

## Fast time resolved micro-CT imaging: visualizing dynamic pore scale processes at high resolution

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### Introduction

Understanding fluid migration and the transport of solutes inside porous geomaterials is essential for numerous applications ranging from CO<sub>2</sub> storage in deep underground reservoirs to the weathering of natural building materials in our everyday environment. When fluids migrate through the pore network they can react with minerals of the geomaterial, altering the mineral matrix and thus changing the pore network. The distribution and migration of fluids, is inherently controlled by the pore size distribution and its connectivity, while the chemical reactions are in turn controlled by the mineral and fluid distribution inside the geomaterial.

In order to fully comprehend the pore scale processes related to reactive fluid, one needs to characterize the fluid distribution in the pore space in 3D and monitor how the distribution and mineral matrix alters through time. In this research fast time resolved and high resolution lab-based micro-CT imaging is applied to analyse solute transport inside a heterogeneous limestone sample. Fast time resolved 3D imaging with a time resolution of 12 seconds was applied to visualize advective and diffusive transport of solutes at the pore scale level, which permits to map preferential flow paths and more stagnant regions inside the heterogeneous pore network. The location and distribution of preferential flow paths and more stagnant areas will influence the reaction kinetics of the interactions between the mineral matrix and pore fluid. This information can be used to predict the dissolution behaviour of the heterogeneous limestone when an acidic fluid is migrating through the sample.

This example illustrates the potential of fast lab-based micro-CT imaging to monitor dynamic processes through time. In the past decade micro-CT imaging became almost a routine 3D characterization technique in the field of geosciences, providing an ever higher spatial resolution (Cnudde and Boone 2013). With this example we want to illustrate the potential of the technique for the future by pushing the limits of the temporal resolution to almost real time 3D imaging to monitor dynamic processes.

### Materials and Methods

Diffusive and advective transport of a tracer salt is investigated in the Savonnières limestone, which is a grain-supported oolitic limestone. The limestone has a dual porosity with well-connected pores between the grains (intergranular porosity) and secondary porosity inside the grains (intragranular porosity), which is connected to the

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rest of the pore network through microporosity. The solute transport inside the pore space was visualized by pumping a highly X-ray attenuating brine (containing 10 wt.% CsCl) into a water-saturated sample at a flow rate of 0.6 ml/min. The sample had a diameter of 6 mm and was placed in a custom built PMMA flow cell.

Fast lab-based micro-CT imaging was performed on UGCT's dedicated 4D micro-CT scanner, designed and built in-house in collaboration with X-Ray Engineering bvba (XRE, Ghent, Belgium) (Dierick et al. 2014). This scanner is optimized to image samples under controlled environmental conditions or during dynamic experiments. The system is equipped with a standard directional microfocus X-ray tube and a CMOS flat-panel detector with CsI scintillator, which allow for fast CT scanning.

Prior to the dynamic flow experiment, the limestone sample was fully saturated with water and a high-quality CT scan was performed (voxel size 7.4  $\mu\text{m}$ ) to obtain a better characterization of the pore network in the sample. Afterwards, while an X-ray attenuating brine was pumped into to the sample, a fast micro-CT scan was acquired every 12 seconds for a total period of 5 minutes. The fast scans were recorded and processed with the proprietary 4D tools of the ACQUILA software (XRE, Ghent, Belgium) which resulted in a total of 72 reconstructions at a voxel size of 14.8  $\mu\text{m}$ .

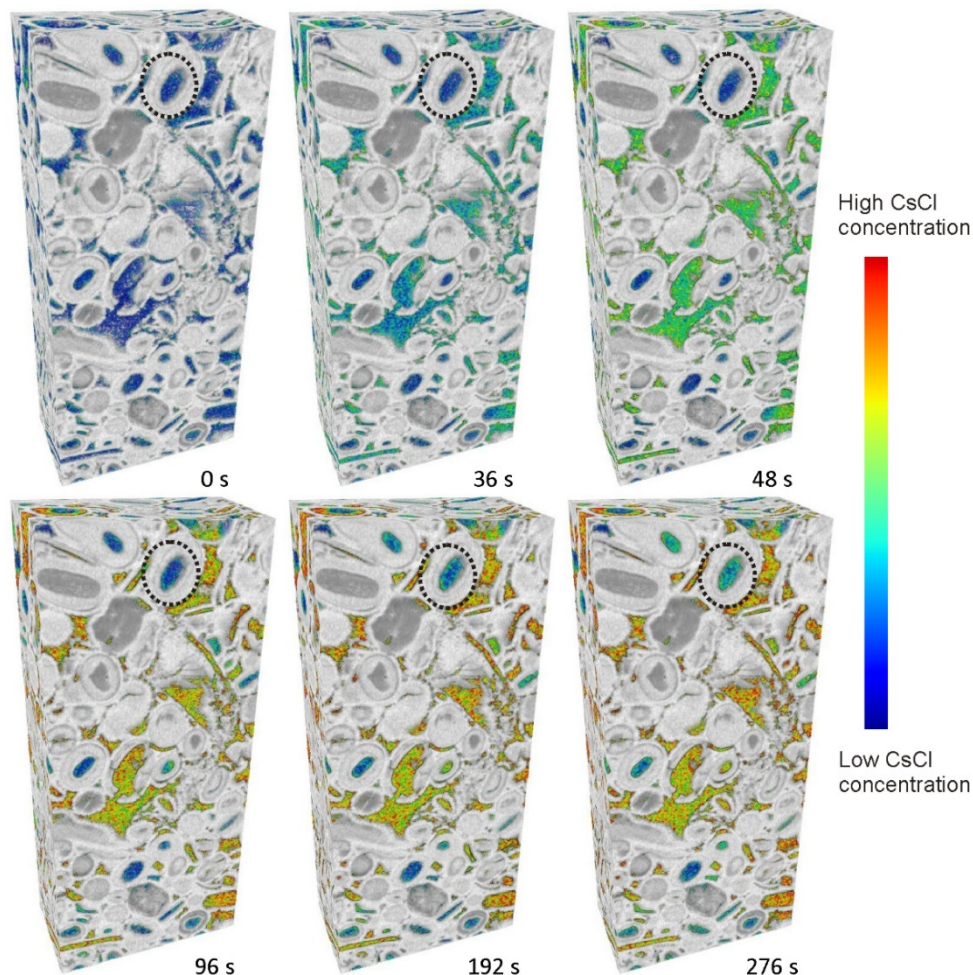


Figure 1. . The renderings illustrate the evolution of the CsCl concentration in the macropores. The dotted circle indicates an intragranular pore in which the flow is stagnant. Time series measurements were performed with the EMCT-scanner at an acquisition time of 12 seconds.

## Results and Discussion

The image quality of the fast micro-CT scans is much lower compared to more standard high resolution micro-CT scans and this is mainly related to the limited X-ray flux of lab-based X-ray sources combined with the fast acquisition time. To improve the image analysis, information from the high quality scan, acquired before brine injection, was combined with the analysis of the fast scans. In the high-quality scan (before CsCl injection) the pore and solid voxels were identified and segmented using a single threshold in the 3D analysis software package Avizo (VSG/FEI, France). The segmented binary image of the pore space was then used as a mask for the fast micro-CT scans. In every scan the change in attenuation in the pore space was analysed. This attenuation is directly linked to the concentration of CsCl, where higher attenuation values indicate a higher CsCl concentration.

In figure 1, renderings of the limestone and the pore space are shown at different time steps during brine injection. The pore space is colour coded according to the CsCl concentration. At 0 seconds no CsCl has entered the pore space, which is illustrated by the homogeneous blue colour. After 96 seconds, the CsCl is heterogeneous distributed and most of the intergranular pore space contains a high CsCl concentration, while the intragranular pores in the dissolved ooids (figure 1, dotted circle) still have a low concentration. In later time steps, there is a more gradual increase of the CsCl concentration in the intragranular pores and the CsCl concentration becomes more homogeneous.

The results illustrate that the distribution of the brine in the intergranular pore space is mainly controlled by advection. In these pores, preferential flow paths can be found. The distribution in the intragranular pores on the other hand is more stagnant, indicating that solute transport in these pores is mainly controlled by diffusion. This shows that important differences in fluid chemistry arise between the more stagnant regions (intragranular pores) and the preferential flow paths with a higher advective transport rate. When acidic solution are injected into the pore space of this rock, which can occur when injecting CO<sub>2</sub> in underground limestone rich reservoirs, this distribution will influence the dissolution behaviour in the carbonate rock. In this limestone sample dissolution will be more pronounced along the preferential flow paths, which will become increasingly wide. This behaviour is often referred to as wormholing and is illustrated in figure 2 where a similar limestone sample was subjected to 94 hours of acidic (pH 3) fluid injection at constant flow rate of 0.5 ml/min. The dissolved rock after 38 and 94 hours of exposure is illustrated in respectively green and red, which illustrates that the dissolution occurs along preferential flow paths.

## Wormhole formation in limestone

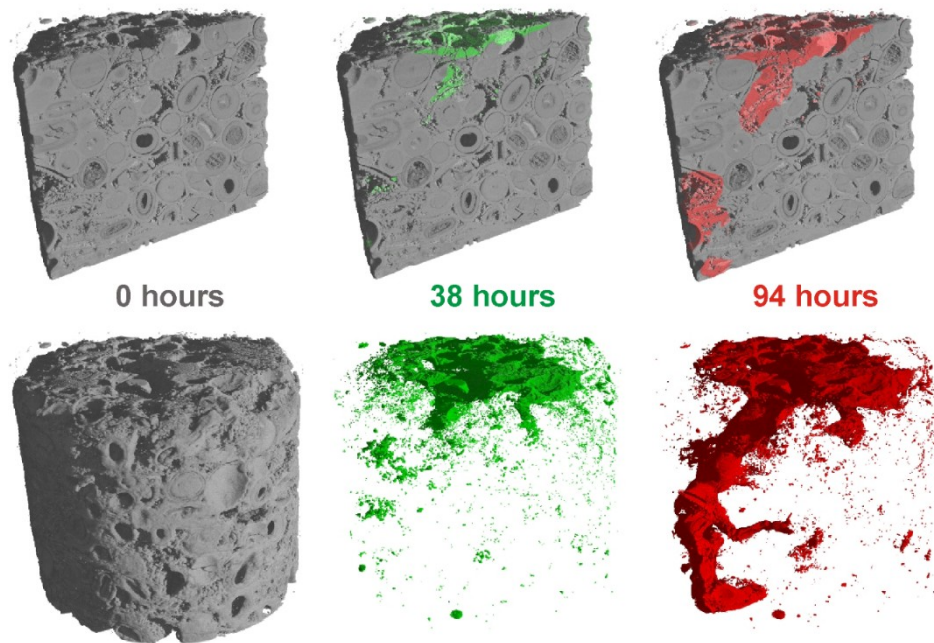


Figure 2. The renderings show the formation of a wormhole in the Savonnières limestone when exposed to an acidic (pH 3) fluid at a constant flow rate of 0.5 ml/min. The dissolved rock after 38 and 94 hours of exposure is illustrated in respectively green and red.

### Conclusion

The results illustrate that fast micro-CT scanning allows to investigate dynamic pore-scale processes and to predict reactive fluid flow. Despite some limitations like reduced image quality due to fast acquisition, pore scale solute transport of a tracer salt (CsCl) in a Savonnières limestone could clearly be visualized, allowing to study the location of stagnant and fast flow regions and the relative time scales of diffusion and advection in this limestone. The study transport processes in geological samples at the pore scale becomes possible, without having to impose quasi-static conditions. We expect that with further improvements in image quality, real-time imaging with laboratory-based micro-CT scanners will be implemented much more widely. This would provide valuable information for understanding the behaviour of fluids in a wide variety of porous materials (e.g. building stone deterioration, development of batteries and fuel cells, food engineering, textile engineering).

### References

- Cnudde, Veerle, and Matthieu Nicolaas Boone. 2013. "High-Resolution X-Ray Computed Tomography in Geosciences: A Review of the Current Technology and Applications." *Earth-Science Reviews* 123 (0): 1-17.
- Dierick, Manuel, Denis Van Loo, Bert Masschaele, Jan Van den Bulcke, Joris Van Acker, Veerle Cnudde, and Luc Van Hoorebeke. 2014. "Recent Micro-CT Scanner Developments at UGCT." *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* 324: 35-40.

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