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New technique for pore-space investigations of clay materials: A case study on Boom Clay - reference host formation for geological disposal of radioactive waste in Belgium.

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Oligocene Boom Clay is being investigated as a reference host rock formation for high-level and long-lived radioactive waste disposal in Belgium since several decades. Reasons for this are its favourable chemical and physical properties (e.g. low permeability, self-sealing capacity, retention abilities by sorption of radionuclides as well as chemical and microstructural homogeneity at formation scale and high stability over geological time-scale).

The knowledge of pore sizes and pore morphologies is important to improve the understanding and modelling of diffusion controlled transport processes of radionuclides in argillaceous materials. Furthermore the relationship between safety-relevant macroscopic properties of a material and its microstructure is a key element of the long-term evaluation of a repository.

However, due to the technical difficulty to investigate microstructures smaller than 1 μm in size, the study of heterogeneities at pore-scale is still difficult.

In this contribution we used the powerful combination of Broad Ion Beam (BIB) cross-sectioning and high resolution SEM-imaging, to get a direct insight into pore-structures smaller than 1 μm in size with unprecedented image quality and a resolution down to 10 nm.

Three samples from different Boom Clay horizons, covering the whole range of grain-sizes and mineralogical compositions, were investigated: Two stem from the upper part of the Boom Clay formation, of which one is a very fine-grained example of Boom Clay with a comparatively high clay-content and one an extremely coarse-grained sample, containing a higher amount of non-clay minerals; the third sample is from the level of the HADES research laboratory (226 meters depth) and ranges in between the other two extremes from a grain-size and mineralogical point of view.

Preliminary results show that samples with similar grain-sizes and mineralogy have as well similar porosities. The two finer grained samples exhibit lower apparent porosities than the coarser grained sample (15 respectively 16.5 % vs. 21 %). More than 99 % of the pores in the fine-grained samples were found in the clay-matrices and around 90 % of them had an equivalent diameter smaller than 250 nm, with a peak of pore sizes at 35 nm. In both samples largest pores were around 2 μm in size. In the coarser grained sample a considerably higher amount of pores ($\sim$ 15 %) was located at the interface between the clay-matrix and non-clay minerals and the maximum pore size was $\sim$ 20 μm.

The interesting point is that if we focus our observations on the clay-matrices only, pore-statistics gives similar results for all samples: Pores in the clay-matrices follow a power-law size distribution with a fractal dimension between 1.9-2.1 (similar to Desbois et al., 2009) and are often elongated with their longest axis being oriented parallel to the bedding of the samples.

When extrapolating the pore-size distributions to smaller as well as to larger values, total porosities between 27 % (most fine-grained sample) and 66 % (most coarse-grained sample) were calculated. These values are in good agreement with results obtained from traditional porosimetry (Boisson, 2005).

The unexpectedly high value for the coarse-grained sample might be due to cracks (esp. at the interface between the clay-matrix and non-clay minerals), developed during the drying of samples.

The approach used in this contribution has a high potential to bring out a new concept for porosity investigations on argillaceous materials by bridging the information based on microstructures at pore-scale with macro-scale properties.