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Hydratation properties of the dehydroxylated-rehydroxylated smectites

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Na- and Ca-exchanged Wyoming montmorillonites have been dehydroxylated by heating during 1 hour at 750°C and rehydroxylated by hydrothermal treatment (350°C, 100MPa) in a Tuttle type pressure vessel with different staying time. Due to the octahedral cations migration induced by the dehydroxylation, the initial layers which are *cv* (vacant octahedral sites in *cis* positions) become *tv* (vacant octahedral sites in *trans* positions) after the treatment [1]. This transformation is accompanied by a motion of the interlamellar cations to compensate the local modifications in the distribution of charges [1, 2]. In the dehydroxylated state the cations go down in the hexagonal holes.

The following results show that the coming back of the initial position is characterized by an intermediary state in which the hydration properties are reduced.

FTIR spectra presented in figure 1 shows the evolution of the bending band of the OH groups between 1 and 10 days of the rehydroxylated treatment. The mean band position increases with the staying time and shifts progressively to the position corresponding to the initial sample indicating the existence of an intermediary state before the total réhydroxylation.

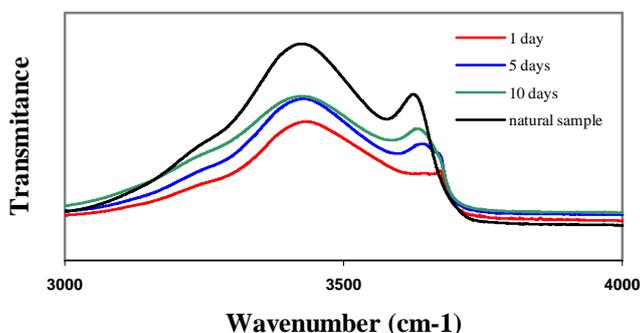


Figure 1 : FTIR spectra of the Wyoming Na-montmorillonite after 1, 5 and 10 days in Tuttle type pressure vessel.

Some experimental results confirm the existence of the intermediary state with reduced the hydration properties of the smectite. For example, after 5 days of treatment, in comparison with the natural state:

- The ability of the samples to form viscoelastic dispersions disappears.
- The number and proportion of the zero water layers, determined with Small Angle X-ray Scattering patterns simulation increased.
- Cation exchange capacity (CEC) performed with a washing cobaltihexamine solution showed an important decreasing of the CEC ($\cong 40\%$).

- The mean shift of the bending band position $\Delta\nu_{OH}$ changed with the interlayer cation ($\Delta\nu_{OH} \cong 32 \text{ cm}^{-1}$ for the Na cation and $\cong 7 \text{ cm}^{-1}$ for the Ca one) indicating a sensibility for the OH group to the cation position.

These results can be explained because of a mean deeper position of the interlayer cations in the hexagonal holes of the tetrahedral sheet, producing a nonswellable structure.

[1] F. Muller, A. Plançon and V.A. Drits, 2000. J.Phys. IV, **10**, 481-487

[2] K. Emmerich, F. Thule Madsen and G. Kahr, 1999, *Dehydroxylation behavior of heat-treated and steam-treated homoionic cis-vacant montmorillonites*. Clays Clay Miner., **47**, 591-604.