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Fast laboratory-based micro-computed tomography for pore-scale research: Illustrative experiments and perspectives on the future

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ABSTRACT

Over the past decade, the wide-spread implementation of laboratory-based X-ray micro-computed tomography (micro-CT) scanners has revolutionized both the experimental and numerical research on pore-scale transport in geological materials. The availability of these scanners has opened up the possibility to image a rock's pore space in 3D almost routinely to many researchers. While challenges do persist in this field, we treat the next frontier in laboratory-based micro-CT scanning: in-situ, time-resolved imaging of dynamic processes. Extremely fast (even sub-second) micro-CT imaging has become possible at synchrotron facilities over the last few years, however, the restricted accessibility of synchrotrons limits the amount of experiments which can be performed. The much smaller X-ray flux in laboratory-based systems bounds the time resolution which can be attained at these facilities. Nevertheless, progress is being made to improve the quality of measurements performed on the sub-minute time scale. We illustrate this by presenting cutting-edge pore scale experiments visualizing two-phase flow and solute transport in real-time with a lab-based environmental micro-CT set-up. To outline the current state of this young field and its relevance to pore-scale transport research, we critically examine its current bottlenecks and their possible solutions, both on the hardware and the software level. Further developments in laboratory-based, time-resolved imaging could prove greatly beneficial to our understanding of transport behavior in geological materials and to the improvement of pore-scale modeling by providing valuable validation.

1. General introduction

Understanding how fluids migrate through porous rocks and how this affects the minerals inside that rock is essential in numerous geological applications, going from the formation and weathering of geological materials (e.g. building stones) to the production and storage of fluids in geological reservoirs. Although fluid migration influences the behavior of these geological materials on the macroscopic scale (meter to kilometer scale for building stones and geological reservoirs, respectively), the essential flow and alteration processes occur on the pore scale (nanometer to micrometer scale), between the minerals of the porous geological material. Comprehending the underlying pore scale processes is crucial to make accurate predictions and decisions regarding important challenges like CO_2-sequestration, environmental remediation of polluted aquifers, enhanced oil recovery and cultural heritage preservation. Of particular interest in this regard are multi-phase and multi-component (reactive) fluid flow, and the coupling of mechanical deformation or failure of the material of interest with such flow phenomena.

Understanding the pore scale behavior which controls these processes, however, is a difficult problem which requires three-dimensional pore space characterization. Pore spaces in geological materials often have a complex microstructure, which can span the centimeter to the nanometer scale and can be very heterogeneous in nature. Therefore, their characterization requires more than experiments which measure global information like porosity and pore size distribution. Three-dimensional imaging techniques fill this requirement by providing local geometrical and topological information. The most mature experimental technique in this category is X-ray micro-computed tomography (micro-CT) [1,2]. Before, researchers either had to extrapolate two-dimensional measurements to obtain pseudo-three-dimensional volume information or perform laborious serial sectioning experiments to obtain such local information.

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Micro-CT however allows the non-destructive visualization of internal pore space structures in three dimensions. The recent increase in the availability of laboratory-scale micro-CT instrumentation has enabled many researchers to characterize the pore space of geomaterials in 3D on an almost routine basis, although a number of limitations in both the acquisition and the analysis of micro-CT data persist. For example, the attainable spatial resolution is often on the order of 1 to a few microns and depends on the size of the studied sample, while the acquisition time is typically on the order of 30 min to a few hours.

While 3D pore space characterization is an essential part of any pore scale transport or degradation study, it does not provide insight into the dynamics of the processes under investigation. To avoid image blurring (and other motion artifacts), the imaged sample should remain unchanged during the micro-CT acquisition. Therefore, there is a need for fast, time-resolved 3D (i.e. 4D) imaging of a material's microstructure while such a process is taking place, as the only alternative is imposing quasi-static conditions by halting the process during every imaging experiment [3]. Extremely fast micro-CT imaging has become available at synchrotrons over the last few years, attaining even a sub-second time resolution [2–5]. Although a comprehensive review of dynamic experiments performed at synchrotrons is out of the scope of this article, we mention some notable examples of pore scale imaging (in a geological context) at the sub-minute time scale of two-phase flow [6–9] and reactive flow [10]. Other examples of fast imaging of porous media at synchrotron institutions can be found in [11–13]. While synchrotron experiments are proving very valuable, the restricted accessibility of synchrotrons limits the amount of experiments which can be performed. Therefore, the development of laboratory-based fast (sub-minute) micro-CT scanning can prove important, as it would drastically increase the availability of 4D imaging with sub-minute time resolutions. Fast lab-based micro-CT would also allow researchers to prepare 4D experiments in the lab before performing them with higher temporal or spatial resolutions and with better image quality at a synchrotron. This would be of high value to help optimize the use of synchrotron beam time.

Despite the desirability of fast lab-based micro-CT, the much smaller X-ray flux in lab-based systems bounds the time resolution which can be attained. Just like an underexposed photograph appears noisy due to photon counting statistics, the restriction on the X-ray flux in laboratory sources limits the image quality for short acquisition times. Other complications which may occur are for example insufficient angular sampling, too long detector read-out times and limited rotation speed. Despite the fact that rather little attention has been given to this topic in the literature (with the exception of work performed by researchers at the Australian National University [14–16]), advances in micro-CT hardware and in reconstruction and analysis software are starting to render sub-minute pore-scale experiments possible at laboratory set-ups. In this work, we illustrate this by the visualization of drainage of a Bentheimer sandstone and convective/diffusive mass transport of a salt (CsCl) in a water-saturated Savonnières limestone. In both experiments, the acquisition time per full micro-CT scan was 12 s, with a voxel size of 14.8 μm. While, naturally, the data quality is not as good as what can be expected from similar experiments performed at synchrotrons, we show that the obtained quality is sufficient to visualize many interesting aspects of the investigated transport processes in individual pores, even without applying special reconstruction algorithms. To our knowledge, the experiments reported in this work are the fastest in-situ, lab-based pore-scale micro-CT measurements of this kind described in the literature until now. Fig. 1 illustrates the progress in attainable spatial and temporal resolution in micro-CT over the years, both at synchrotron and in typical laboratory-sources [3].

Given the importance of the development of fast 4D lab-based micro-CT to the pore scale modeling and experimentation community, we describe the current state of the art and the expected future development of this field. Note, however, that this work is not intended as a comprehensive review but rather aims to provide a concise outline of the current and future possibilities of this experimental technique. In Section 2, we discuss the optimization of various hardware and software components. In Section 3.1, we show the real-time imaging of drainage at the pore scale in a Bentheimer sandstone, and in Section 3.2 the imaging of advective-diffusive mass transport of a tracer salt in a water-saturated limestone is presented. These experiments simultaneously illustrate the use of fast 4D lab-based tomography in pore scale experiments and the current state of the art of this method.

2. Technological advances

In order to obtain a high-quality micro-CT scan, the accumulated X-ray dose in the detector during the acquisition of the projection images has to be sufficiently high, as the signal-to-noise ratio varies approximately as the square root of the amount of photons which hit the detector. In laboratory-based micro-CT systems, this sets a lower limit to the acquisition time, as the used X-ray flux emitted by the X-ray source is typically low (taking into account also the necessary minimum distance between the source and the detector, due to considerations regarding image magnification, sample size and cone beam artifacts). The limitations of these sources with respect to dynamic imaging are explained in Section 2.1.1, as well as technological developments which might mitigate these limitations in the future. Other limitations on the image acquisition may be posed by the X-ray detector (Section 2.1.2). During dynamic experiments on porous materials, the sample conditions are usually controlled in some way (e.g. applying mechanical loading, imposing (reactive) fluid flow, controlling temperature and humidity). Therefore, the sample usually has to be contained in a cell or a similar set-up. These cells can affect the image quality, and should therefore be designed carefully (Section 2.1.3).

After treating the hardware challenges, we discuss the possibilities of smart image reconstruction and analysis to compensate for low image quality (Section 2.2). Fast scans, acquired while dynamic processes are going on in the pore space, can and should be supplemented with as much prior information as possible. In many experiments, prior information about the pore geometry can be acquired with a long, high-quality scan while the sample is in static conditions. Other prior information might pertain to the dynamic process itself, for example, in immiscible two-phase flow experiments the assumptions of incompressible fluids and the presence of only three material phases can improve reconstructions with a limited number of projections [14]. By scanning faster, the quality will typically decrease, hence approaches using a priori information gain importance. Another principle which we expect will prove important in the future, is the incorporation of the time dimension in the image analysis. On the one hand, there is a need for software tools which allow researchers to track changes in their sample over time (e.g. deformation of a sample, velocities of fluid interfaces) and help them to make sense of the typically huge amount of data they acquire during a dynamic experiment. On the other hand, truly treating micro-CT time series data as 4D datasets may improve image analysis results, as it allows to incorporate more information. However, one thing is certain: 4D data analysis is computationally intensive, both when it comes to data storage as processing. Section 2.2.2 treats these challenges and opportunities.

2.1. Hardware evolutions

2.1.1. X-ray sources

X-ray sources in laboratory-based micro-CT set-ups are typically microfocus X-ray tubes, in which an electron beam is focused on a very small focal spot on the anode target. When the electrons hit the target after being accelerated by a high-voltage electric field, they create X-rays through their interaction with the anode

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