| ESRF | **Experiment title:** WAXS study of the deformation of dehydrated nano-silicate clay particles during water intercalation | **Experiment number:** 01-02 718 |
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| **Beamline:** BM01A | **Date of experiment:** from: 04/052005 to: 07/05/2005 | **Date of report:** |
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Report:

In this experiment, we addressed a situation in which dehydrated fluorohectorite particles behave as intercalation compounds inside of which water molecules intercalate mono-layer by mono-layer. The number of intercalated water layers depends on the ambient humidity and temperature. The pure hydration states and the hydration transitions have been studied from data obtained at the synchrotron in Brookhaven (USA) in 1999, using a point detector [1, 2]. Dried-pressed samples exhibit a strong anisotropy of the particles' orientations, since most particles ly with their planes nearly perpendicular to the direction of the applied pressure. Most Bragg diffraction from the stacks occurs along a direction perpendicular to the mean particle orientation. Spectra measured along a direction parallel to the mean particle orientation, that is, perpendicular to the mean meso-sheets of the clay particles, are denoted "in-plane peaks". They account for the deformation of the nano-silicate lattices during water intercalation: the hypothesis is that during transition, water molecules coming between platelets can, by thermal agitation, and before having found its equilibrium position, hit fluor and make them move from the original position to (in average) thrust them deeper into the platelet, with the effect of contracting the unit cell and making the in-plane peak move slightly. Hence, the way the peaks' position, amplitude and shape change during the transition were expected to provide information on the deformation of the nanosilicate lattice during water intercalation.This hypothesis is mentioned in the literature [3] and observations on previous data of ours had suggested a slight change in the main peak abscissa.

Our previous ESRF experiments, as well as most similar experiments present in the literature, were designed for an optimal measurement of the peaks that are characteristic of the clay structural stacks. In this experiment, nr. 01-02 718, we chose to position the samples perpendicular to the incoming beam, so that mose scatterers would meet the Bragg condition for in-plane scattering. The main goal of the proposal was to obtain good two-dimensional data of these in-plane peaks, with a satisfying statistics, so as to be able to monitor their evolution during the hydration transition.

This main goal of the experiment was unfortunately not met, as the analysis of the obtained fine resolution (0.01 Å) data showed no evidence of a shift in the position of the in-plane peaks. The so-called "in-plane peaks" were observed to have an asymmetrical shape, resulting from them consisting in fact of a complex assembly of several peaks. While the maximum, corresponding to the real in-plane peak (i.e. issued from the in-plane periodicity of crystal unit cells in a platelet), does not move, others peaks positioned very close to it, and therefore being part of the observed "complex" peak, such as multi-Miller indexed peaks (101, 102,...), do move during water intercalation, because of the displacement of the 001 peak, and this makes the resulting shape of the "complex" peak evolve.

These interesting findings were made possible by the very good resolution of the setup at beamline BM01A, as well as the chosen sample-to-beam geometry, which was specially designed at measuring in-plane peaks, as mentioned above. The experiment therefore resulted in a significant complement to our knowledge of our systems. However, we did not consider these findings as important enough in themselves so that they could be the topic of an independent publication. They might be published later together with results from other related experiments. This experiment, together with complementary thermogravimetry measurements, has been the core of a 6-month student project (Simon Dagois-Bohy, student at cole Normale Suprieure in Paris); it is well documented in the project report [4]. Note also that the experiment was coupled to a SAXS experiments carried out later that year (july 2005) at beamline 26B on the same sample. The SAXS experiment did provide more immediate results and lead to a publication [5].

# References


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