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Comparative analysis of capillary threshold pressure measurement techniques: mercury intrusion porosimetry vs. innovative measuring tool for carbon storage

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Threshold pressure is a critical parameter in evaluating the capillary sealing capacity of rocks, especially in reservoir and caprock studies related to CO₂ storage and hydrocarbon recovery. In this research, we conducted a comparative analysis of two methods for measuring threshold pressure: the traditional mercury intrusion porosimetry (MIP), which operates under different fluid dynamics, and a new gas-water/brine system (e.g., CO₂-water) using novel equipment. The new apparatus employs techniques to directly measure the threshold pressure by monitoring the displacement of water by gas, such as CO₂, in core samples. This system is designed to more accurately replicate *in-situ* subsurface reservoir conditions by accounting for key parameters such as CO₂-brine interfacial tension, and pressure. By directly measuring the point at which gas begins to displace water from rock pores, it provides a more realistic assessment of the threshold pressure for CO₂ sequestration projects. For example, in the case of Sample 1, the direct measurement under a confining pressure of 15 MPa resulted in a threshold pressure of 0.4 MPa. In contrast, mercury intrusion data converted for CO₂ drainage estimated the threshold pressure at only 0.1 MPa. Similarly, for Sample 2, the direct measurement yielded a threshold pressure of 0.24 MPa, while the MIP method indicated a significantly lower threshold of 0.05 MPa. These discrepancies underscore the limitations of mercury intrusion when applied to CO₂-water/brine systems. Underestimation of threshold pressure can have significant consequences for the design and safety of CO₂ storage projects, as this parameter is essential to assess the sealing efficiency of the caprocks and determine the storage capacity.

KEYWORDS

capillary threshold pressure, CO₂ sequestration, mercury intrusion porosimetry (MIP), caprock integrity, CO₂ injection

1 Introduction

The rise in global warming is related to increased CO₂ emissions in the atmosphere (IPCC, 2007). The urgency to mitigate climate change has forced advancements in Carbon Capture and Storage (CCS) technologies, focusing on the geological sequestration of carbon dioxide (CO₂) (War and Arnepalli, 2021). This involves injecting CO₂ into underground geological formations

such as saline aquifers, depleted oil and gas fields, and deep coal seams (Callas et al., 2022). A fundamental aspect of ensuring the effectiveness and safety of these storage methods is understanding the dynamics of capillary pressure, which significantly influences CO₂ containment within these geological formations (Hu et al., 2017). Capillary pressure affects the movement of fluids within porous media, determining how CO₂ interacts with brine and other fluids present in the formations (Al-Menhali and Krevor, 2016). Understanding these interactions is crucial for predicting the long-term stability of stored CO₂ and assessing potential leakage risks, which can undermine the effectiveness of CCS as a climate mitigation strategy (Shojai Kaveh et al., 2016). The importance of capillary threshold pressure in the context of CO₂ storage cannot be overstated, as it fundamentally influences the containment and structural integrity of geological storage sites (Song and Zhang, 2013). Capillary threshold pressure is the minimum pressure required to allow the non-wetting phase (CO₂ in the case of sequestration) to enter the pore spaces of a caprock, overcoming the capillary forces that favor the wetting phase (typically brine) (Krevor et al., 2015). This parameter is critical in determining the effectiveness of caprock as a seal to prevent the upward migration of stored CO₂, thereby ensuring the long-term security of the storage site (Naylor et al., 2011). Understanding and accurately measuring capillary threshold pressure are essential for predicting CO₂ storage capacity and containment security (Iglauer, 2017). If the threshold pressure is too low, there is a risk that CO₂ could leak through the caprock, compromising the integrity of the storage system and potentially leading to environmental concerns (Ostrowski and Uelker, 2007). Therefore, extensive research is necessary to assess the properties of caprocks and their ability to maintain effective seals under varying geological conditions. On the contrary, a high threshold pressure indicates a robust caprock that can effectively trap CO₂ (Tan et al., 2024). There are several methods on how to measure this parameter, most commonly for fast estimation of the capillary threshold pressure mercury intrusion porosimetry (MIP) is used (Lohr and Hackley, 2018) but that does not correspond to the actual value due to many reservoir parameters most significant being the confining pressure (Boulin et al., 2013), and MIP may not actually represent the core sample in case there are small scale heterogeneities (Brandimarte et al., 2017). Unlike mercury, which is a non-reactive and non-wetting fluid that does not chemically interact with rock surfaces, CO₂ exhibits distinctive IFT and wettability traits when it encounters water/brine and rock minerals. While some methodologies include the standard or residual approach which record the brine/water production at the outlet, although some studies use combination of these methods that highlight innovative practices for measuring this pressure and its implications for storage capacity and safety as they introduce a fast and accurate method to measure threshold capillary pressure under representative conditions, which is crucial for realistic assessments of caprock performance (Egermann et al., 2006) but still there is a margin of error that can be the delay in recording the pressure right at the movement when the fluid starts to be displaced. Furthermore, some studies explore how capillary threshold pressure affects the estimation of CO₂ column heights in depleted gas fields, which is vital for maximizing storage capacity (Naylor et al., 2011). Moreover, the wettability of the caprock, which can vary significantly under different conditions of pressure, temperature, and salinity, also affects the capillary threshold pressure (Al-Yaseri et al., 2016). Studies also show that changes in wettability can lead to variations



in storage capacity and containment security, emphasizing the need for thorough characterization of caprock properties in CO₂ sequestration projects (Alkan et al., 2010; Arif et al., 2016). In summary, capillary threshold pressure is a key parameter in ensuring the effectiveness and safety of CO₂ geological storage. Accurate measurement and understanding of this pressure, along with caprock wettability, are crucial for designing and operating secure CO₂ storage facilities. Thus in this study a similar approach (Zuta et al., 2022) has been adopted but a completely new system is designed and instead of predicting the threshold pressure by the flow at the outlet (offset of water/brine) the pressure is directly recorded which indicates the threshold of the particular sample. We have also performed a comparative analysis of these two methods for determining threshold pressure; the conventional mercury intrusion porosimetry (MIP) using Autopore V 9600 porosimeter, and a newly developed gas-water/brine system using novel instrumentation which directly measures threshold pressure in core samples. The actual intrusion of CO₂ displacing water/brine is more realistic than mercury intruding the core sample for storage conditions. By measuring the exact point at which gas starts to displace water from the rock pores, offers a more practical evaluation of threshold pressure, particularly relevant for CO₂ sequestration projects. Although mercury intrusion can provide a rough estimation of the threshold pressure, a more accurate assessment requires using techniques that simulate real subsurface conditions, such as the gas-water/brine system.

2 Experimental methodology

2.1 Sample preparation

Two core cuttings were procured from a proposed CO₂ storage well site in Poland, each measuring 31 mm in length and 22.3 mm in diameter as shown in Figure 1. These dimensions were chosen to ensure compatibility with the high-pressure vessel utilized for capillary threshold measurements. Additionally, reserve samples were extracted from the same core cuts for subsequent analysis

TABLE 1 Porosity and permeability of caprock samples.

Caprock sample	Porosity %	Permeability mD
Sample 1	7.30	0.036404
Sample 2	2.09	0.007668

using mercury intrusion porosimetry (MIP). The permeability and porosity of the core samples were also measured which is given in Table 1.

2.2 Experimental procedure

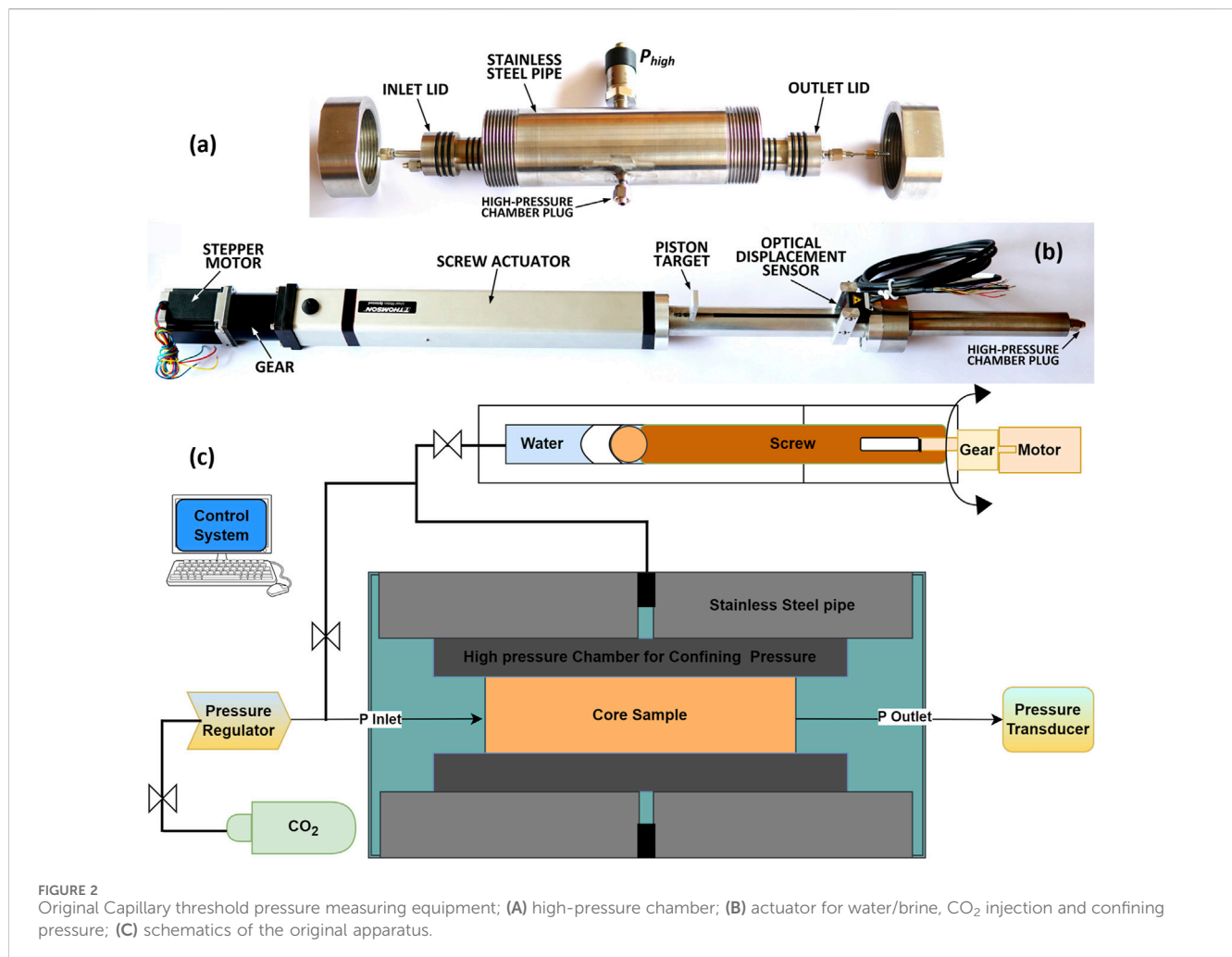
The initial experiments were conducted using the designed apparatus shown in the Figure 2, which consist of high pressure vessel made up of stainless steel pipe where the core is placed and is subjected to a confining pressure using an injection system consist of a piston that is responsible for maintaining the confining pressure using water and also injecting CO₂ in the core sample. The inlet and outlet is connected to pressure transducers. The pressure at the inlet and outlet of the sample is measured using pressure transducers S-10 (WIKA) with a measuring range of 1.6 MPa and a measuring error

of 0.25% of the full scale. At first the core is saturated with water and a confining pressure is applied that represent *in situ* conditions, here the pressure was set to 15 MPa. As Threshold pressure can be defined as the minimum pressure required to instigate fluid displacement in the caprock, a low pressure is applied at the inlet and is left for a period of time and the pressure is increased until any change at the outlet is observed signifying its corresponding threshold pressure. The capillary threshold pressure was determined for these core samples. Later the same rock samples were used to evaluate the capillary threshold pressure using a mercury intrusion porosimeter where the mercury intrusion data was scaled to obtain CO₂ drainage curves using the equation. The threshold pressure was extracted by drawing a tangent through the drainage curve when the saturation is 100% (Vespo et al., 2024). The values from both these methods have been reported.

3 Results

3.1 Original equipment measurements

The graphs in the Figures 3, 4 show the capillary threshold pressure values which are marked in red (the outlet) while the



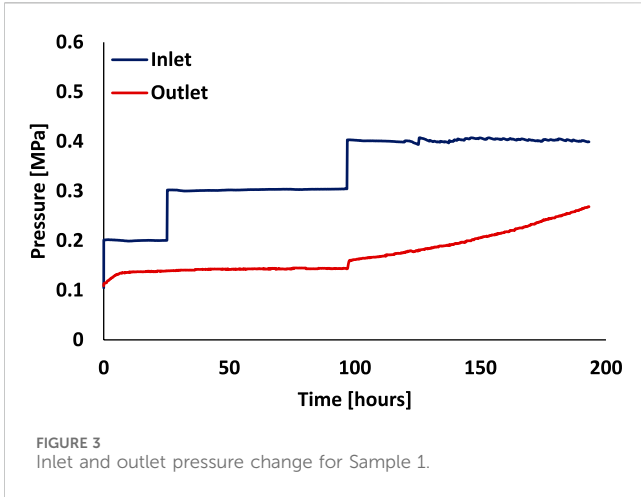


FIGURE 3 Inlet and outlet pressure change for Sample 1.

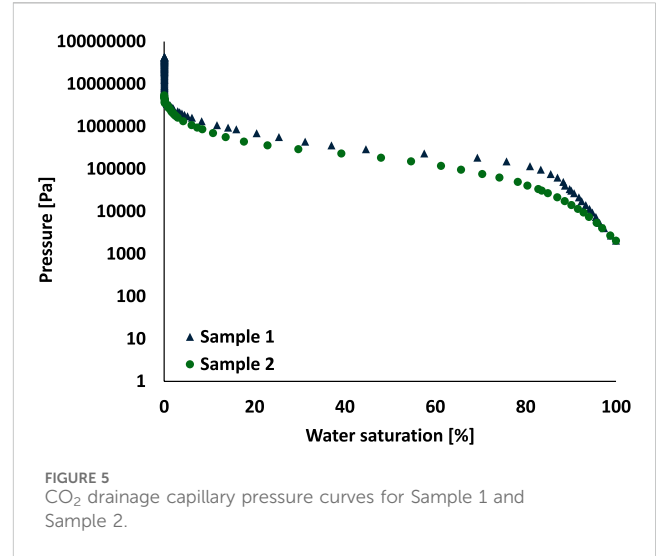


FIGURE 5 CO₂ drainage capillary pressure curves for Sample 1 and Sample 2.

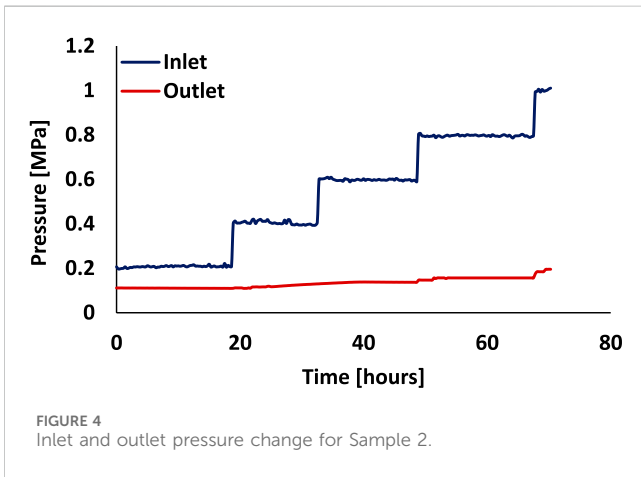


FIGURE 4 Inlet and outlet pressure change for Sample 2.

blue line indicates the pressure increments over time (the inlet), until the threshold pressure is reached. For Sample 1 the threshold pressure is recorded between 0.3 MPa and 0.4 MPa, while for Sample 2 it can be seen that there is a slight rise at the outlet pressure at 0.2 MPa even the porosity and permeability of this sample is lower than Sample 1, so we continue to analyze it for more pressure points over time to avoid uncertainty until there was a significant rise at the outlet. As there was a continuous rise at the outlet, so the initial pressure value between 0.2 MPa and 0.3 MPa was regarded as the capillary threshold pressure of this sample.

3.2 Mercury intrusion porosimetry measurements

The same rock cuttings of the Sample 1 and Sample 2 were subjected to Mercury intrusion porosimeter as it the quickest way to determine the threshold pressure. Autopore V 9600 which is a mercury intrusion porosimeter was used to measure the cumulative intrusion vs. pressure. This mercury intrusion data was scaled to obtain CO₂ drainage curves which represent how water/brine displaces CO₂ from the caprock using Equation 1, Where P(CO₂)

is the CO₂ capillary pressure, P(Hg) is the mercury intrusion pressure, γ (CO₂) is the water/brine-CO₂ interfacial tension (72 mN/m), γ (Hg) is the mercury-air interfacial tension (480 mN/m), while $\cos \theta$ for the initial value is taken as 0 considering fully water wet for initial capillary threshold measurements. From these capillary pressure curve we can extract the threshold pressure which correspond to percolation threshold.

$$P(CO_2) = \frac{P(Hg)\gamma(CO_2) \cos \theta(CO_2)}{\gamma(Hg) \cos \theta(Hg)} \tag{1}$$

Figure 5 shows the CO₂ drainage curves for Sample 1 and 2, the threshold pressure was extracted from these curves using tangent method where a tangent through the drainage curve plateau is extrapolated to pressure axis when the saturation is 100% (Vespo et al., 2024). The tangent method shows the threshold pressure for Sample 1 to be 0.1 MPa, however the direct measured value using the original equipment for this sample was 0.4 MPa under confining pressure of 15 MPa which is 4 times higher than the mercury intrusion value. For Sample 2 the threshold pressure was estimated to be 0.05 MPa while the direct measured value using the original equipment was 0.24 MPa which is 5 times higher than the mercury intrusion value.

4 Conclusion and implications

This study highlights the critical role of capillary threshold pressure in evaluating the sealing capacity of caprocks for CO₂ geological storage. Through a comparative analysis of two measurement techniques, traditional mercury intrusion porosimetry (MIP) and a new gas-water system. we demonstrated significant discrepancies in threshold pressure values. The direct measurements obtained using our new apparatus revealed threshold pressures that were substantially higher than those estimated by MIP, indicating that reliance on mercury intrusion data may underestimate the sealing efficiency of caprocks. The results from Sample 1 showed a threshold pressure of

0.4 MPa compared to 0.1 MPa from MIP, while Sample 2 exhibited values of 0.24 MPa versus 0.05 MPa. These findings underscore the limitations of MIP in accurately representing CO₂-brine interactions and emphasize the necessity for more reliable measurement methods in CO₂ sequestration projects.

The implications of these findings extend beyond mere measurement accuracy; they directly impact the CO₂ height column in geological storage scenarios. A higher capillary threshold pressure indicates a more effective seal, which is crucial for preventing the upward migration of CO₂. If the threshold pressure is underestimated, there is a risk that CO₂ could leak through caprocks, thereby compromising storage integrity and safety. Conversely, accurate assessments suggest that a robust caprock can support a taller CO₂ column, thereby maximizing storage capacity. Furthermore, understanding the wettability characteristics of caprocks is essential, as variations can significantly impact capillary threshold pressure and, consequently, the safety and effectiveness of CO₂ storage sites. This research not only contributes to the field by providing a more accurate assessment technique but also highlights the need for ongoing studies to refine methodologies for evaluating caprock integrity under realistic subsurface conditions. In conclusion, ensuring robust caprock performance is vital for the long-term security of CO₂ storage, and our findings advocate for adopting advanced measurement techniques to enhance the reliability of threshold pressure assessments in future geological storage projects.

Data availability statement

The original contributions presented in the study are included in the article/supplementary material, further inquiries can be directed to the corresponding author.

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Author contributions

AS: Conceptualization, Formal Analysis, Investigation, Writing—original draft, Writing—review and editing. LA: Conceptualization, Supervision, Writing—review and editing. MK: Writing—review and editing. KD: Supervision, Writing—review and editing. SN: Supervision, Writing—review and editing.

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