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Technical note

Effective porosity measurements of poorly consolidated materials using non-destructive methods

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ABSTRACT

The porosity characterization of poorly consolidated sediments or soils is a key factor in understanding fluid flow and geochemical processes in surface and subsurface environments. However, porosity quantification is a challenging task due to the low cohesion of the extracted sample. In this study, we proposed and assessed a method combining X-ray computed micro-tomography (μCT) and He-gas pycnometry for the measurement of connected porosity in unconsolidated subsurface sands. The principle is based on the calculation of the total sample volume from reconstructed μCT volume and the measurement of the solid volume from He-gas pycnometry. Disaggregation of the sample may occur during handling and is taken into account by correcting the μCT volume using image processing. The measurements obtained for reference consolidated sandstone were compared with those obtained from water absorption under vacuum. The use of such non-destructive techniques offers the advantage of preserving loose sample structure. Moreover, it is a rapid and simple method that requires no sample preparation and could be easily implemented as routine analysis.

1. Introduction

Anthropic activities, such as industrial and mining activities, impact the composition of surface and subsurface water resources. Accurate petrophysical characterizations of soils and shallow subsurface reservoir rocks, such as effective porosity and pore connectivity, are required to understand the mobility of contaminants in natural environments and the geochemical processes involved in water/rock interactions. The solid framework of these materials can be very fragile, depending on its consolidation state. The framework usually consists of aggregates of finely divided minerals (*e.g.,* clays), detrital grains, and sometimes includes organic matter. The porosity characterization of a sample should preserve its fragile structure and should include consideration of microporosity. Many efficient methods are commonly used to quantify the porosity of consolidated materials; however, these methods are often invasive, and sample damage is often suspected when such methods are applied on poorly consolidated samples. Porosity, permeability, and pore connectivity data are typically obtained from laboratory measurements by using either image analysis techniques (*e.g.,* Scanning Electron Microscopy (SEM), electron microprobe, ^{14}C-PMMA method, X-ray computed micro-tomography (μCT) (Bruand et al., 1996; Lindquist et al., 2000; Sammaljärvi et al., 2012; Cnudde and Boone, 2013; Sardini et al., 2015), chemical or physical measurements using fluid intrusion (*e.g.,* mercury intrusion, water pycnometry, diffusion of tracers) (Tullborg and Larson, 2006; Descostes et al., 2008 and references therein), or coupling several of these approaches (*e.g.,* mercury intrusion and μCT or μCT and ^{14}C-PMMA) (Cnudde et al., 2009; Voutilainen et al., 2012; Fusi and Martinez-Martinez, 2013). However, these data can be difficult to obtain from poorly consolidated samples because the simple and traditional measurement of porosity involves intrusion of liquids into the pores, which is a process that can substantially alter the structure of a poorly consolidated sample. Structural damage is enhanced when the sample exhibits petrologic variations at centimetric scale (*e.g.,* fine sedimentary facies). Moreover, the use of traditional imaging methods (*i.e.*, μCT and SEM) alone cannot resolve the microporosity of clays and/or organic matter (lignite) aggregates (Pret et al., 2010). In addition to the fact that sample preparation for SEM or microprobe evaluation requires the intrusion of resin inside the sample, it also requires a careful and cumbersome preparation, which is often suspected to alter the pore structure of the rock.

In this context, we propose a non-destructive method that allows bulk connected porosity measurements of poorly consolidated samples containing finely divided components.

In this study, the connected porosity of very poorly consolidated sands from the Chu-Saryssu basin (South Central Kazakhstan) was obtained by combining two non-destructive techniques. The methodology is based on gas (helium) pycnometry measurements and image analysis of X-ray computed microtomographic data and does not require any impregnation of the sample. The procedure was initially validated on a coherent reference material (Fontainebleau sandstone) that had been

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previously characterized by water gravimetry. This method provides accurate porosity measurements on small samples with minor alteration of their initial structure regardless of the size distribution of the pores. A rapid and convenient procedure without any specific sample preparation is proposed below for porosity measurements of materials with low levels of cohesivity.

2. Materials

Poorly consolidated medium sand of Eocene sandy deposits from the Chu-Saryssu basin (Kazakhstan) was studied previously (Robin et al., 2015a). Estimating the porosity of these deposits is of prime interest in a context of mining environment remediation because acidic solutions had been injected into this sand formation to extract uranium (ISR process) (Robin et al., 2015b). The sandy deposits are mainly composed of quartz, feldspars and micas coated by clay minerals (smectite and kaolinite). The intergranular porosity sometimes appears filled with a fine polycrystalline matrix composed of clay minerals and fragments of detrital minerals (Robin et al., 2015a). The detrital minerals are also accompanied to a certain degree by disseminated organic remains (brown coal). Therefore, the sands exhibit a wide range of pore sizes, from micropores (<2 nm) in clay matrix, to macropores (>50 nm) (Robin et al., 2015a). Three samples with volumes of less than 1 cm^3 were used in this study for a reproducibility test and for testing the effect of the drying temperature. Two samples were dried for two days at 60 °C (KzM1_1 and 3) and one sample at 105 °C (KzM1_2). Because of the low cohesion of the sand, the samples were extracted from a freshly drilled core (7-cm diameter) using a knife. Care was taken to avoid damage to the samples during this last step because the shearing of the rock during this extraction should hypothetically create damage on the sliced surfaces. However, according to the μCT observations, no major damage was observed.

Two blocks of Fontainebleau sandstone (France) were used for validation of the proposed procedure by measuring porosity by using usual technique. This Oligocene age (Stampian) sandstone is almost exclusively composed of quartz (>99%) (Bourbie and Zinszner, 1985). The porosity of the Fontainebleau sandstone has been extensively studied using different techniques (*e.g.*, mercury intrusion, water absorption under vacuum, μCT segmentation) and the results of the studies have shown a wide and similar range of porosity values, from 2 to 30%, depending on the sample consolidation, and without variation of the grain size (Bourbie and Zinszner, 1985; Fredrich et al., 1993; Lindquist et al., 2000, among others). For samples with porosity values higher than 10%, it has been shown that both the connected and total porosity are similar (Lindquist et al., 2000). The two samples used in this study were collected from highly consolidated sandstones that did not display any fine-grained matrices.

3. Methods

3.1. μCT analysis and image processing

X-ray computed microtomography was performed on unconsolidated sand samples with a VISCOM X8050 microtomograph available at the University of Poitiers. Radiographs were acquired on a 360-degree angular range using a 0.4-degree angular step (thereby leading to the acquisition of 900 radiographs), for an acquisition time of approximately 1 h. Twenty white images were also acquired to use for the flat-field correction (each radiograph was divided by the average white image). A set of radiographs of a test pattern (mire) was also collected for accurate determination of the system geometry used during image acquisition. Before sample reconstruction, an attenuation of ring artifacts was performed using the Boin and Haibel (2006) algorithm. Sample reconstruction was performed using DigiCT v.2.4.1. software (DIGISENS) with a filtered back projection image-reconstruction algorithm. The final resolution of the isotropic voxels ranged from 12.18 to 21.62 μm.

The image analysis of the μCT data was performed in 3D using Aphelion™ software, which operates for two- or three-dimensional images. The different image analysis operators used for filtering and analyzing the volume images are described in Appendix A (the entire imaging sequence used for determining rock porosity is described Section 4); these operators are described in more detail by Pirard and Sardini (2011); Russ (2011) and Soille (2013).

3.2. He-gas pycnometry

The volumes occupied by the grains of the samples were measured using a Micromeritics AccuPyc 1330 He pycnometer. Several measurements were performed for each sample (repeatability tests) until the standard deviation between three consecutive measurements was less than 1 mm^3. The maximum sample size was determined by the size of the He-pycnometer sample holder, which was approximately 10.7 cm^3 (1.85 cm diameter × 3.98 cm height for standard sample holder).

3.3. Water absorption under vacuum

Porosity measurements were performed on the reference consolidated sandstone using a method adapted from Flint and Flint (2002). A sample's pore volume is determined from the difference in weight under dry and water-saturated conditions, which is obtained after water saturation of the sample under vacuum. The total volume of the sample can be determined by submerging the saturated sample in water and then weighing it. The weight corresponds to the volume of displaced water. This measurement was not performed on the sand samples from Kazakhstan because the samples would have been totally disaggregated when placed into water.

3.4. Water pycnometry

Coating samples with paraffin is typically used for analysis of unconsolidated samples. The final mass is then corrected for the mass of sample lost during the handling (Blake and Hartge, 1986; Rossi et al., 2008). Water pycnometry can be used to measure the total volume of the sample (solid and pore volume) after coating the sample with paraffin. The total sample volume can be determined from the volume of water displaced by the immersed sample that has been embedded with paraffin to prevent the intrusion of water into the pore space. The displaced volume is obtained by weight measurements and is corrected for the volume of paraffin used for the embedding. This sample volume is then used to obtain a second estimation of the porosity and can be compared to the non-destructive method proposed in this study.

4. General principle of the method and calculations

The samples from Kazakhstan exhibit large (100–300 μm), unconnected quartz and feldspar grains in 2D, forming large intergranular porosity (Fig. 1). Accurate segmentation of the pore space at the scale of the SEM observations (in black, Fig. 1) is not possible due to the presence of highly microporous and fine polycrystalline matrices mainly composed of clay minerals.

A porosity measurement method is proposed based on the evaluation of the total volume of the sample obtained from μCT volume analysis ($V_{\mu CT}$), the measurement of the solid volume from He-gas pycnometry ($V_{Hepyc.}$), and the evaluation of the volume of grains that separated from the sample during handling (V_g). Because the samples can be easily disaggregated, they were placed in small polyethylene (PE) vials (12-mm diameter) after extraction from the main bore core. Plastic vials were chosen because the X-ray absorption factor of the PE is low compared to silicate vials, which enables an easy distinction between vial and rock. However, in carefully placing each of the samples in a vial, a small amount of grains separated from the main sample volume and fell to the bottom of the vial (Fig. 2a). The quantification of the