



Further insight into the microstructure of compacted bentonite–sand mixture

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ABSTRACT

Compacted bricks of bentonite/sand mixture are planned to be used as sealing plugs in deep radioactive waste disposal repositories because of their low permeability, high swelling capacity and favourable properties with respect to radionuclide retention. The isolating capacity of compacted bentonite/sand mixture is closely related to microstructure features that have been often investigated, in particular by using scanning electron microscopy (SEM or ESEM) and mercury intrusion porosimetry (MIP).

In this work, microfocus X-ray computed tomography (μ CT) observations were used in parallel with MIP measurements to further investigate at larger scale the microstructure of a laboratory compacted bentonite/sand disc (65/35% in mass). Qualitative observation of μ CT images showed that sand grains were inter-connected with some large pores between them that were clearly identified in the bimodal pore distribution obtained from MIP measurements. Due to gravitational and to frictional effects along the specimen periphery, a higher density was observed in the centre of the specimen with bentonite grains more closely compacted together. This porosity heterogeneity was qualitatively estimated by means of image analysis that also allowed the definition of the representative elementary volume. Image analysis also provided an estimation of the large porosity in good agreement with MIP measurements.

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1. Introduction

In deep radioactive waste disposal concepts, sealing plugs made of compacted bentonite/sand blocks are planned to be used to close the galleries and to limit water transfers. Compacted bentonite/sand plugs are used for their low permeability, high radionuclide retention capability and sealing/swelling abilities when infiltrated by water (e.g. Pusch, 1979; Yong et al., 1986). Once the disposal galleries are closed, plugs will be progressively infiltrated by the pore water of the host rock. They will swell and seal the so-called technical voids of the system, i.e. the voids remaining between blocks and at the interfaces with the rock. These technical voids are estimated at 14% of the total volume of the plugs by IRSN (Institut de Radioprotection et de Sûreté Nucléaire, the French expert Institution in the field of nuclear safety) in the in-situ SEALEX experiment that they carry out in their Tournemire Underground Research Laboratory.

Various investigations of the microstructure of compacted bentonites and sand bentonite mixture have been carried out by using mercury intrusion porosimetry (MIP) and scanning electron microscopy (SEM, ESEM, Komine and Ogata, 1999; Villar and Lloret, 2001; Cui et al., 2002; Montes-H, 2002; Lloret et al., 2003; Agus and Schanz, 2005; Delage et al., 2006). These techniques require a cautious preliminary

dehydration of the samples, most often by freeze-drying. They provide local observations on a small part of millimetric samples. These localized analytical techniques can be fruitfully complemented by use of microfocus X-ray computed tomography (μ CT), a high-resolution non-destructive 3D observation technique. The μ CT does not need any sample pre-treatment and gives further 3D information on the whole sample (including grain size distribution as well as pore size distribution and pore inter-connectivity).

Previous applications of μ CT have been devoted to the monitoring of hydro-chemo-mechanical processes (Comina et al., 2008), to the detection of desiccation cracks (Gerbrenege et al., 2006; Mukunoki et al., 2006), to the visualisation of diffusion/hydration phenomena, to the study of fluid movements (Rolland et al., 2003, 2005; Carminati et al., 2006; Koliji et al., 2006) and to the investigation of the microstructure of compacted bentonite based materials (Kozaki et al., 2001; Van Geet et al., 2005; Kowaragi et al., 2009). In this work, μ CT was coupled to MIP for further microscopic investigation of a compacted bentonite/sand sample.

2. Material and methods

2.1. Material

The studied material is a compacted mixture of Wyoming MX-80 bentonite (65% in dry mass, commercial name Gelclay WH2) and

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sand (35%). The MX80 bentonite contains 92% of montmorillonite with several other minerals including quartz, alumina, and hematite (Tang et al., 2008). The sand is quartz sand (commercial name TH1000).

The sand–bentonite mixture was provided in boxes containing a powder with an initial water content of 10.2% and a suction of 73.3 MPa measured with a chilled mirror dew point tensiometer (Decagon WP4). Samples of sand and bentonite were also provided separately. The grain size distribution of the bentonite powder obtained by dry sieving is presented in Fig. 1 together with that of the deflocculated bentonite obtained by sedimentation. The grain size distribution of the sand is also plotted in Fig. 1.

The grain size distribution curves show that the bentonite powder grains are larger than the sand grains with D_{50} values of 1.2 and 0.6 mm respectively. Both curves are representative of well sorted materials. The unit mass of the bentonite particles that constitute the bentonite grains was measured by using a pycnometer with water and was found equal to 2.77 Mg/m^3 in agreement with published data (Madsen, 1998; Karnland et al., 2006). The unit mass of the bentonite powder was determined by using a pycnometer with a non-aromatic hydrocarbon fluid (commercial name Kerdane). The bentonite grains appeared to be stable once immersed in Kerdane and a value of 2 Mg/m^3 was obtained. This value could suffer from some uncertainty due to possible Kerdane infiltration into the grains. A comparable unit mass value was however obtained from the cumulative pore size distribution curve presented later in Fig. 2 that allows determining the unit mass of the bentonite grains from the value of inter-grain porosity. The unit mass of the sand grains was found equal to 2.65 Mg/m^3 .

Compacted samples were prepared by uniaxial static compaction (strain rate of 0.1 mm/min) in a cylindrical mould in order to obtain a disc (diameter 50 mm, height 10 mm) at the targeted dry density (1.8 Mg/m^3 obtained at a maximum compaction stress of 25.5 MPa). The sample water content was 10%, resulting in a degree of saturation of 55% and a suction of 76.3 MPa. Note that this value is close to that of the powder prior to compaction (73.3 MPa). The slightly higher value could be due to some effects of evaporation during the process of compaction. In any case, these small changes in suction after compaction are consistent with the observations of Gens et al. (1995), Li (1995) and Tarantino and De Col (2008) on soils compacted on the dry side of Proctor optimum. It confirms that suction is governed by aggregates that are not much affected by the compaction. As a consequence, there is little dependency of the water retention properties on the sample density in compacted bentonite based materials.

2.2. Mercury intrusion porosimetry

The pore size distribution of the compacted samples was measured on freeze dried samples by using an Autopore IV 9500 mercury intrusion porosimeter (Micromeritics) working to a maximum pressure of 230 MPa. Instantaneous freezing was carried out by plunging small

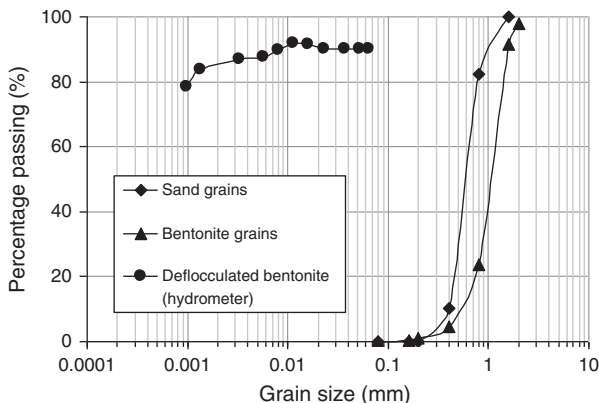


Fig. 1. Grain size distribution curves.

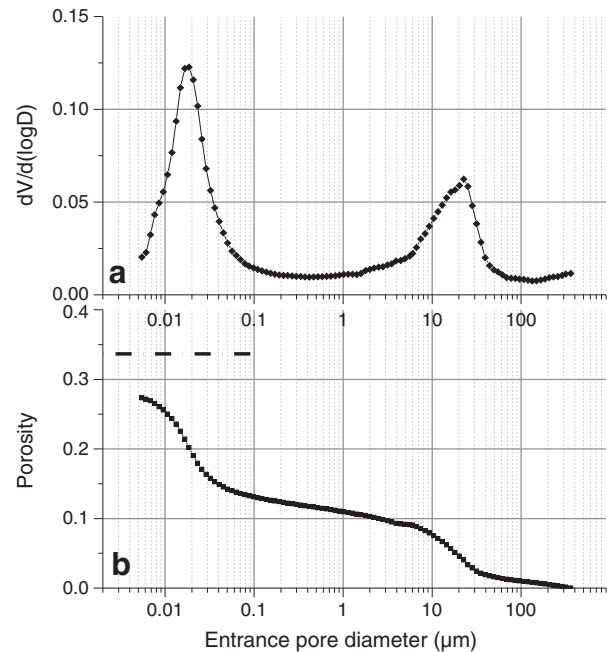


Fig. 2. (a) Pore size distribution curve and (b) cumulative porosity curve.

samples (volume 0.40 cm^3) into slush nitrogen (liquid nitrogen cooled down from -195°C to -210°C by vacuum application, Delage et al., 2006). In such conditions, there is no nitrogen boiling around the samples when plunging them into nitrogen, resulting in an optimized quick freezing and good microstructure preservation. The intruded porosity was determined from the total volume of mercury intruded into the sample and the pore size distribution was obtained, in a standard fashion, assuming parallel, cylindrical nonintersecting pores of different radii, using the Autopore IV 9500 V1.09 standard software package. The intruded porosity was systematically compared to the total porosity obtained by standard methods so as to detect the smaller porosity (entrance equivalent diameter smaller than 5.5 nm) not intruded by mercury at the highest applied pressure (200 MPa).

2.3. Microfocus X-ray tomography and image analysis

Microfocus X-ray computed tomography (μCT) was used to examine in three dimensions the internal microstructure of the compacted bentonite/sand mixtures. μCT is a non-destructive observation technique that has been proven to be useful in the investigation of various geological porous media including compacted bentonite (Kozaki et al., 2001), bentonite pellet/powder mixture (Van Geet et al., 2005) and compacted bentonite/quartz mixture (Kawaragi et al., 2009). μCT consists firstly of recording a set of two-dimensional X-ray radiographs of an object at several angles (typically at 180° or 360°) and secondly in reconstructing the 3D slices from the radiographs using a mathematical algorithm. The final 3D image of the internal structure is obtained by stacking the slices. The final measurement is the attenuation coefficient to X-ray which depends on the mass density and the atomic number of the object (Ketcham and Carlson, 2001; Van Geet et al., 2005).

The μCT scans presented here were carried out with the device of the Navier laboratory (Ecole des Ponts ParisTech), an “Ultratom” device specifically designed and manufactured by RXsolutions (France). Images were reconstructed using the software Xact-reconstruction developed by RXsolutions. The system is a dual-head and dual-imager scanner; two sources [a nano-focus xs-160hpnf/GE-Phoenix (160 kV, 15 W, $0.9 \mu\text{m}$ min spot size) and a micro-focus xs-225d/GE-Phoenix (225 kV, 320 W, $5 \mu\text{m}$ min spot size)], two interchangeable imagers: [HD camera PhotonicScience VHR (4008×2672 pixels, $9 \mu\text{m}$ pixel

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