



Research Programme
- Short Call 2022 –
- Final Report -

Lead Applicant Name	Budi Zhao
Host Organisation name	University College Dublin
Project Title	Impact of salt precipitation on hydro-mechanical properties of sedimentary rocks: an X-ray μ CT study
Contract Number	2022-sc-003

This report covers the period 1st November 2022 to project end date **(31/12/2023)**



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2. (a) Project information:

Final project report (max **2500** words, excluding figures and headings):

Executive summary (extended abstract, e.g. for use by policy/other stakeholders):

This project addresses critical challenges in carbon capture and storage (CCS) with profound implications for Ireland's energy sustainability. By developing innovative tools and employing an interdisciplinary approach, the study enhances our understanding of brine drying and salt precipitation dynamics in porous media, a crucial aspect of sustainable CCS.

Integrating advanced imaging techniques, notably X-ray micro-tomography (CT) and microfluidics, the study achieved unprecedented insights into pore-scale dynamics and the consequential impact on rock properties. Key findings, presented at the InterPore2023 conference and published in a high-impact journal, underscore the impact of salt precipitation on permeability, with direct implications for well injectivity. This knowledge contributes to short-term optimization of injection plans and informs the use of surface wettability alteration chemicals to mitigate salt precipitation influences.

In the medium to long term, the project significantly enhances our ability to predict the long-term performance of CO₂ injection wells. Beyond CCS, the study's outcomes extend to engineering applications, including protection strategies for heritage buildings and critical infrastructures in coastal areas, showcasing its broader societal relevance.

These outcomes can help optimize CCS operations and enhance infrastructure resilience. The project's impact on relevant research areas positions it as a catalyst for ongoing advancements, promising enduring contributions to energy sustainability, environmental protection, and the broader field of hydro-chemo-mechanical studies in porous media.



- (i) Results, scientific/engineering targets reached beyond the state of the art and conclusions (please include additional information in Section 3 below)

Summary of Research Outcomes

In the realm of salt weathering study, where theoretical and experimental efforts have abounded, the intricate impact of salt precipitation on rock properties have proven challenging. This challenge arises from the multitude of factors influencing brine drying and salt precipitation, constituting a complex interplay of multi-physics phenomena.

This project focused on investigating the intricate interplay of brine drying and salt precipitation on the hydro-mechanical properties of sandstones, with a particular relevance to potential issues such as reduced injectivity and reservoir damages in carbon geological storage. The combination of advanced imaging techniques, including X-ray micro-tomography (CT) and microfluidics, allowed for unprecedented insights into the pore-scale dynamics and the impact of salt precipitation on rock properties.

In the microfluidic experiments, radial-flow microfluidics chips with pre-defined micron channels (Fig. 1) were fabricated to elucidate the role of capillary backflow in the accumulation of salt precipitation near the injection well. Two distinct types of salt formation were identified through these experiments. Computational fluid dynamics (CFD) simulations further predict that the precipitated salt crystals could lead to a substantial 80% reduction in injectivity, emphasizing the practical implementation of salt precipitation in subsurface reservoirs.

A significant contribution of the project lies in the development of a miniature loading apparatus, designed to continuously monitor the salt weathering of sandstone cores under confinement using X-ray micro-tomography (CT). This apparatus, detailed in Fig. 2, facilitated stress-controlled or displacement-controlled loading processes through a feedback loop system. The inclusion of transparent sample chamber enabled real-time observations through X-ray CT, enhancing our understanding of salt-induced damage.

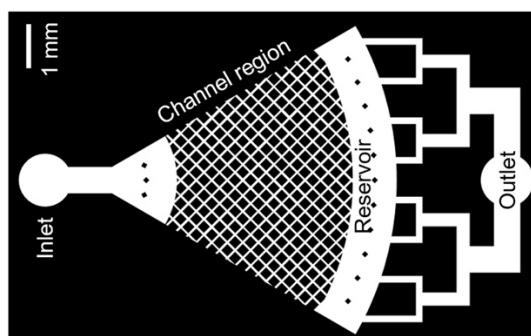


Fig. 1. Schematic plot of radial flow microfluidic chips

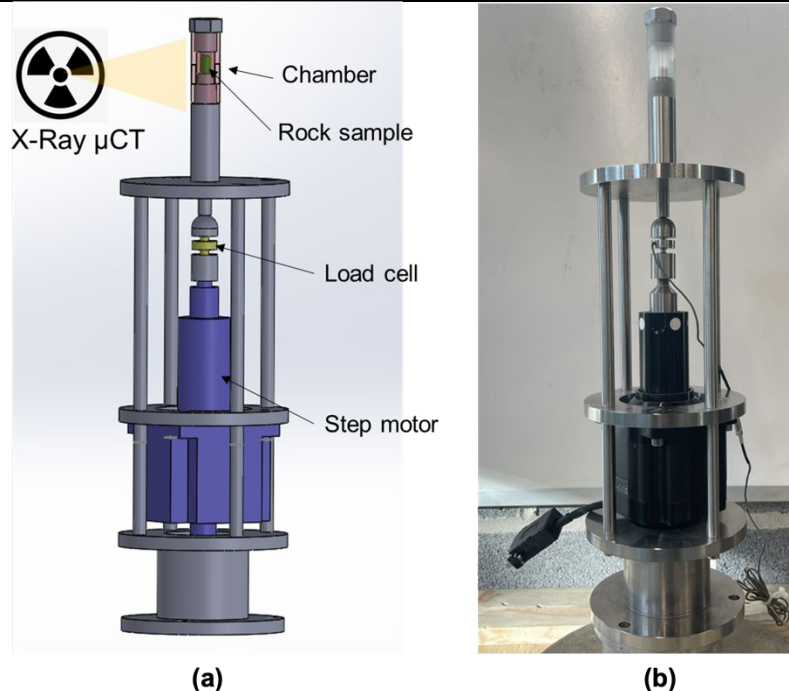


Fig. 2. (a) Schematic plot of loading apparatus. (b) Photo of loading apparatus compatible with X-ray CT

Methodology and Key Results

The project's key results showcase ground-breaking advancements in understanding salt weathering dynamics in sandstone cores under confinement, achieved through the development of a miniature loading apparatus and advanced imaging techniques.

The miniature loading apparatus, a pivotal component of this study, was meticulously designed and fabricated to facilitate continuous monitoring of salt weathering in sandstone cores under confinement using X-ray micro-tomography (CT). As illustrated in Fig. 1, the apparatus has a sample chamber made of strong transparent plastic, allowing seamless X-ray penetration. It incorporates a precision stepping motor for applying vertical compression loads, an LVDT for monitoring displacement, and a load cell for measuring vertical force. The integration of a datalogger and a loading control system, developed using LabVIEW, enables stress-controlled or displacement-controlled loading process via a feedback loop between the stepping motor and LVDT/Loadcell.

To ensure the reliability of results, sandstone samples with a diameter of 6 mm were chosen, striking a balance between sample size and image resolution for monitoring pore-scale dynamics. Four distinct types of sandstone, varying in strength and permeability, were selected for this study. Fig. 3 presents a X-ray CT scan of a small rock core. The image processing procedure includes denoising the raw image using 3D median filter (Fig. 3b), segmenting rock grains (Fig. 3c), and extracting the three-dimensional pore topology (Fig. 3d). We applied an AI-enhanced image segmentation algorithm to extract three-dimensional pore structure. Subsequently, the three-dimensional pore structure was imported into an open-source CFD software, OpenFOAM, to quantify the permeability of rock cores.

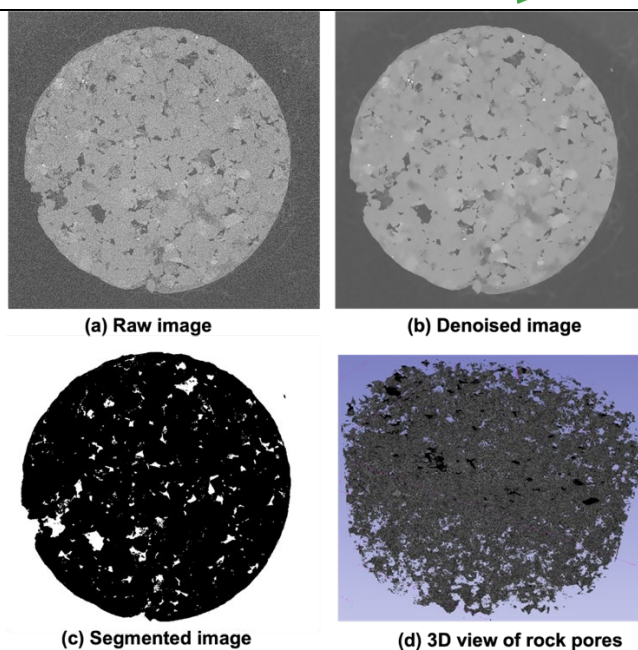


Fig. 3. Image processing of miniature rock cores

The drying test initiates with the application of a constant vertical stress to confine the sandstone core within the sample chamber. A precision syringe pump injects brine solution into rock core at a constant speed, while a peristaltic pump introduces dry air, accelerating water evaporation near the rock core. Also, a hot water tubes around the sample chamber increases the internal temperature, expediting the drying rate. Throughout this process, stress and strain inside rock core are continuously monitored, while a series of X-ray CT scans were collected using the X-ray CT scanners located in UCD Rosemount Research Centre. The test concludes upon achieving substantial deformation under constant vertical stress.

Fig. 4 presents the aftermath of the brine drying test, indicating a significant impact of salt weathering. The X-ray scans revealed dynamic process of the salt weathering process. Notably, the failure mechanism of salt weathering is dramatically influenced by the confining stress, drying rate, and brine concentration etc. The two samples in Fig. 5 shows that two rock cores subjected to large and small vertical confinement have distinct failure patterns. Particularly, the significant vertical stress preventing significant degradation of rock core, indicating the interplay between internal stress distribution and rock damage.

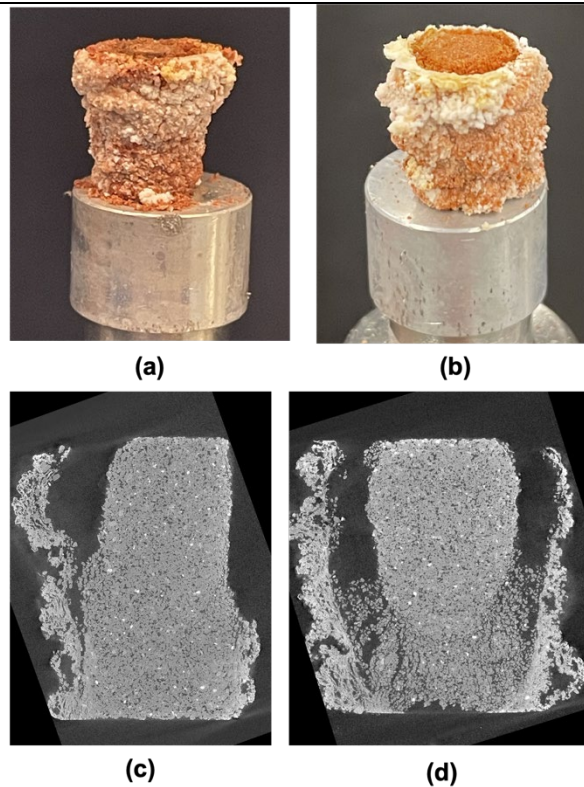


Fig. 4. Image of sandstone sample after salt weathering under large vertical stress (a,c) and small vertical stress (b,d). (c,d) Vertical grey-scale image of rock cores from X-ray scans

In summary, the project's results underscore its contributions to advancing scientific understanding in salt weathering studies. The innovative combination of a miniature loading apparatus, advanced imaging techniques, and meticulous testing procedures has unveiled unprecedented insights into the hydro-mechanical properties of sandstone under the influence of brine drying and salt precipitation. The findings hold significant implications for enhancing reservoir management and injectivity in carbon geological storage, positioning the project at the forefront of scientific and engineering progress in this field.

(ii) Project Implementation (milestones, deliverables, project management)

The implementation of this comprehensive study unfolded through three distinct working packages (WPs), each designed to address specific facets of the intricate dynamics of salt weathering in sandstone cores. The project management was organized in a structured manner, comprising milestones and deliverables, ensuring a systematic progression of tasks.

WP1 Loading apparatus commissioning (M1-M9)

Task 1.1 – Completed in M4: This task was pivotal in designing of a miniature loading apparatus compatible with X-ray microtomography (μ CT) to perform confined compression tests on sandstone cores. The sample chamber's dimensions and material were meticulously determined to ensure compatibility with the X-ray scanner, allowing for high-resolution imaging of the pore space. Additionally, the design incorporated a brine



saturation and gas injection path a brine saturation and gas injection path.

Task 1.2 – Completed in M8: Subsequent to the design phase, digital components including a precision stepping motor, LVDT, loadcell, and datalogger were procured. The design of the loading frame was then finalized, and a company with expertise in precise loading apparatus fabrication was selected based on competitive cost. The company fabricated various components of the apparatus based on the provided design graphs and then shipped the components to Dublin.

Task 1.3 – Completed in M9: The loading apparatus was assembled and fully calibrated during this phase. The loading algorithm, developed using LabVIEW, facilitated the control of the stepping motor, ensuring constant pressure during brine drying and constant loading velocity for subsequent failure tests.

WP2 Impact of precipitation on permeability (M3-M9)

Task 2.1 – Completed in M5: This critical phase focused on obtaining sandstone cores with different porosity, permeability, and mineralogy. Due to the challenges of drilling mini rock cores of 6 mm diameter, a company was enlisted to prepare mini rock cores with provided sandstones from Ireland and China. The GE Phoenix Nanotome M located at UCD Rosemount was utilized for scanning mini rock cores before and after drying tests. Cyclic drying-wetting tests were initiated to access cumulative impacts through multiple brine drying cycles.

Task 2.2 – completed in M7: Analysis of X-ray images of sandstone cores involved sophisticated image processing and analysis techniques, as depicted in Fig. 3. Challenges arose in differentiating precipitated salt from rock grains due to similar density. This task incorporated the use of open-source image processing software, Fiji, and customised Python codes.

Task 2.3 – completed in M9: Pore topology of rock cores was successfully imported into a Computational Fluid Dynamics (CFD) model. The analysis of permeability changes using CFD simulations, conducted with the open-source package, OpenFOAM, provided insights into the influence of salt precipitation on absolute and relative permeability.

WP3 Impact of precipitation on strength (M9-M12)

Task 3.1 & 3.2 – completed in M12: This final phase aimed to examine the salt weathering process under constant compression stress. The loading apparatus developed was crucial in investigating brine drying and salt precipitation within confined rock samples. Heated gas flow was employed to initiate and accelerate brine drying and salt precipitation. The loading apparatus was fixed on the rotation stage of the X-ray μ CT scanner to avoid moving out and in the sample from the scanner during the loading process. During the loading process, a few X-ray scans were collected to capture the relationship between fracture patterns and salt precipitation was scrutinized to understand stress degradation due to crystallization pressure.

(iii) Outputs to date

- The project successfully designed and fabricated a loading apparatus compatible with X-ray microtomography (μ CT). This apparatus is unique in its ability to conduct brine



drying tests under confinement while allowing for high-resolution X-ray scanning. This innovation of this apparatus represents a crucial contribution to experimental methodologies in studying salt weathering processes.

- The research conducted a comprehensive investigation into the impact of salt precipitation on permeability during radial flow injection. The findings were presented at the InterPore2023 conference, Edinburgh UK, through an oral presentation delivered by a PhD student. This presentation effectively communicated key research outcomes, fostering scientific discussions in the field and received positive feedback from the scientific community.
- A significant outcome includes the publication of a journal paper on Water Resource Research, a reputable journal with an impact factor of 6.159. This paper focuses on the impact of salt precipitation on rock permeability at the microscale. The acceptance of this paper underscores the scientific merit and significance of the research.
- The X-ray CT tests have revealed a dramatic impact of confining stress on the salt weathering process. These tests have elucidated the negative impact of confining stress on the internal damage process. This results are being summarized for publication in the Journal of Geophysical Research: Solid Earth, a journal with an impact factor of 4.39. The anticipated submission reflects the rigor and depth of the research findings.

(iv) Impact

- Relevance/application to GSI programmes, where applicable

The study contributes to the Geological Survey Ireland (GSI) programs, particularly in the context of addressing challenges outlined in the GSI Research Roadmap, i.e., Sustainability and Management of Earth Resources. The relevance of the project lies in its alignment with carbon capture and storage (CCS), an important technique to decarbonise the heat and transport sectors in Ireland.

The project has made strides in advancing experimental tools to investigate brine drying and salt precipitation in porous media, a hydro-chemo-mechanically coupled process. This interdisciplinary approach spans fields such as rock physics, petroleum engineering, and geochemistry. The study's impact is evident in bridging knowledge gaps between micro-scale understanding and a macro-scale implication. Notably, the reduction in well injectivity due to salt precipitation near injection wells has practical implications for CCS applications.

The novel experimental and numerical approaches developed have enhanced the physical understanding of the dynamics of brine drying and salt precipitation, impacting rock properties. The resulted publication in a high-impact scientific journals attests to the significance of the findings. The study not only contributes to a deeper understanding of hydro-chemo-mechanical processes at the microscale but also provides a foundation for predicting dynamic processes at involving gas invasion and salt precipitation, representing a significant advancement in pore-and particle-scale analyses.

Furthermore, the development of experimental tools, including microfluidics and in-situ loading device for X-ray CT, hold promise for investigating multi-physics processes in various geomaterials. These tools have the potential to explore phenomena such as multiphase flow, fines migration and clogging, and microbially induced carbonate precipitation. The broad applicability of these tools extends to groundwater resources, geothermal energy, soil



physics, contaminant transport, and beyond. The combined experimental and numerical approaches developed in this project offer valuable insights applicable to a wide range of research and industrial endeavours, aligning with the overarching goals of the GSI programs.

- Other impacts

The project's outcomes are poised to exert a considerable impact on both short and medium-to-long-term perspectives, particularly in the realm of carbon capture and storage (CCS). In the short term, the findings contribute to a heightened understanding of the dynamics of brine drying and salt precipitation, shedding light on the intricacies of this hydro-chemo-mechanically coupled process.

In the medium-to-long term, the project's impact extends to predicting the enduring efficacy of CO₂ injection wells. The knowledge gleaned from this study will play a pivotal role in refining and fine-tuning long-term performance predictions, offering valuable guidance for sustainable and efficient CCS operations. The ability to mitigate the influence of salt precipitation through optimization strategies or the application of surface wettability alteration chemicals represents a tangible and lasting contribution to the CCS domain.

Beyond the immediate scope of CCS, the study's outcomes have broader implications for engineering applications, encompassing areas such as heritage buildings and critical infrastructures situated in coastal zones. In the medium to long term, the insights gained from this project could lead to the development of innovative strategies to counteract salt weathering damage in critical infrastructures. The application of engineered materials and the design of remediation strategies anchored in a nuanced understanding of salt precipitation dynamics may become integral components of long-term infrastructure planning.



Publications:

Zhang, H., Sun, Z., Zhang, N., & Zhao, B. (2024). Brine drying and salt precipitation in porous media: A microfluidics study. *Water Resources Research*, 60(1), e2023WR035670. <https://agupubs.onlinelibrary.wiley.com/doi/full/10.1029/2023WR035670>

Zhang, H., Sun, Z., Zhang, N., Zhao, B. (2023). Brine Drying and Salt Precipitation in Porous Media: Microfluidics Quantification of Pore Heterogeneity and Wettability Impact. *15th Annual International Conference on Porous Media*, Edinburgh, Scotland, 22 – 25 May.

Paper in preparation

Zhang, H., Zhao, B.. Impact of confining stress on salt weathering in sandstone. *Journal of Geophysical Research: Solid Earth*