Geochemical reactions in reservoir rock and capillary sealing capacity of caprock during hydrogen admixing in the underground gas storage complex Rehden

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Abstract. A comprehensive laboratory study evaluated the feasibility of hydrogen storage in the Rehden underground gas storage (UGS) complex, emphasising geochemical interactions and capillary sealing capacity. Core samples from reservoir rocks and caprock were tested under hydrogen and methane atmospheres at reservoir conditions. Geochemical analyses indicated minor mineralogical and fluid compositional changes due to hydrogen, suggesting minimal impact on reservoir integrity. Capillary entry pressure experiments demonstrated robust sealing efficiency of caprock, comparable to methane, with slight variations in interfacial tension and wettability. Geochemical modelling predicted negligible changes in porosity and permeability, affirming the geological suitability of Rehden UGS for hydrogen storage, providing vital insights into the risk assessment for future hydrogen storage operations.

1 Introduction

The European Union (EU) has initiated several directives to decarbonise its energy system, with hydrogen playing an important role in this transition. The REPowerEU strategy, launched in 2022, set forth the objective of producing 10 million tonnes and importing an additional 10 million tonnes of renewable hydrogen by 2030. By 2050, renewable hydrogen is expected to cover approximately 10% of the EU’s energy needs, significantly decarbonising energy-intensive industrial processes and the transport sector.

To achieve these targets, the EU recognises the necessity of developing large-scale hydrogen storage solutions. Underground hydrogen storage (UHS) in geological formations, such as depleted natural gas reservoirs, salt caverns, and aquifers, need to be developed. These formations offer substantial storage capacities and are integrated into the existing infrastructure.

Depleted gas reservoirs present several advantages for UHS. Their known geology, existing wells, and established infrastructure can reduce development costs and timelines. Moreover, their proven sealing capacities and containment properties make them suitable candidates for hydrogen storage. However, the transition from natural gas to hydrogen storage introduces new challenges that necessitate a thorough investigation.

One of the primary concerns is the geochemical reactivity of hydrogen with reservoir rocks and fluids. Hydrogen's high reactivity, particularly as a reducing agent, can alter the geochemistry of a porous media storage. When injected, it may reduce oxidised minerals such as iron oxides, leading to mineral dissolution or conversion. Coupled with pH shifts, a destabilisation of the mineralogical composition of both the reservoir and caprock may occur. Moreover, many of these processes are poorly constrained under subsurface conditions, and their kinetics are often slow but irreversible. However, in recent years, several researchers have set out to investigate the influence of hydrogen on the subsurface during hydrogen storage with interesting results [1-5].

Another critical aspect is the caprock's sealing capacity under hydrogen exposure. Factors such as hydrogen-induced embrittlement, changes in wettability, and alterations in interfacial tension can affect the caprock's sealing performance. Therefore, assessing the caprock's behaviour under hydrogen conditions is essential.

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The Rehden underground gas storage complex in Lower Saxony, Germany, serves as an exemplary site for evaluating these considerations. As one of Western Europe's largest natural gas storage facilities, Rehden offers the opportunity to study hydrogen storage in a real-world setting. The facility's infrastructure and well-characterised geology make it an ideal candidate for transitioning to hydrogen storage.

This study aims to assess the feasibility of hydrogen admixing in the Rehden complex by focusing on two critical aspects: geochemical interactions and capillary sealing capacity. Laboratory experiments were conducted on core samples from the reservoir and caprock to simulate hydrogen exposure under reservoir conditions. Geochemical laboratory experiments, including mineralogical assessments and fluid composition measurements, were performed to evaluate potential reactions and property alterations. Capillary entry pressure tests, contact angle measurements, and interfacial tension analyses were conducted to assess the caprock's sealing performance under hydrogen conditions.

By integrating these experimental results, the study provides insights into the geochemical stability and sealing integrity of the Rehden complex under hydrogen admixing. The findings contribute to the broader understanding of UHS in depleted gas reservoirs.

2 Geological Setting

The Rehden Underground Gas Storage (UGS) facility, situated in Lower Saxony, Germany, stands as one of Western Europe's most significant pore storage installations. With a working gas capacity of approximately 4 billion cubic meters, it accounts for about one-fifth of Germany's total gas storage capacity.

Originally developed for natural gas production in the 1950s, the Rehden field was converted into a storage facility in 1993. The storage operations occur at depths ranging from 1,900 to 2,100 meters within the Zechstein Group formations, primarily the Hauptdolomit (Main Dolomite) of the Upper Permian Zechstein Formation.

2.1 Reservoir Rock

The reservoir rocks at Rehden predominantly comprise the Hauptdolomit, characterised by its dolomitic composition. The reservoir exhibits a horst block structure, which, despite being heavily faulted, provides a stable and secure environment for gas storage.

The porosity and permeability of the Hauptdolomit are enhanced by natural fractures and dissolution features, facilitating gas flow. However, the presence of faults emphasises the need to ensure the integrity of the storage system, especially when considering the transition to hydrogen storage, which may interact differently with the geological formations.

2.2 Caprock

At Rehden, the primary caprock consists of anhydrite layers within the Zechstein Formation, known for their low permeability and high sealing capacity. Additionally, red claystone and salt can be found in the caprock. The effectiveness of the caprock is enhanced by its thickness and lateral continuity.

2.3 Suitability for Hydrogen Storage

The geological characteristics of the Rehden UGS facility make it a fitting candidate for the study of hydrogen admixing. The existing infrastructure and historical data provide a solid foundation for assessing the feasibility of hydrogen injection and withdrawal. However, the unique properties of hydrogen require comprehensive laboratory studies.

Understanding the geochemical and geomechanical responses of the Rehden geological formations to hydrogen exposure is essential for ensuring safe and efficient storage. This includes assessing potential reactions between hydrogen and the mineral constituents of the reservoir and caprock and evaluating the storage system's long-term stability under cyclic loading conditions associated with hydrogen operations.

3 Workflow and Methodology

This chapter outlines the methodological workflow employed to assess the feasibility of hydrogen storage in the Rehden Underground Gas Storage (UGS) facility. The focus is on evaluating geochemical interactions and the capillary sealing capacity of the caprock under hydrogen exposure. The workflow encompasses core sample selection, laboratory experiments execution, data interpretation, and geochemical simulation.

3.1 Geological Evaluation

The investigation started with a study of the vast amounts of reservoir data that is available from the storage facility. The dataset comprises a full reservoir model with log data, core descriptions, standard core analyses, thin section data and compositional data. Over the decades, numerous coring campaigns have been conducted.

3.2 Core Sample Selection and Preparation

The study set out by an investigation of the vast amounts of geological data that the operator provided. The geological structure of the reservoir and caprock were evaluated. The reservoir samples were primarily obtained from the Main Dolomite from the Zechstein Formation, characterised by its dolomitic composition and favourable porosity and permeability properties. Caprock samples were extracted from overlying anhydrite and red claystone layers.

Initial analyses included low resolution (35 µm) computed tomography (CT) scans to evaluate sample integrity, identify structural heterogeneities, and confirm the absence of fractures. The samples were cleaned using refluxing Soxhlet extraction with Methanol to remove residual drilling fluids and salt. Subsequently, they were dried in a vacuum oven at 60°C to eliminate moisture content without altering mineralogical properties.

The dried core plugs were cut into standardised dimensions suitable for the laboratory tests (6 x 3 cm). Trim pieces were polished to ensure uniformity for contact angle measurements.

Prepared samples were stored in desiccators under inert atmospheric conditions to prevent any pre-experimental alterations. Before the utilisation of core plugs for experiments, porosity and permeability were measured using standard laboratory methods to ensure representative reservoir conditions. The porosities were determined with a Helium porosimeter at ambient conditions. Permeabilities to brine were measured at reservoir temperature in a biaxial core holder at a representative confining pressure and overburden pressure. Permeabilities were determined at pore pressures that represent the production cycle of the gas storage.

3.3 Capillary Entry Pressure

Two parallel approaches were used to determine the capillary entry pressure of the caprock samples. Firstly, a direct measurement by the step-by-step approach was utilised [6]. This is the main approach followed in this investigation. The core material was stored for several decades; therefore, the consistency of the obtained core plugs was a potential concern. The phyllosilicates in claystone strongly retain water molecules. Over extended periods, the water is released, leading to a significant volume change. This process leads to internal stresses and possible fractures in the rock material. Such fractures make a capillary entry pressure measurement meaningless. A secondary approach was utilised, which determines the capillary entry pressure through the Laplace Equation [7]:

$$P_c = (2\gamma \cos\theta) / r \qquad (1)$$

Where P_c is the capillary entry pressure, γ is the gas/brine interfacial tension (IFT), and r is the pore radius. The pore size distribution was measured at ambient conditions by mercury injection capillary pressure measurements (Autopore IV, Micrometrics). The IFT was measured by the pendant drop method, and the contact angle was determined by the tilted plate method. Both measurements were conducted at reservoir temperature and pressure.

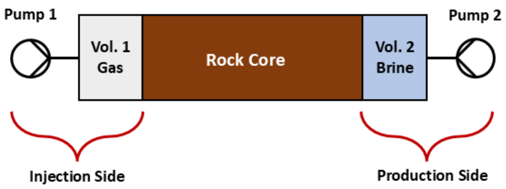

Fig. 1. Measurement principle of the step-by-step approach for the determination of the capillary entry pressure. Both pumps are operating in a constant pressure mode and set to the desired differential pressure. If a fluid flow occurs, it can be detected by the volume change in the pumps.

3.4 Geochemistry

The geochemical changes were investigated in this study by simulating the reservoir conditions under a methane and hydrogen atmosphere for a prolonged period. Reservoir rock samples were characterised, saturated and placed in brine (salinity ca. 300 g/L) while maintaining the respective gas atmosphere for two months at reservoir temperature and maximum storage gas pressure. After one month, the temperature was further increased to accelerate possible geochemical changes. Extensive sampling was conducted before, during, and after the experiment to monitor changes in the brine, gas, and rock composition.

3.5 Geochemical Simulation

Geochemical data obtained from batch experiments were systematically processed to quantify chemical interactions between hydrogen, reservoir fluids, and rock minerals. The experimental results provide essential input parameters for subsequent geochemical simulations.

Geochemical modelling was conducted using The Geochemist's Workbench® software, incorporating experimental data to simulate long-term interactions and predict mineral stability, dissolution, and precipitation reactions under hydrogen exposure. The models assessed the potential impact of geochemical reactions on reservoir porosity, permeability, and fluid composition, enabling a comprehensive evaluation of the reservoir's long-term suitability for hydrogen storage.

4 Experimental Setups and Procedures

All laboratory experiments in this study were conducted at reservoir temperature and pressure (103 °C/ 280 bar) to simulate reservoir conditions and precisely assess the interactions between hydrogen and the geological materials.

4.1 Contact Angle and Interfacial Tension Measurements

The measurement of the IFT and contact angle were conducted in a high-pressure view cell at reservoir conditions. For the contact angle measurement, a brine droplet is placed on the rock sample surface under a hydrogen/methane atmosphere. The cell is rotated at a constant rate of 1 °/sec until the droplet slips on the inclined rock sample surface. At the last moment before slippage, the receding contact angle is recorded.

The wettability of reservoir and caprock samples was assessed by measuring contact angles between the rock surfaces and hydrogen or methane gases. Interfacial tension (IFT) measurements were also conducted to understand the fluid-fluid interactions, which are critical for capillary sealing behaviour.

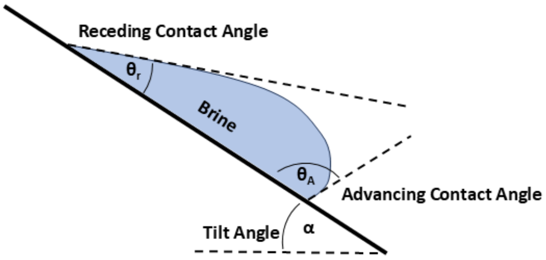

Fig. 2. Measurement principle of the tilted plate method for contact angle determination. A camera records the deformation of the droplet right before slipping. The receding contact angle is used for the capillary entry pressure calculation.

4.2 Batch Reactor Experiments

Batch experiments were conducted to simulate geochemical interactions between hydrogen/ methane gas and reservoir rock samples under reservoir conditions. The rock core plugs were saturated with synthetic reservoir brine under anaerobic conditions. The core samples were placed in high-pressure stainless-steel batch reactors, which were pressurised with hydrogen or methane gas at reservoir temperature and pressure. For one month, the reactors were maintained at 103 °C; afterwards, the temperature was increased to 130 °C for an additional month to accelerate geochemical changes.

Before, during, and after the experiments, rock, fluid, and gas samples were extracted, stored under Argon atmosphere and analysed using Scanning Electron Microscope (SEM-EDS), Ion Chromatography (IC), Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), and Gas Chromatography (GC). This approach documents alterations in fluid chemistry, gas composition, and morphological and mineralogical changes of the rock samples.

4.3 Data Interpretation and Modelling

The experimental data were interpreted using geochemical modelling software to predict long-term interactions between hydrogen and geological materials. Models considered factors such as mineral stability, reaction kinetics, and transport mechanisms to forecast the behaviour of hydrogen within the reservoir and caprock over extended periods.

5 Results

This chapter presents the findings from the comprehensive laboratory experiments conducted to assess the geochemical interactions and capillary sealing capacity of the Rehden Underground Gas Storage (UGS) facility under hydrogen exposure. The results are organised into two main sections: geochemical interactions and capillary sealing capacity.

5.1 Geochemical Interactions

5.2 Rock Integrity

The following photographs show the condition of the samples before and during the experiments (Fig. 3-5). The rock samples from the dolomite and limestone samples exhibited integrity issues during the autoclave experiments (red arrows). Crack formation was observed along the interfaces between different minerals. The anhydrite samples did not exhibit this behaviour.

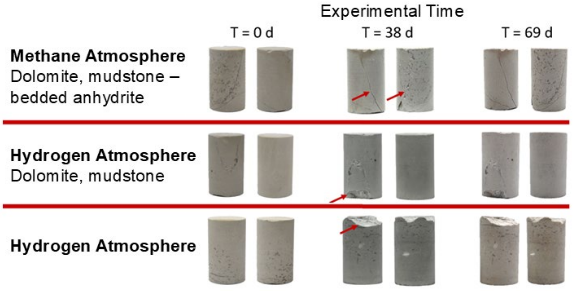

Fig. 3. Three dolomite samples before, during, and after the batch experiment.

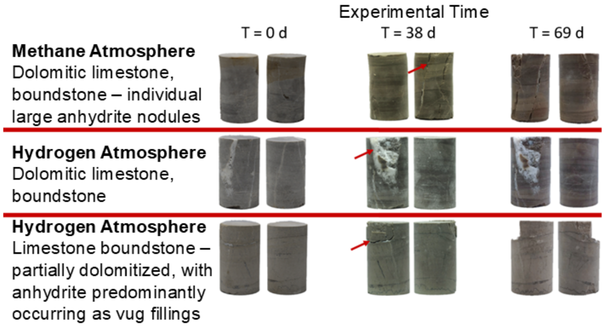

Fig. 4. Three limestone samples before, during and after the batch experiment.

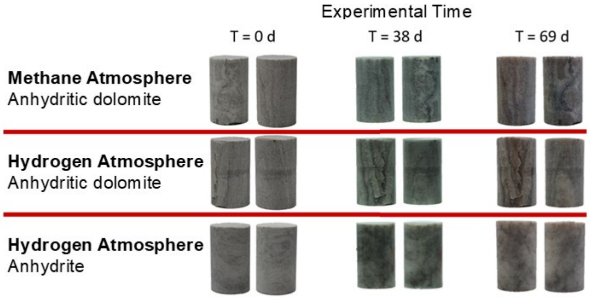

Fig. 5. Three anhydrite samples before, during and after the batch experiment.

5.2.1 *Mineralogical Changes*

Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDX) analyses were conducted on all samples at three intervals: before the experiment, after 39 days, and after 69 days.

Samples from a single autoclave—exposed to either methane or hydrogen gas for varying durations—exhibited consistent mineralogical compositions and no discernible geochemical alterations throughout the 69 days. Variations in accessory minerals such as halite, celestine, and phyllosilicates within a single experimental series are attributed to their heterogeneous distribution and localised occurrence within the samples.

Samples of the same lithology subjected to different experimental conditions did not exhibit significant geochemical variations attributable to the differing gaseous environments (Fig. 6).

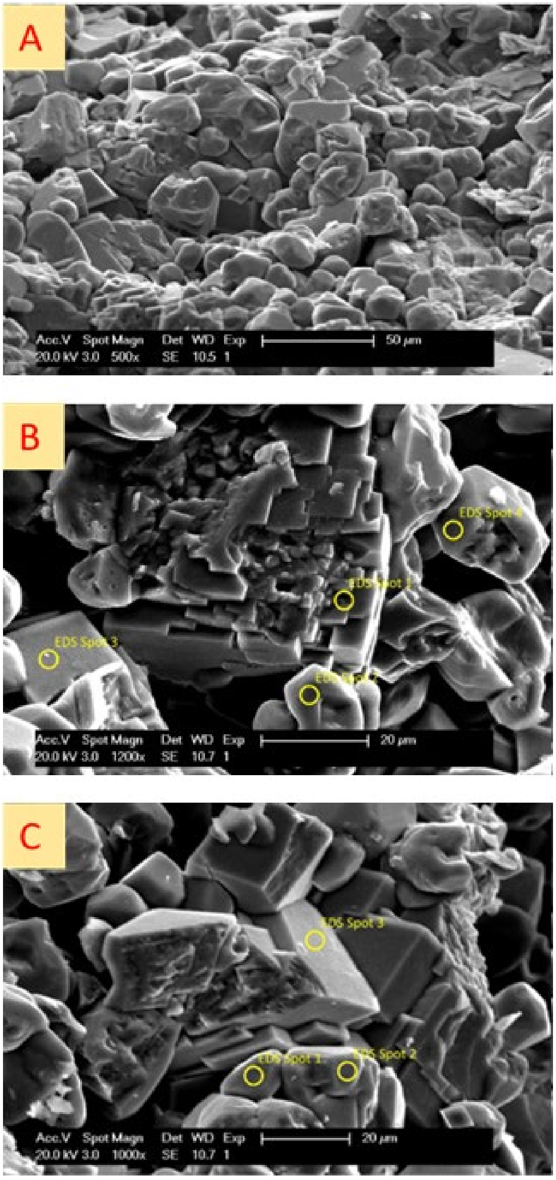

Fig. 6. SEM image of a dolomite sample before and after batch experiment under hydrogen atmosphere for 69 days, (A) before experiment, (B) after 39 days, (C) after 69 days.

Dolomite rock types comprised large framework crystals of calcite and dolomite, overlaid by smaller carbonate crystals and accessory minerals including halides, phyllosilicates, and anhydrite.

Limestone samples demonstrated mineralogical variability of calcite and dolomite, along with minor amounts of halite, anhydrite, celestine, and phyllosilicates.

Anhydrite rock types consisted of large anhydrite framework crystals surrounded by minor amounts of halite, celestine, phyllosilicates, and dolomite. No lithological variations were observed among these samples.

5.2.2 *Fluid Composition Alterations*

Ion Chromatography (IC) analyses of the fluid samples showed minimal changes in ion concentrations, with slight increases in calcium and magnesium ions, likely due to limited dissolution of carbonate minerals. These findings suggest that hydrogen exposure does not lead to substantial geochemical reactions that could compromise reservoir rock integrity and performance.

5.3 Geochemical Modelling

5.3.1 Equilibrium Model

The equilibrium calculations were performed using the software SpecE8 from Geochemist's Workbench®, which relies on the LnL-Worley database [8] for equilibrium constants. In all cases, the calculations were based on the chemical data of the individual samples.

The analyses of water samples enable the assessment of chemical equilibrium between the fluids and the minerals present in the reservoir rocks. The mineralisation or precipitation of solids can be examined to identify changes in reservoir properties concerning porosity and permeability. The geochemical model allows for intermediate and sequential changes in temperature, partial pressure, and gas phase components. With each programmed change, an equilibrium analysis of the fluid system is conducted, determining the relative volumes and constituents of fluids and minerals. Using appropriate petrophysical correlations, modelled changes in mineral volumes can be applied to porosity and permeability models to ascertain relative changes in both parameters.

Although none of the samples showed significant precipitation, the samples exposed to methane exhibited higher mineral saturations ($\log Q/K > 1$) both initially and during the final analysis after 69 days compared to samples exposed to hydrogen. This effect was notably more pronounced in anhydrite samples compared to other rock types. None of the lithologies showed porosity or permeability reductions exceeding 1%.

None of the samples exhibited an increase in porosity beyond their initial values, the results indicate that hydrogen has a distinct dissolution effect on various minerals such as dolomite, quartz, and strontianite. Once the precipitation of these minerals was suppressed by hydrogen, only minerals with low concentrations of iron and silicate remained.

5.3.2 Reaction Path Model – Equilibrium

For the rock samples in this work, the sequence and quantities of reactants were selected to optimally match the modelled outcomes, and sequential analyses were performed on each sample over 69 days. Although multiple components and species can be queried with the React model, the optimal choice for the carbonate reservoir under study was to match the Ca^{2+} and Mg^{2+} concentrations as functions of time. The extent of reaction was determined by relating the number of days elapsed before sampling to the total experimental duration of 69 days.

For the anhydrite sample in methane, a dissolution effect on barite, dolomite, and eventually quartz is observed during a process in which hydrogen partial pressure is increased while methane partial pressure is decreased. In this reaction path, the methane partial pressure is reduced to 222 bar, while the hydrogen partial pressure is increased from zero to 56 bar. When the reaction progress reaches 70 %, the hydrogen partial pressure has reached approximately 2 bar, and barite brine concentrations increase. Similarly, at a reaction progress of 0.8 (hydrogen partial pressure = 7 bar), the mineral volume of dolomite starts to decrease.

5.3.3 Geochemical Model – Long-Term Effects

The equilibrium model for the brine sample in contact with limestone and under a hydrogen atmosphere indicates that Ca^{2+} concentrations tend to increase throughout the 69-day testing cycle. To extend the model as a predictive tool for long-term changes in reservoir properties, reaction rates for the dissolution and precipitation of key reservoir components need to be defined. For the Rehden gas storage facility, calcium represents the common element (species) among the lithologies of calcite, dolomite, and anhydrite.

To model the long-term effects (> 3.5 a), incremental equilibrium steps constrained by the reaction rates of calcite, dolomite, and anhydrite are employed. Critical values for aligning the model with experimentally determined reaction rates include reactive volume, reactive mineral surface area, and reaction rates.

The resulting model is then extended over the study period. The table below shows the development of porosity for different samples that are exposed to hydrogen.

Tab. 1. Projected porosity changes of dolomite, limestone and anhydrite rock under a hydrogen atmosphere for prolonged times.

Sample	Phi_0 [-]	Phi Max [-]	Time (d)	Increase [%]
Dolomite (H2)	0.159	0.161	2849	1.2
Limestone (H2)	0.022	0.022	1323	1.1
Anhydrite (H2)	0.006	0.006	2100	3.2

5.4 Capillary Sealing Capacity

5.4.1 Capillary Entry Pressure Tests

The capillary entry pressure was measured for two caprock samples from the UGS. The measurements were conducted at reservoir temperature of 103 °C, and a pore pressure of 282 bar. The first sample consists predominantly of anhydrite. Brine permeability was determined under reservoir conditions to be 1.93E-21m^2. (1.96E-6 mD) An entry pressure measurement against a methane atmosphere was attempted at increasing differential pressures of 10 to 60 bar, no gas flow was detected over 200 hours. The measured differential pressure (60 bar) is far above the expected maximum pressure differential at the interface between the reservoir and the caprock. It was therefore assumed that the capillary entry pressure lies above 60 bar. A second measurement was conducted with hydrogen gas. Again, no gas flow was detected at 60 bar differential pressure. Therefore, the capillary entry pressure for hydrogen lies well above the expected pressure differential.

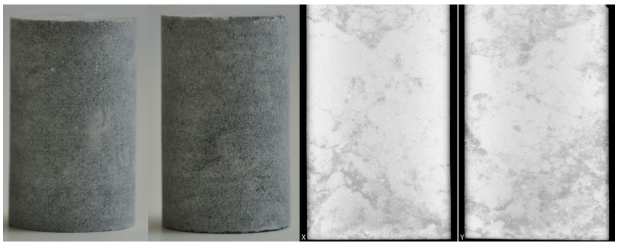

Fig. 7. Anhydrite core plug before capillary entry pressure measurement and CT scan of the same plug for quality control.

Fig. 8. Anhydrite core plug before capillary entry pressure measurement and CT scan of the same plug for quality control.

The second investigated caprock sample is a red claystone that is prone to form fractures due to drying out. Brine was injected into the sample at reservoir conditions. Brine was injected at up to 65 bar differential pressure over 670 hours until a constant fluid flow was detected. The brine permeability of the sample is 6.96E-22 m^2. The first experiment was conducted under a methane atmosphere. A differential pressure of 5 to 25 bar was applied for 950 hours with no detection of gas flow into the core. The same experiment was conducted with hydrogen for 400 hours. Again, no fluid flow was detected. It is concluded that the capillary entry pressure is above 25 bar for both methane and hydrogen. This conclusion was confirmed by the indirect measurement through the Laplace equation (next section).

5.4.2 Indirect Measurement of the Capillary Entry Pressure

Based on the Young-Laplace equation (Eq. 1) the calculated sealing pressure of the red claystone is 76 bar for methane and 82 bar for hydrogen. These results complement findings from the laboratory experiments, which indicated that the sealing pressure of the anhydrite sample exceeds 60 bar.

Calculating the sealing pressure for anhydrite is more complex. The MICP measurements revealed a surprisingly broad pore-size distribution. The largest measured pores ranged between 0.3 and 0.4 µm, corresponding to a sealing pressure between three (3) and four (4) bar. However, laboratory testing showed that the sealing pressure exceeds 25 bar.

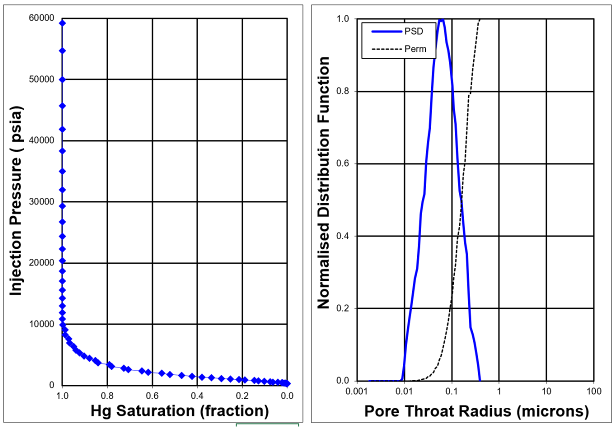

Fig. 9. Injection pressure versus Hg saturation and pore size distribution of the investigated anhydrite caprock sample. During MICP measurements, erroneous data can occur when mercury is initially forced into surface roughness of the rock sample. These surface irregularities are subsequently incorrectly represented in the pore-size distribution. Taking this factor into account, the calculated sealing pressure increases to approximately 10 bar. This value, however, remains below the measured values from the laboratory tests. It is hypothesised that the measured pore sizes primarily reflect isolated pores within the dolomite fraction of the anhydrite sample, which do not influence the sealing pressure.

6 Discussion

6.1 Geochemical Stability of Reservoir Rocks

The experimental results indicate that hydrogen exposure leads to minimal geochemical alterations in the reservoir rocks of the Rehden UGS. Scanning Electron Microscopy (SEM) analysis revealed negligible changes in mineralogy, suggesting that short-term hydrogen exposure does not significantly impact the mineral composition of the reservoir.

Long-term simulation of the geochemical changes points towards a porosity increase between 1.1 and 3.2 % for the dolomite, limestone, and anhydrite over several years. However, the expected changes of the reservoir properties are limited.

6.2 Caprock Sealing Capacity

The direct capillary entry pressure measurements indicate that the investigated caprock material has excellent sealing properties against methane and hydrogen. For the red claystone, this finding was confirmed by the indirect measurement. The indirect measurement for anhydrite showed the limitation of the measurement. The determined capillary entry pressure depends highly on the interpretation of the MICP data.

These observations are consistent with findings from other studies [1-5], which emphasise the importance of caprock integrity in maintaining the structural stability of underground hydrogen storage facilities.

7 Conclusion and Outlook

This study evaluated the feasibility of admixing hydrogen to the underground storage in Rehden, focusing on geochemical interactions and caprock sealing capacity. The findings indicate that the Rehden site possesses favourable geological characteristics for safe and efficient hydrogen storage.

7.1 Key Findings

Laboratory experiments demonstrated minimal geochemical reactivity between hydrogen and the reservoir rocks, primarily composed of dolomite, limestone, and anhydrite. Minor mineralogical changes were detected post hydrogen exposure, suggesting that hydrogen storage would not significantly alter the reservoir's mineral composition.

Capillary entry pressure tests on the anhydrite and claystone caprock samples indicated that the sealing capacity remains excellent under hydrogen exposure for several weeks and at reservoir conditions. The caprock's low permeability and high sealing capacity is critical for preventing hydrogen leakage.

7.2 Outlook and Recommendations

The positive outcomes from this study suggest that depleted gas reservoirs can be repurposed for hydrogen storage, contributing to energy transition goals. Additional investigation of the geomechanical alterations of the reservoir rock and caprock as well as investigation of the gas loss by diffusion.

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