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Layer charge characterization of smectites by different methods in the FEBEX in situ test

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Layer charge is a relevant parameter in smectites and related clay minerals. It plays a significant role in metastable contexts due to its capacity to reflect physicochemical changes in the environment.

The FEBEX Project is an "in-situ" real-scale test performed throughout more than 18 years in Grimsel, Switzerland. It consists of compacted bentonite, mainly composed of smectite (> 92 wt.% montmorillonite), isolating the canisters (simulated by heaters) from the hosting rock. Its main objective was to demonstrate the viability of the engineered barrier system (EBS) as a high level nuclear waste (HLW) disposal in a crystalline host rock. Therefore, one of the goals of FEBEX was the verification of the long term stability of the clay barrier after years of natural hydration and heating (up to 100 C). In this work, in order to test the behavior of the clay barrier, we have focused our studies in the evolution of the layer charge of the smectites (ξ), as one of the main parameters which controls smectite properties, such as swelling, cation exchange capacity and ion exchange between the clay and fluids in the EBS.

During the last phase of the project (FEBEX-DP Project), the dismantling of the second heater was performed and different bentonite sections were analyzed.

In this study the samples belongs to sections located at different distances, both vertically and laterally, to obtain possible variations of the layer charge along the barrier which could imply structural changes in the smectite. To characterize the layer charge we have used 4 different methodologies: a) structural formula method by means of X-ray fluorescence data (SFM $_{XRF}$), b) structural formula method determined by electron transmission microscopy (SFM $_{TEM}$), c) the alkylamonium method (AAM), according to the rapid estimation proposed by Olis et al. (1990), and d) infrared spectroscopy method (IRM), as proposed by Petit et al. (1998). The obtained values were compared to total CEC values measured with a 0.01 M copper triethylenetetramine solution according to Ammann et al. (2005).

The obtained layer charges were in the field of smectites. ξ values were between 0.36 and 0.39 eq/O₁₀(OH)₂, while the CEC values ranged from 88 to 110 meq/100g. Every used methodology shows there are no significant variations in the layer charge of the analyzed smectites.

We conclude that there are no significant changes in the layer charge of the smectite particles after the 18 years of hydration and heating. Therefore, the bentonite from the FEBEX in situ test maintained its performance as an engieneered barrier.

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