

# Quantification of sub-resolution porosity in carbonate rocks by applying high-salinity contrast brine using X-ray microtomography differential imaging



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## ABSTRACT

Characterisation of the pore space in carbonate reservoirs and aquifers is of utmost importance in a number of applications such as enhanced oil recovery, geological carbon storage and contaminant transport. We present a new experimental methodology that uses high-salinity contrast brine and differential imaging acquired by X-ray tomography to non-invasively obtain three-dimensional spatially resolved information on porosity and connectivity of two rock samples, Portland and Estailades limestones, including sub-resolution micro-porosity. We demonstrate that by injecting 30 wt% KI brine solution, a sufficiently high phase contrast can be achieved allowing accurate three-phase segmentation based on differential imaging. This results in spatially resolved maps of the solid grain phase, sub-resolution micro-pores within the grains, and macro-pores. The total porosity values from the three-phase segmentation for two carbonate rock samples are shown to be in good agreement with Helium porosity measurements. Furthermore, our flow-based method allows for an accurate estimate of pore connectivity and a distribution of porosity within the sub-resolution pores.

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## 1. Introduction

Carbonate rocks are important geological porous media which are associated with many applications including hydrocarbon recovery (Fredd and Fogler, 1998), carbon storage (Gaus et al., 2008) and contaminant transport (Gelhar et al., 1992). Estimates show that carbonate reservoirs hold more than half of the world's hydrocarbon reserves (Sayers, 2008). They are known to have a very complex pore space whose structure and connectivity, dependent on the processes studied, have a significant impact on single and multiphase flow, and transport and reactive properties (Bijeljic et al., 2013; Norouzi Apourvari and Arns, 2015; Cantrell and Hagerty, 1999; Moctezuma-Berthier et al., 2004; Gjetvaj et al., 2015; Scheibe et al., 2015). Therefore, the main goal of our study is to obtain a full characterisation of the pore space, which hitherto has not been fully resolved.

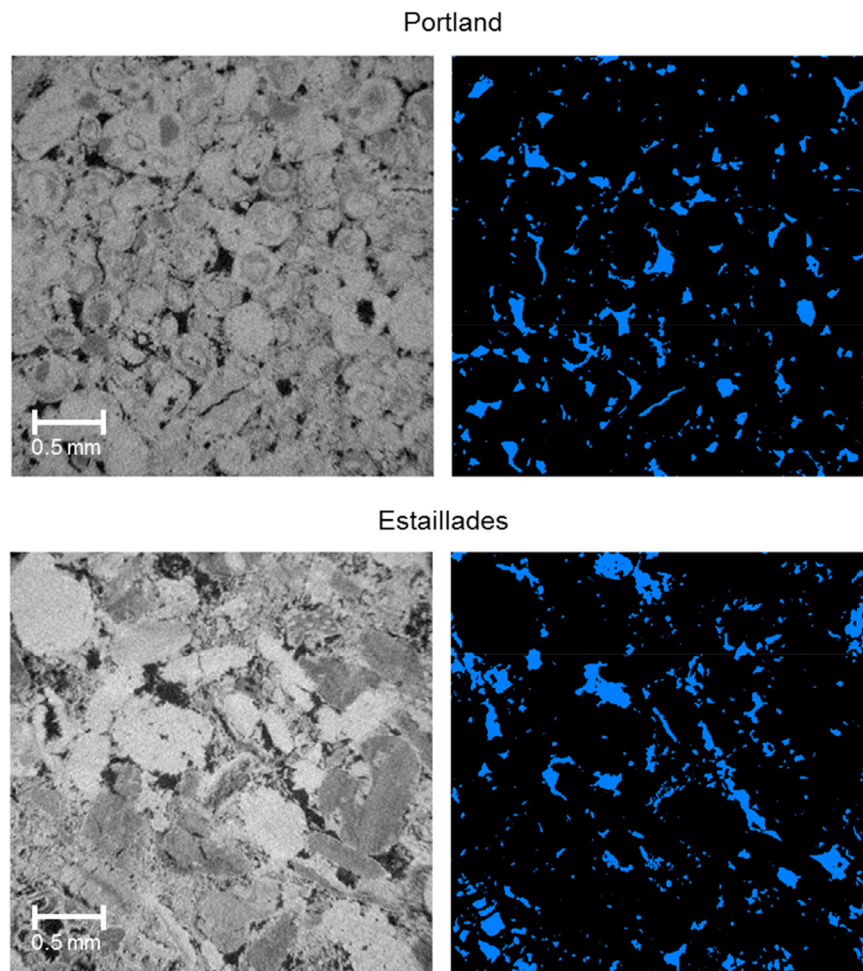
Anovitz and Cole (2015) have reviewed different methods for measuring porosity, including direct measurements such as Helium (He) porosimetry and Mercury Intrusion Capillary Pressure (MICP)

measurements, as well as imaging methods and techniques such as Optical Petrology, Scanning Electron Microscopy (SEM) and Nuclear Magnetic Resonance (NMR) imaging. However, although some direct measurement methods can yield accurate porosity values, it is challenging to resolve all the pore space, particularly sub-micron porosity. Two-dimensional methods may have higher resolution, but they do not show how the pore spaces are connected. Hence, having a technique which can provide spatially-resolved three-dimensional (3D) porosity values is important for both experimental and simulation studies.

X-ray microtomography (also called XMT or micro-CT) and image processing has been widely used in geologically-related studies (Ketcham and Carlson, 2001; Lin et al., 2015; Cnudde and Boone, 2013; Lin et al., 2016a; Dhawan et al., 2012; Lai et al., 2015; Lin et al., 2016b), and in recent years this technique has been applied in visualisation of multiphase flow and reactive transport in geological porous media (Andrew et al., 2014; Menke et al., 2015; Iglauer et al., 2012; Iassonov et al., 2009; Schlüter et al., 2014; Carroll et al., 2013). However, one of the main limitations of this method is the limited spatial resolution, which is typically of order of 1  $\mu\text{m}$  or larger (Iassonov et al., 2009). High resolution images (e.g. tens of nano meters voxel size by nano CT) may be obtained, but for a smaller samples (e.g. tens of microns sample size)

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**Fig. 1.** Example two-dimensional slices of a three-dimensional image (with dimensions of  $650^3$  voxels at voxel size of  $5\mu\text{m}$ ) for the Portland and Estailades samples. The grey-scale image is shown on the left and the segmented image on the right. The porosity of the segmented image is 0.078 for Portland and 0.130 for Estailades; this contrasts with Helium (He) porosity values of  $0.195 \pm 0.006$  and  $0.293 \pm 0.007$  respectively (conducted at Imperial College London, UK).

that may not capture the full range of pore sizes in a representative elementary volume. In theory, the grey-scale of the grains could be used to estimate the intra-granular porosity, but this assumes a uniform mineralogy and complete connection of all the sub-resolution porosity. To illustrate this, we provide example XMT images for our Portland and Estailades carbonate cores in Fig. 1. The XMT measured porosity, based on the larger pores only, is much lower than the porosity obtained by Helium porosimetry, as presented later. There is a range of grey scales for the grains, but as we will show, this alone does not quantify the extent of connected sub-resolution pores.

Sub-resolution porosity is one of the main parameters required for a full description of flow and reactive transport processes in porous media, such as the determination of permeability and dynamics of dissolution-precipitation mechanisms using XMT (Mangane et al., 2013; Qajar and Arns, 2016; Garing et al., 2015). Combining XMT images and local two-dimensional Scanning Electron Microscopy (SEM) images was shown to be able to resolve sub-resolution pores (Peters, 2009; Smith et al., 2013). However, the main limitations for fully utilising SEM images are that they require extensive time and effort to acquire. In most cases, only part of the sample is imaged, while at the same time pore-space connectivity of sub-resolution pore space is not confirmed from the experiment, as in flow based methods. In some studies, segmenting sub-resolution pores directly from dry scans has been attempted by assuming that grains with an intermedi-

ate grey-scale were regions with sub-resolution pores (Mangane et al., 2013; Bauer et al., 2012; Bultreys et al., 2015; Garing et al., 2014; Soullaine et al., 2016; Luquot et al., 2014). However, most of the porosity values measured from image analysis cannot be matched with the porosity values obtained by other methods. Freire-Gormaly et al. (2015) studied the sensitivity of different thresholding techniques to segment the pore space, including sub-resolution pores for carbonates, applied to Indiana Limestone and Pink Dolomite. They showed that a single imaging technique and thresholding method cannot alone determine porosity from a dry scan only. The main problem for segmenting sub-resolution pores from dry scan is, as we present below, that a grain with an intermediate grey-scale may have sub-resolution pores, or simply be composed of a different mineralogy, or contain pores that are not connected to the macro-pore space. Thresholding a dry scan alone cannot distinguish between these cases. Moreover, some of the main issues such as connectivity of the sub-resolution pores could not be addressed.

One of the potential solutions is to introduce a new material phase to provide better image contrast for sub-resolution pores. Boone et al. (2014) performed a differential imaging experiment in Massangis limestone comparing dry scans with scans obtained once the rock sample was saturated with 5% CsCl to account for the contribution of sub-resolution pores to the total porosity values. However, their results were not close to the porosity obtained by MICP measurements. Ghouse et al. (2007) used a mixture of

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