Experimental report

21/03/2016

Proposal: 9-10-1398 **Council:** 10/2014

Title: How the water dynamics is modifiedthrough the hydration process in clay minerals?

Research area: Chemistry

This proposal is a new proposal

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Samples: Li1.2 Mg4.8 Li1.2 Si4 O10 F2
Ni0.6 Mg4.8 Li1. 2 Si4 O10 F2
Na1.2 Mg4.8 Li1.2 Si4 O10 F2

Instrument	Requested days	Allocated days	From	To
IN5	4	4	01/05/2015	05/05/2015
IN16B	4	2	22/06/2015	24/06/2015

Abstract:

Swelling of layered smectite particles causes changes in the interlayer repetition basal distance (d-spacing) as a function of temperature and humidity allowing for their application in a number of different fields ranging from the food industry to waste management. Here we will use the synthetic smectite fluorohectorite as model to understand the water interactions with different interlayer cations and how these interatctions change as a function of relative humidity (RH) . Three samples will be studied Na- fluorohectorite, Li-fluorohectorite and Ni-fluorohectorite, where Na, Li and Ni are the respective interlayer cation, each at two different hydration state. The main objectives of the present study are to determine the influence of the interlayer cation in the water mobility as well as to follow how the dynamics and geometrical environment of the intercalated water is modified by the hydration process.

EXPERIMENTAL REPORT

EXPERIMENT N°: 9-10-1398 INSTRUMENT: IN5

DATES OF EXPERIMENT: 01.05.2015 – 05.05.2015

TITLE: How the water dynamics is modified through the hydration process in clay minerals?

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Date of report:

The aim of the experiment was to verify how the water mobility in fluorohectorite clays is influenced by the interlayer cation along the different hydration paths, and to confirm if these different hydration lead to different diffusion coefficients of the water into the studied clays.

In order to do that, the NaFh, LiFh and NiFh samples were dried (RH = 0%) in an oven with vacuum, or exposed to different relative humidity (33%, 55% and 70%) by using saturated salt solutions and a dissector. Due to the low amount available, the NiFh samples were only prepared at RH 70%, resulting in a total of nine different samples to be measured.

The IN5 measurements were done using standard rectangular flat sample holders in four different temperatures (150K, 200K, 250K and 300K) and two wavelengths: 5Å and 8Å. In the case of 5Å, the data were collected in 15 min and, in the case of 8Å, in 30 min. An empty cell

and a Vanadium sample were also measured in these same conditions for background and resolution corrections. From figure 1 we compare the quasielastic signal for all clay samples at 300K, RH = 70% and Q = 1.54 $Å^{-1}$ at both resolutions. It seems that the valence of the interlayer cation indeed influences the mobility of the H_2O molecules. The generalized density of states (GDOS) for all samples at the same conditions is depicted in figure 2. This result also indicates for the monovalent cations, i.e. Li^+ and Na^+, water the behavior similar, while in the case of the divalent cation, Ni^{2+}, the spectral weight is shifted to lower energies. The data is now being analyzed as a function of temperature and RH.

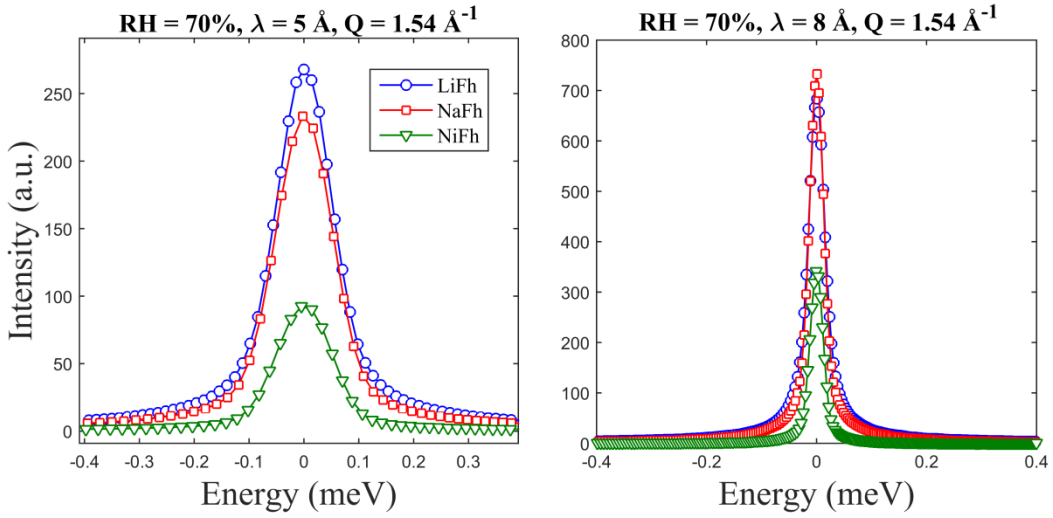

Figure 1: QENS spectra of LiFh, NaFh and NiFh at the same condition. The only difference between these samples is the interlayer cation.

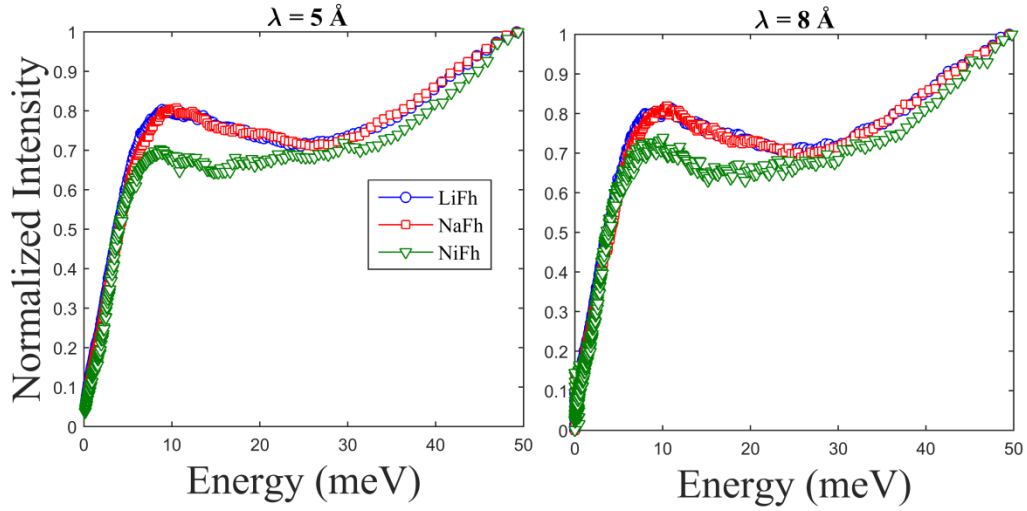

Figure 2: Generalized density of states for LiFh, NaFh and NiFh.