Microstructure of Bentonite and Dilute Water Erosion

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Why to study microstucture?

MX-80 bentonite is considered as a possible material for a clay buffer in high-level nuclear waste repositories.
The evaluation of bentonite applicability is based on macroscopic experiments and modelling. As all macroscopic properties stem from the microscopic properties it is essential to know how the microstructure relate to properties observed in experiments.

Materials and methods

The microstructure of bentonite is studied using a set of complimentary methods. Small-angle X-ray scattering (SAXS), nuclear magnetic resonance (NMR), ion exclusion (IE) and transmission electron microscopy imaging (TEM) are used to characterize the samples' structure. Our approach included state of the art preparation of the frozen and embedded samples for TEM using high pressure freezing (HPF) method and novel NMR measurements in low temperatures.

The materials studied were purified Ca-[1] and Na-montmorillonites and MX-80 bentonite. The influence of different procedures of preparation of water-saturated, compacted bentonite samples was tested[2]. The information on microstructure has been collected from the range of different compaction levels and different salinity conditions.

Results

It has been demonstrated that the different ways of sample preparation have influence on the sample microstructure[2]. An example is shown in Figure 1, where SAXS patterns of compacted MX-80 bentonite prepared according to various procedures show clear differences.

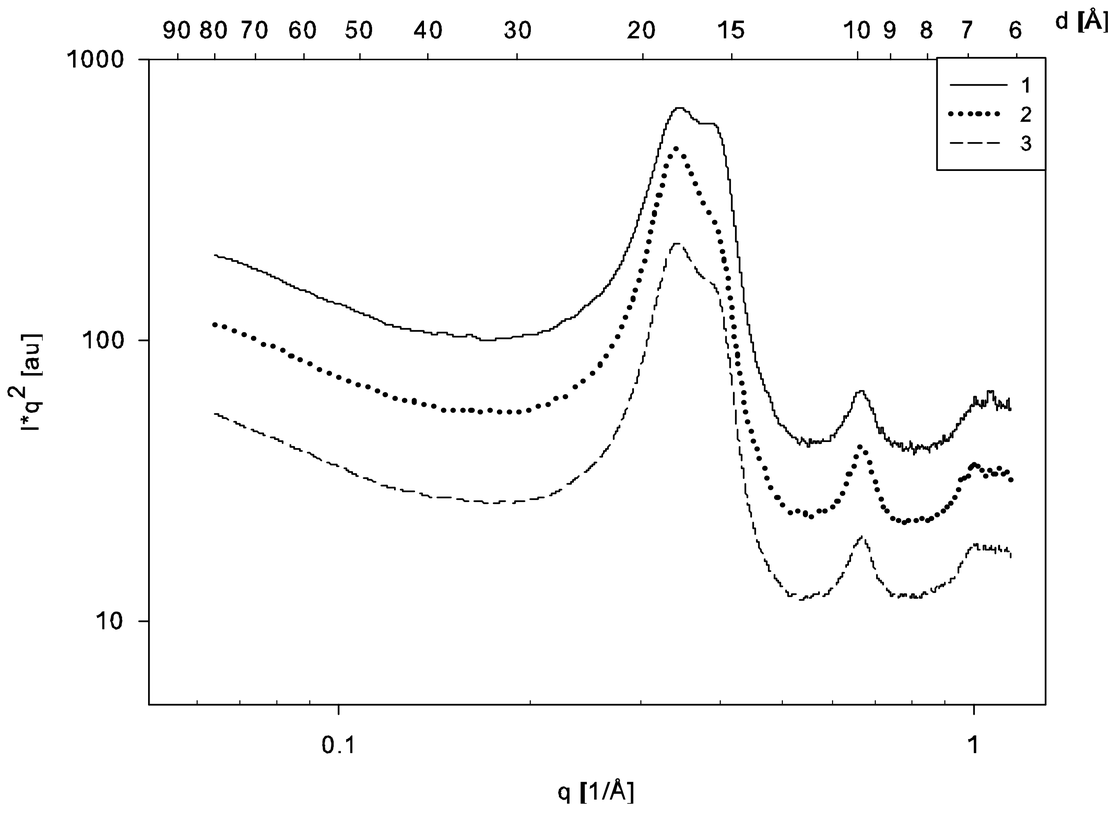

Figure 1. X-ray scattering curves of the MX-80 samples at 1.5 g/cm³ dry bulk density. Equilibration time 53 days. Numbers in the legend correspond to different ways of sample preparation: 1 – compacted to final density; 2, 3 – compacted to 1.8 g/cm³ and swelling to final density; 1, 2 – saturated with MilliQ water; 3 – saturated with 0.1M NaCl[2]

Combining the information from SAXS, NMR and IE quantification of the volume of the slit-like pores between the clay layers (interlamellar (IL) pores)) has been made and compared with the total pore volume. Figure 2 shows the plot of the volume of IL pores in Ca-montmorillonite and in MX-80 bentonite based on the SAXS calculation as a function of dry density of the clay. A clear tendency of forming larger stacked structures is visible for the calcium montmorillonite than for the predominantly sodium MX-80 bentonite.

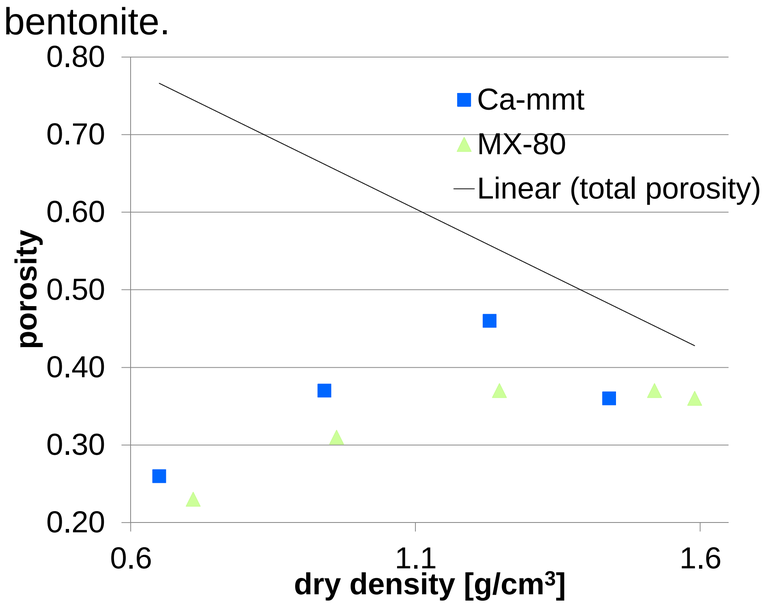

Figure 2. Estimation of the interlamellar porosity calculated basing on SAXS patterns of the Ca-montmorillonite and MX-80 samples saturated with MilliQ water. The solid line corresponds to the total porosity of the sample.

The TEM studies showed a clear difference in the microstructure of the MX-80 bentonite and the montmorillonite obtained by the purification of MX-80. Apart from removing the accessory minerals, aggregates of clay layers have been destroyed and montmorillonite layers organized differently. As can be seen in the Figure 3 the platelets seem to be more oriented in the purified clay, whereas in the MX-80 their orientation appears more random. This effect could be caused by the sedimentation after the purification process or the uniaxial compression method used to prepare the sample[3].

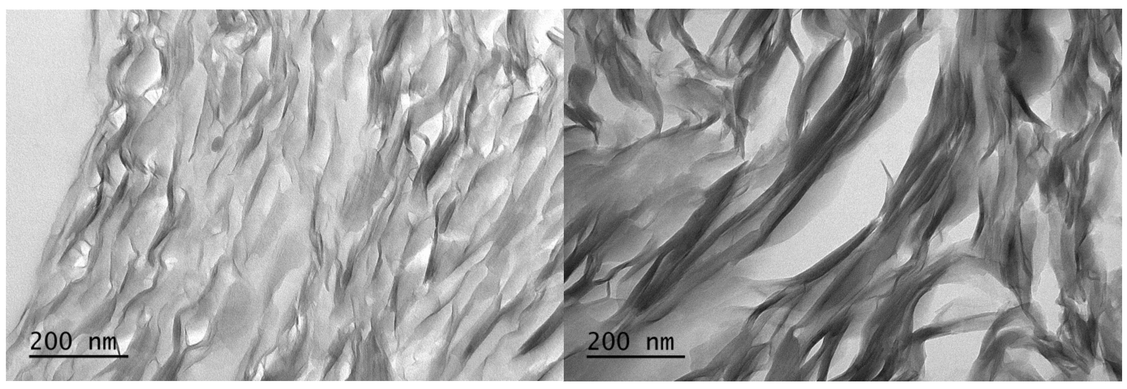

Figure 3. TEM micrograph of Na-montmorillonite (left) and MX-80 bentonite (right) magnified 23 000 times. Both samples have the bulk dry density of 0.7 g/cm³ and have been saturated with 0.1 M $NaClO_4$ solution.

Main applications

The knowledge acquired by the investigation of the microstructure is used to increase the basic understanding of the material and for bentonite model development. Especially, this knowledge is needed to understand the evolution of bentonite structure to a state where bentonite can erode. Consequently, it is an important contribution to ensure the safety of a deep geological disposal of high-level nuclear waste.

References

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[2] M. Matusewicz, V.-M. Pulkkanen, and M. Olin, "Influence of the sample preparation on MX-80 bentonite microstructure," accepted manuscript, Clay Minerals

[3] J.-P. Suuronen, M. Matusewicz, M. Olin, and R. Serimaa, "X-ray studies on the nano- and microscale anisotropy in compacted clays: Comparison of bentonite and purified calcium montmorillonite," Appl. Clay Sci., vol. 101, no. 0, pp. 401–408, 2014.

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