal with the production of greenhouse gases and subsequent unpleasant global warming [1]. This approach is highly tied

with the knowledge of regional geological settings and capability of the hosting rock to maintain injected CO<sub>2</sub> for long-term, and so, to prevent potential societal consequences.

Dissolution trapping of CO<sub>2</sub> in formation water, residual trapping of a two-phase fluid (formation water and CO<sub>2</sub>) in pore network, stratigraphic trapping of a buoyant CO<sub>2</sub> plume beneath a sealing rock layer, and mineral trapping by precipitation of a mineral phase involving the injected CO<sub>2</sub> are among the well-known potential mechanisms for trapping gaseous or supercritical CO<sub>2</sub> (SC-CO<sub>2</sub>) [2].

With this regard, conventional carbonate hydrocarbon reservoirs are considered as attractive targets for mineral trapping mechanisms due to the reactivity of calcite with CO<sub>2</sub> [3]. However, due to the acidification of pore fluid caused by CO<sub>2</sub> dissolution in water ([4]; [14]), dissolution and destabilization of the host rock may occur as a result of chemical reactions triggered by in-situ pressure and temperature conditions [5].

According to previous work, pore collapse and mechanical compaction are likely to be occurred in carbonate rocks hosting the injected CO<sub>2</sub> that creates dissolution channels, and so, raise the porosity and permeability [6]. Analysis of these phenomenons in real reservoir conditions and through lab-based experimental procedures is highly sensitive should be conducted with attention to the details [7].

Pore volume compressibility (C<sub>p</sub>) is among the major parameters that can be varied during injection of CO<sub>2</sub>. In fact, C<sub>p</sub> is the most common type of compressibility measured to evaluate the behavior of porous media that shows the significance of a good knowledge from its variations. However, laboratory determination of C<sub>p</sub> usually encounters some difficulty mainly due to acquiring accurate data as well as a laborious and expensive sample preparation procedure. Moreover, there may also be cases in which it is difficult to obtain an accurate value of C<sub>p</sub> due to the lack of an appropriate representative core sample. To deal with these issues, several

empirical relationships have been proposed as an alternative for lab-based estimation of  $C_p$  (e.g. see [8-13]).

The present work provides a comparison between two different lab-based approaches to determine the effect of dissolution on  $C_p$  in selected limestone samples exposed to supercritical  $CO_2$ . This includes conducting experiments on sister plugs and a single plug to measure the variation in  $C_p$ .

## II. Theoretical background

**Compressibility:** is defined as the fractional change of volume ( $V$ ) with respect to the applied stress ( $\sigma$ ), per unit of volume, at a constant temperature (see Eq. 1):

$$C = -(1/V)(dV/d\sigma) \quad \text{Eq. 1}$$

In general, three types of compressibility can be distinguished including rock matrix compressibility, rock bulk compressibility, and pore compressibility [14]. Moreover,  $C_{pc}$  and  $C_{pp}$  are two different types of compressibility named as “formation compaction coefficient” and “effective pore compressibility” by [15], respectively. They define the response of pore volume to variation in confining pressure or pore fluid pressure, in which are different in terms of the applied stress and pore pressure.

**$CO_2$  storage in carbonate reservoirs:** Evaluation of subsurface geological formations for  $CO_2$  storage purpose is based on their storage capacity, injectivity and confinement for a secure  $CO_2$  sequestration. As a result, depleted or existing oil and gas reservoirs are of among practical candidates for  $CO_2$  storage purpose, especially, carbonate reservoirs in which are estimated to contain 60% of global conventional and unconventional HC resources [16]. As a note to present the importance of depleted hydrocarbon reservoirs in geological storage plans, estimated storage capacity in oil and gas fields are from 675-900 Gt $CO_2$  [17].

Carbonate reservoirs are known with strong heterogeneous structure and are therefore hard to be characterized. Heterogenic properties are rooted in complex diagenetic, reactive, depositional, and deformational processes. Vugs and natural/induced fractures are important aspects in carbonate rocks that originate these heterogenic properties [18]. As a result of this heterogeneous structure, permeability may vary over three to four orders of magnitude at a given porosity; moreover, pore type varies significantly in the reservoir in terms of shape and size. Carbonate pore-size distribution can range from micro porosity to vugs. It has been shown that pore volume compressibility ( $C_p$ ) of carbonate reservoirs is highly sensitive to a range of petrophysical properties including pore type [19].

**Chemical reactions:** During injection of  $CO_2$  into a subsurface carbonate reservoir,  $CO_2$  will react with formation water and promote dissolution of the rock [20]. During this process,  $CO_2$  will be dissolved in formation water through a series of chemical reactions represented as below:

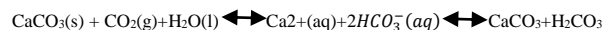


Here, gaseous  $CO_2$  reacts with water ( $H_2O$ ) and forms carbonic acid ( $H_2CO_3$ ). The final product will form bicarbonate ions ( $HCO_3^-$ ) that can be further separated and form carbonate ions. Subsequently, the release of  $H^+$  ions will reduce the pH in carbonic acid which makes it a weak acid with a pH normally between 3-5. Note that pH in the carbonic acid is around 3 at typical storage conditions while it usually

decreases at conditions where  $CO_2$  is more soluble in water. Solubility of  $CO_2$  in water depends on water salinity, pressure, and temperature.

Limestone are mainly composed of calcium carbonate ( $CaCO_3$ ), named as calcite, that is a highly reactive mineral and soluble in water. During  $CO_2$  injection calcite reacts with carbonic acid through the chemical the reaction below:

Main theoretical background behind the present work is



based on the fact that the above-mentioned chemical reactions are potential to vary the pore compressibility of limestone. However, analysis of the variation in pore compressibility must be carried out with enough attention to details as presented in following sections.

## III. Methodology

Experiments were conducted on three selected limestone samples, drilled from a block of outcrop rock sample, each 3.8 mm in diameter and averagely 71 mm long.

The samples own average initial porosity of 16% and average permeability of 1.2 md. They were scanned using available CT scanning facilities at pixel size of 150  $\mu m$ .

Core samples were first placed in ultrasonic bath, cleaned with methanol using a soxhlet extraction apparatus for 24 hours, and then dried out in a vacuum oven at 100 °C for 24 hours. Next, they were saturated with a solution of 6 wt% sodium chloride (NaCl) salt in deionized water using a desiccator.

Supercritical  $CO_2$  was then flooded into the samples using the apparatus shown in Figure 1 with injection pressures of 3800 psi and 4800 psi within confining pressures of 4500 psi and 5800 psi. Eventually, the sample was left in the applied condition for 10 hours.



Figure 1: A view of the set-up used for injection of SC- $CO_2$  into selected limestone core samples.

Next,  $C_p$  was estimated according to the procedure shown in at four confining pressures of 500, 1000, 2000 and 4000 psi. This was conducted through combined sister- and

single-plug approaches to compare their overall pros and cons.

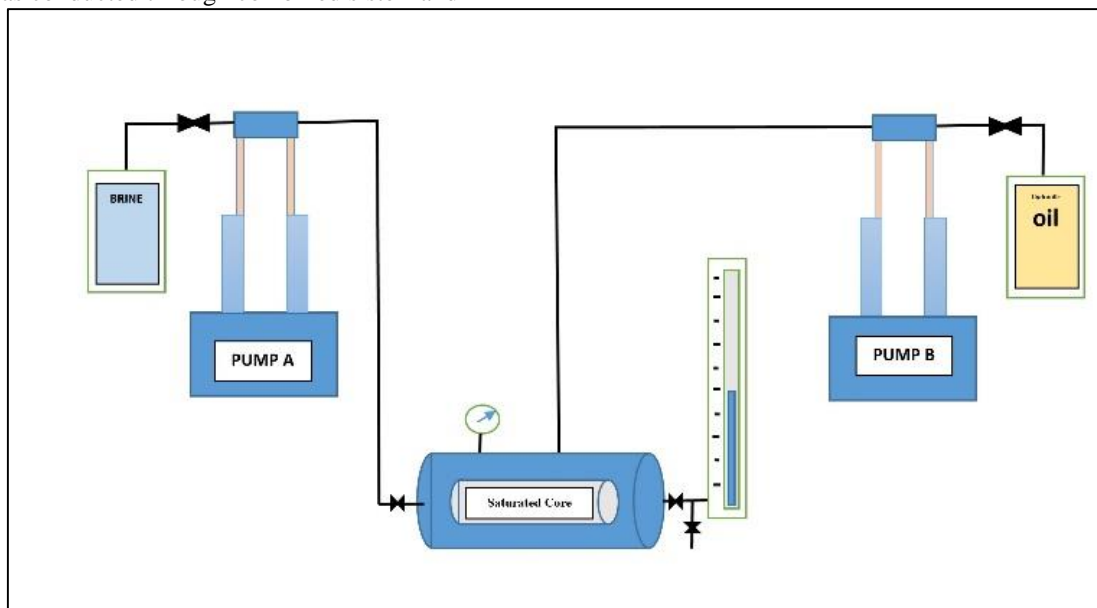


Figure 2: Schematic procedure for measurement of pore volume compressibility at this study.

#### IV. Results and Discussion

Figure 3 shows a view of samples and their pore-structure pre- and post-injection of SC-CO<sub>2</sub> that evidences the dissolution and offers almost similar pore structure for samples#1 & 2 and a different one for sample#3.

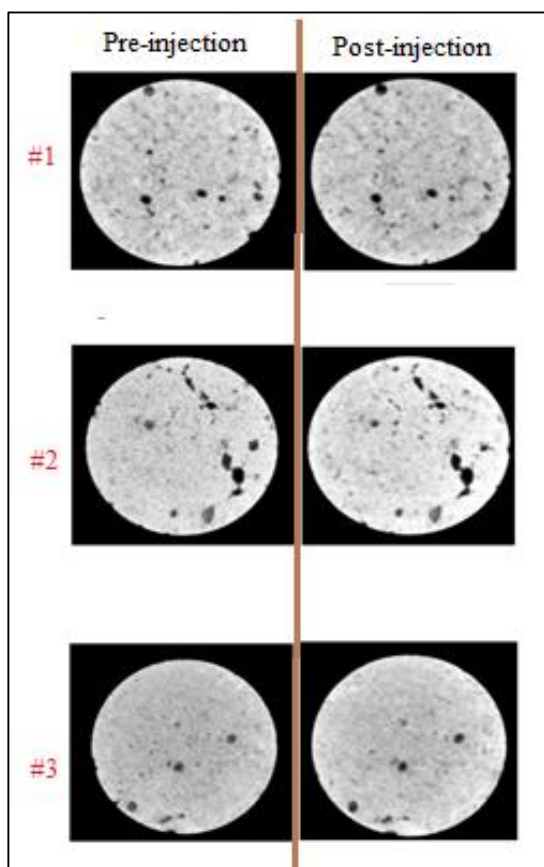


Figure 3: Pre- and post-injection CT-scan images of samples used in the present study showing their pore structure.

Figure 4 shows the measured  $C_p$  for sample#1 (after injection of SC-CO<sub>2</sub> at 3800 psi) and the one measured for sample#2 as a sister-plug for sample#1. It offers a 12% reduction in  $C_p$  of sample#1 due to injection of SC-CO<sub>2</sub>.

In addition to the results presented at Figure 4, estimated  $C_p$  for sample#2 after injection of SC-CO<sub>2</sub> at 3800 psi is shown at Figure 5 that allows a comparison between sister- and single-plug approaches.

According to Figure 5, despite the fact that at net pressures lower than 2000 psi both approaches provide almost similar estimation for variation of  $C_p$  after injection of SC-CO<sub>2</sub>, but at net pressure of 4000 psi greater variation of 15% in  $C_p$  of sample#2 was observed that is greater than the one estimated for sample#1 through sister plug approach. This difference could be due to the pore damage imposed to sample#2 during measurement of  $C_p$  prior to injection of SC-CO<sub>2</sub>.

To investigate the effect of injection pressure and pore structure, sample#3 was flooded with injection pressure of 4800 psi. Results of  $C_p$  measurement on sample#3 before and after injection of SC-CO<sub>2</sub> is shown at Figure 6 that suggests a different behavior of the pore structure during the test and possibility of pore collapse in pre-injection test. According to the CT images, sample#3 shows a lower magnitude of dissolution compared to the other two samples. However, the observed average 18% of variation in  $C_p$  for samples#3 at net pressure of 4000 psi points to a significance loss of pore strength due to possible effect of injection pressure.

Figure 7 a cumulative presentation of the results achieved from all three samples. According to the results, sample#3 cannot be used as a sister plug for sample#1 to identify the variation of  $C_p$ , here due to injection of SC-CO<sub>2</sub>. This shows the significance of paying attention to the test conditions including confining and injection pressures as well as pore structure as crucial parameters in selection of sister plug approach.

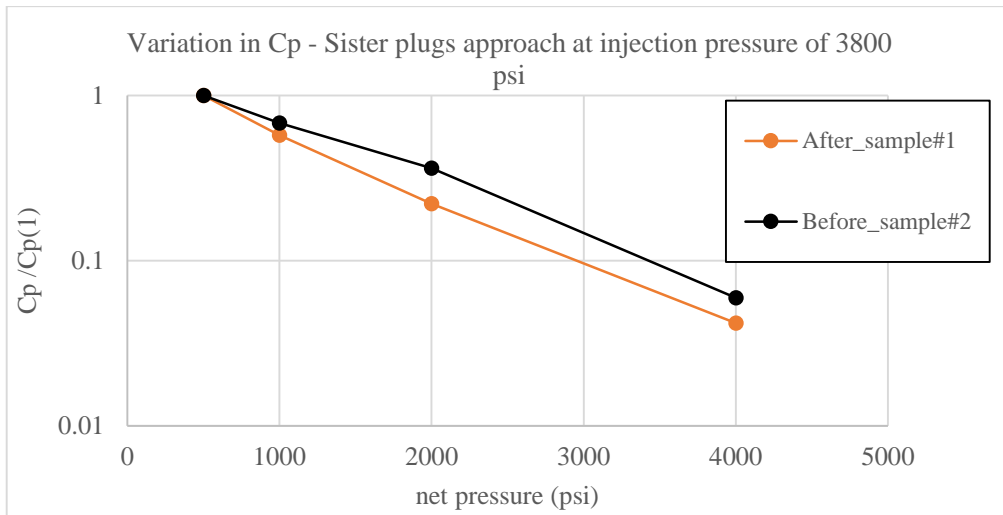


Figure 4: Variation of  $C_p$  in sample#1 (post-injection of SC-CO<sub>2</sub>) compared to non-treated sample#2 as its sister plug.

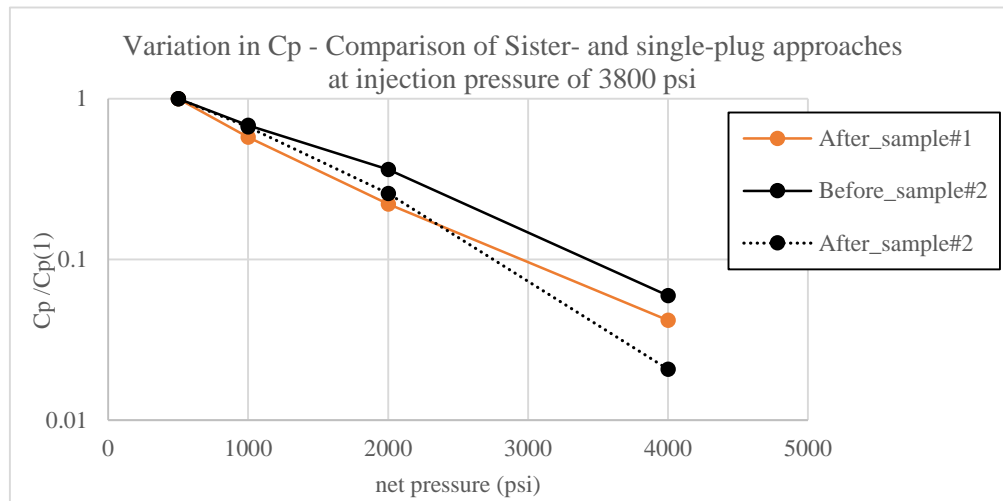


Figure 5: Variation of  $C_p$  in sample#1 and sample#2 after injection of SC-CO<sub>2</sub>; comparison of sister- and single plug approaches.

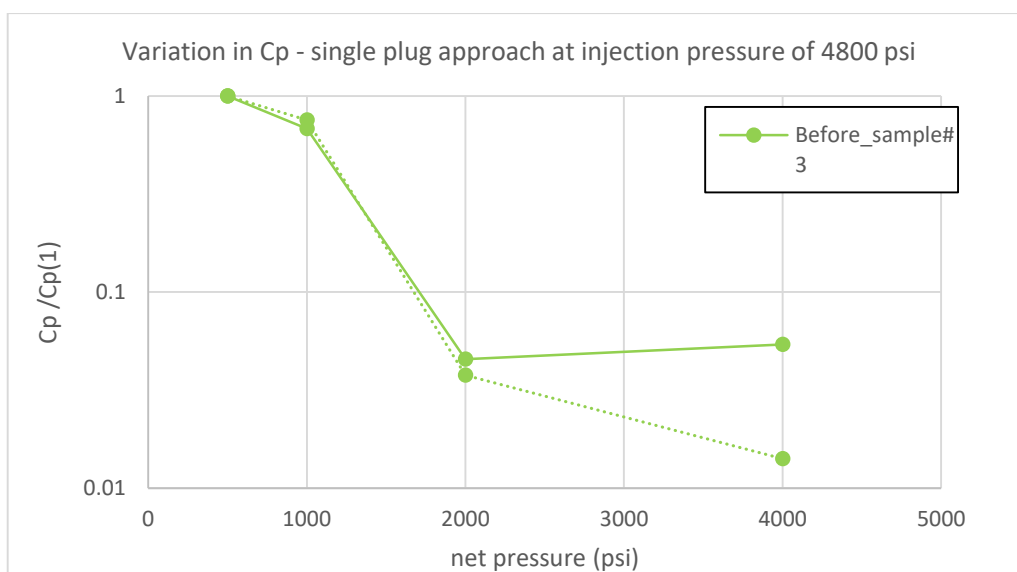


Figure 6: Variation of  $C_p$  in sample#1 and sample#2 after injection of SC-CO<sub>2</sub>; comparison of sister- and single plug approaches.

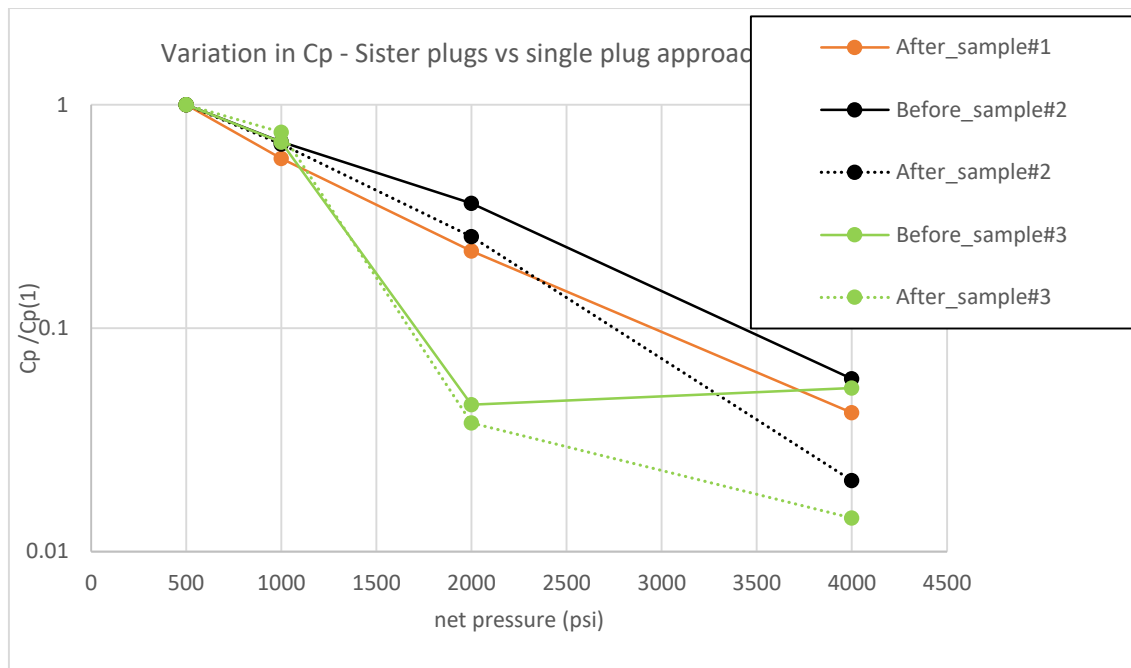


Figure 7: Variation of  $C_p$  in sample#1, sample#2 and sample#3 after injection of SC-CO<sub>2</sub>; comparison of sister- and single plug approaches in various pore structure and injection pressures.

## V. Conclusion

The results show the significance of both single- and sister-plug approaches in determination of  $C_p$  variation, here due to supercritical CO<sub>2</sub> (SC-CO<sub>2</sub>) injection.

There is an inherent inaccuracy in the single plug approach mainly because of pore damage that occurs in  $C_p$  measurement before the test. Moreover, it was revealed that the use of a sister sample for the estimation of variation in pore compressibility depends on the pore structure of the rock sample.

In the case of investigating the effect of SC-CO<sub>2</sub>, it is recommended to take care of the injection pressure and its effect on reducing the overall pore strength.

In the end, in case of the availability of data on rock structure and mechanical behavior of rock samples, the use of the sister-plug approach is recommended to measure the variation in pore compressibility with a certain percentage of error.

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### Conflict of Interest

The authors declare that there are no conflicts of interest.

### Compliance with Ethical Standards:

This article does not contain any studies involving human or animal subjects. References

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