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# Morphological evaluation of heterogeneous oolitic limestone under pressure and fluid flow using X-ray microtomography



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## ABSTRACT

Pore-scale analysis of carbonate rock is of great relevance to the oil and gas industry owing to their vast application potentials. Although, efficient fluid flow at pore scale is often disrupted owing to the tight rock matrix and complex heterogeneity of limestone microstructures, factors such as porosity, permeability and effective stress greatly impact the rock microstructures; as such an understanding of the effect of these variables is vital for various natural and engineered processes. In this study, the Savonnières limestone as a carbonate mineral was evaluated at micro scales using X-ray micro-computed tomography at high resolutions (3.43 µm and 1.25 µm voxel size) under different effective stress (0 MPa, 20 MPa) to ascertain limestone microstructure and gas permeability and porosity effect. The waterflooding (5 wt% NaCl) test was conducted using microCT in-situ scanning and nanoindentation test was also performed to evaluate microscale geomechanical heterogeneity of the rock. The nanoindentation test results showed that the nano/micro scale geomechanical properties are quite heterogeneous where the indentation modulus for the weak consolidated area was as low as 1 GPa. We observed that the fluid flow easily broke some less-consolidated areas (low indentation modulus) area, coupled with increase in porosity; and consistent with fines/particles migration and re-sedimentation were identified, although the effective stress showed only a minor effect on the rock microstructure.

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# 1. Introduction

Carbonate rocks are heterogeneous rocks with vast application potentials in contaminant transport (El Yaouti et al. 2008), enhanced oil recovery (Dai et al. 2016, Haugen et al. 2012, Nwidee et al. 2017), and carbon dioxide storage (Al-Yaseri et al. 2017, Blunt et al. 2013, Iglauer et al. 2015, Jiang et al. 2016, Lebedev et al. 2017b, Zhang et al. 2016d). This rock type is gaining significant research interest as they in principal, presents colossal  $CO_2$  sinks and hold huge amount of global oil reserves (Gozalpour et al. 2005; Sayers 2008). An understanding of fluid flow in such porous media is vital for various natural and engineered processes (Wildenschild and Sheppard 2013; Gonçalves et al. 2017) as production from limestone reservoirs are usually inefficient owing to the heterogeneous structures and poor water imbibition which inhibits efficient fluid distribution and displacement at pore scale (Riaz et al. 2007; Dawe et al. 2011; Nwidee et al. 2017).

With the continuous growth in imaging technology, effective quantification of rock microstructure, composition and petrophysical properties using new petrological approach in two and three dimension microstructural analysis is increasing (Jerram and Higgins 2007; H. Li et al. 2017, Z. Li et al. 2017; Liu and Ostadhassan 2017a). Technological tools such as scanning electron microscopes (SEM) (Huggins et al. 1980; Roels et al. 2003; Freire-Gormaly et al. 2015; Baud et al. 2016; Xu et al. 2016; Lyu et al. 2018) and transmission electron microscopes (TEM) (Eshed et al. 2011; Son et al. 2015; Zargartalebi et al. 2015) have been used to evaluate rock microstructure and heterogeneity, however, the sample preparation method for these tests usually need polishing, vacuum conditions or involve drying or freezing which alters the microstructure of water-saturated samples (Kozaki et al. 2001). More so, effective stress is not considered during SEM and TEM evaluation and only 2D images are obtainable for pore morphological evaluation which is insufficient for permeability estimation as 3D supercedes 2D (Stauffer 1979). As such, new technological advances are been sought to better understand the fluid flow distribution in limestone microstructures.

Interestingly, the advent of microscale X-ray computed tomography (microCT) pore scale imaging has allowed for improved understanding

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of fluid displacement and distributions in rocks and pore scale characterization (Iglauer et al. 2011; Andrew et al. 2013; Blunt et al. 2013; Andrew et al. 2014; Rahman et al. 2017; Roshan et al. 2018). MicroCT technique has shown efficiency for use in effective stress evaluation (e.g. Iglauer et al. 2013, Zhang et al. 2016c, 2016e, 2017a) with scanned in-situ in 3D at high resolution. Another effective technological advancement is nanoindentation tools, used to ascertain microscale mechanical properties on the surface of materials (Oliver and Pharr 2004; Fischer-Cripps 2004; Fischer-Cripps 2006) and heterogeneous materials via grid indentation analysis (Constantinides et al. 2006). Technically, the elastic modulus on each point is obtained by the nano indenter loading - unloading curve (Fischer-Cripps 2004). Such nanoindentation has already been successfully used for evaluating the mechanical properties of shale (Bobko et al. 2011; Liu and Ostadhassan 2017b), sandstone (Zhu et al. 2009), coal (Yu et al. 2017, Zhang et al., 2017b, 2018a, 2018b), and also limestone (Lebedev et al. 2014; Vialle and Lebedev 2015). Lebedev et al. (2014) showed that a wide range of indentation modulus existed for Savonnières limestone with the weak areas and associated high pore pressure upon injection of outsource fluids into the limestone reservoir for oil and gas recovery. However, there is currently no evidence in literature as to how the injected fluid flow influences the microstructure of the rock, especially the weak area with low indentation modulus. Moreover, the effective stress is changing during fluid injection into the underground, which is the main factor (effective stress or the hydraulic force during the fluid flow) for such heterogeneous oolitic porous media microstructure during the water flooding as directly affect the porosity/permeability are poorly understood. Thus in this study, we conducted the nanoindentation test, and microCT in-situ analysis of the limestone under different effective stress and brine injection to evaluate the effect of the petrophysical properties of the rock, stress change and fluid flow on the limestone microstructure.

## 2. Experimental procedure

## 2.1. Sample selection

The core plugs selected for this experiment are Savonnières limestone, a carbonated rock acquired from eastern France classified as deposits of Jurassic age (Fronteau et al. 2010). The core sample composition was measured using XRD with a Bruker-AXS D9 Advance Diffract Meter. The samples are mainly composed of calcite (97 wt%)



**Fig. 2.** (A) schematic of the indenter penetrating into the sample during loading process under brine saturated condition; (B) an example of loading-unloading curve (the penetration depth - ht -  $\mu$ m with loading force P - mN) for the calibration fused quartz sample (where the Poisson's ratio 0.17, Young's modulus 72.5 GPa, and the measured indentation modulus is 74.5 GPa).

and biotite (3 wt%), see Fig.1 for the XRD data. Varying sample sizes and shapes were carefully prepared from the same Savonnière limestone block for the following multi-scale experiments: gas permeability, nanoindentation, and microCT in-situ scanning.



Fig. 1. The XRD data for the tested Savonnières limestone sample.

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