Experimental report

16/06/2022

Proposal: DIR-230 **Council:** 10/2020

Title: Unraveling the structure of water clusters in microporous aluminophosphates AlPO4-34 and AlPO4-LTA

Research area: Materials

This proposal is a new proposal

Main proposer: Janez VOLAVSEK

Experimental team: Gabriel Julio CUELLO

Local contacts: Henry FISCHER
Gabriel Julio CUELLO

Samples: AlPO4(H2O)x
[(SiO2)(AlO2)w]Hw(H2O)x

Instrument	Requested days	Allocated days	From	To
D4	4	4	08/06/2021	12/06/2021

Abstract:

Microporous aluminophosphates AlPO4-34 and AlPO4-LTA are among the most promising materials for sorption-based heat storage. They store energy in their dry state and release it during hydration. With the proposed project, we are aiming to gain insight into the mechanism of (de)hydration of these materials with neutron total-scattering experiments. In particular, we would like to obtain information about the short-/mid-range order of the hydration-induced framework deformation and about the structure of hydrogen-bonded water clusters within the pores, because they are considered decisive for energy storage performance.

Unravelling the structure of water clusters in microporous aluminophosphates $AlPO_4$-34 and $AlPO_4$-LTA

Results

The major data obtained from the experiments are atomic pair-distribution functions (PDFs) of hydrated and dehydrated aluminophosphates $AlPO_4$-LTA and $AlPO_4$-34. All PDFs were produced using reciprocal data up to Qmax = 23.5 $Å^{-1}$. We are particularly interested in the positional correlation length of disordered hydrated $AlPO_4$-LTA (dehydrated is structurally very ordered), which was observed to be very short (Figure 1), telling us there is only short range order within water-clusters inside this microporous material. This is especially important, since X-ray PDF shows a medium range order, which very likely originates from the framework.

The experimental data, which we consider secondary, was also collected for two additional solid state materials, specifically two zeolites of which framework structure does not significantly change when drying, but have the same topology as $AlPO_4$-LTA. These zeolites may serve as references when analysing water contribution to the PDFs.

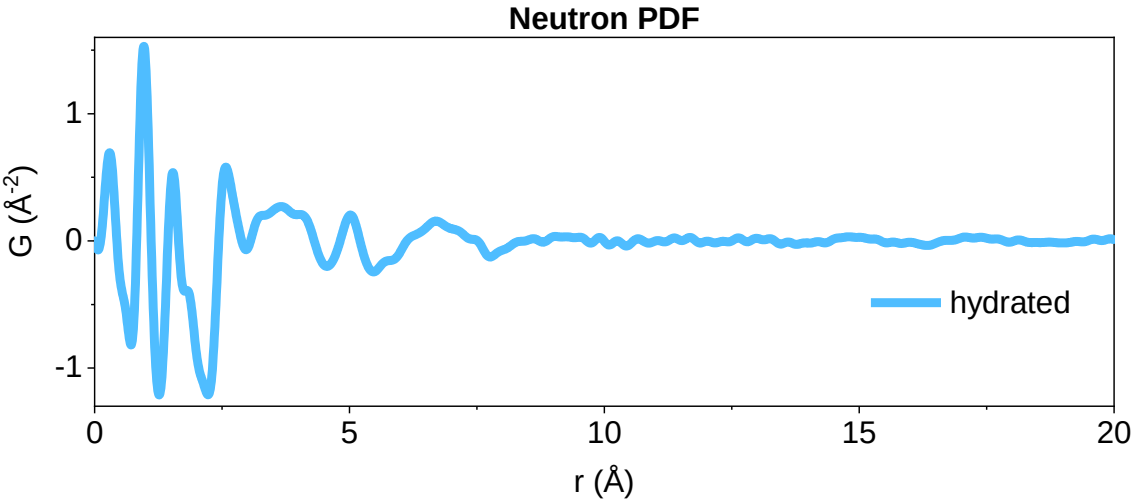

Figure 1: neutron PDF of hydrated aluminophosphate $AlPO_4$-LTA at room temperature.

Conclusions and future work

This will enable us to analyse local atomic structure of wet and dry materials in question; this is especially relevant for the wet material, since most of the scattering signal is contained within the broad diffuse scattering, making it impossible to analyse with traditional crystallography. Furthermore, this data might help us to confirm a proposed mechanism of wetting/drying from our previous study [1].

A special interest to us is the $AlPO_4$-LTA in the wet (very disordered) phase, of which atomic structure of the framework and of the water clusters within them is currently unknown and impossible to obtain from traditional experimental methods.

The future work will include combining data from neutron PDF (presented in this report), X-ray PDF, solid state nuclear magnetic resonance spectroscopy (NMR) and computer simulations to explain what happens during drying of these materials and what is the atomic structure of the materials deformed framework and contained water clusters in a wet state.

References

[1] A. Krajnc, G. Mali et al.; *Adv. Energy Mater.*, 2017, 7, 1601815—1601815