

# Experimental report

22/03/2021

**Proposal:** 6-07-62

**Council:** 10/2019

**Title:** Dynamics of water confined inside inorganic nanotubes with variable hydrophilicity: a continuation

**Research area:** Physics

**This proposal is a resubmission of 6-07-41**

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**Samples:** (OH)AlOGe(OH)

Instrument	Requested days	Allocated days	From	To
IN16B	6	0		
WASP	8	5	11/03/2021	16/03/2021

## Abstract:

Imogolite Nanotubes (INT) of interest for our team are inorganic nanotubes having an inner diameter of ~1 to 3 nm, with nominal formula  $(\text{OH})_3\text{Al}_2\text{O}_3\text{Ge}(\text{A})$ ; from the outside surface to the core surface of the nanotube. Changing the internal group  $\text{A}=[\text{OH}, \text{CH}_3]$  allows to vary the hydrophilicity of the inner surface of the tube (modification of water-surface interactions). Our objective with these versatile nanochannels is to obtain a consistent set of new data about the multiscale dynamics of water in model nanoporous media with variable hydrophilicity. In this continuation proposal, we want to focus on the melting process of the inner hydration layer, in strong interaction with the inner surface of the nanotube. For that we want to use IN16B to understand the microscopic diffusion mechanism of this hydration layer.

## Experiment #6-07-62

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Local contact: P. Fouquet

The experiment took place the 11-16 march 2021.

Four samples, composed of Single Wall Imogolite Nanotubes (SWINT) with formula  $(\text{OH})_3\text{Al}_2\text{O}_3\text{Si}(\text{OH})$ , were prepared at different hydration states: the dry sample, and three hydrated samples with 3wt%, 6wt% and 11wt% of  $\text{H}_2\text{O}$  respectively.

With WASP we wanted to measure 1) the dynamics of the hydroxyls of the nanotube itself 2) the diffusion of water molecules confined into the nanotube. For this purpose, we measured the echo of the dry sample at 2, 300 and 400K and of the three hydrated samples at 2, 250 and 300K. The incident wavelength used was  $4 \text{ \AA}$ , in order to access the Q-range  $Q=[0.4, 2] \text{ \AA}^{-1}$  and the timescale range 1ps-1ns. The two hydrated states 6wt% and 11wt% were also investigated with an incident wavelength of  $7 \text{ \AA}$ , in order to cover the low-Q range and an extended time window, up to 6 ns.

The results show:

- 1) The dynamics of the hydroxyls being inactive at 300K, but appearing at 400K.

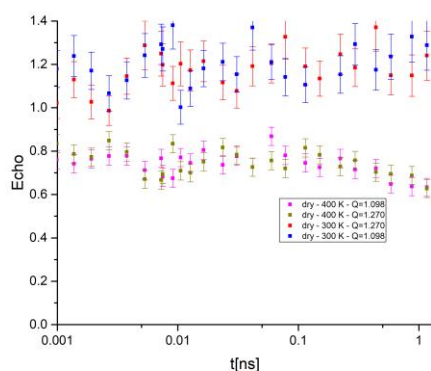


Figure 1. Echo for the dry sample, at 300K and 400K, for  $Q=1.098$  and  $Q=1.270$

- 2) The dynamics of water molecules appearing already at 250 K and increasing in intensity at 300K. All the data showed almost the same intensity loss in the echo as a function of time, with a little dependence on the water content and on Q.

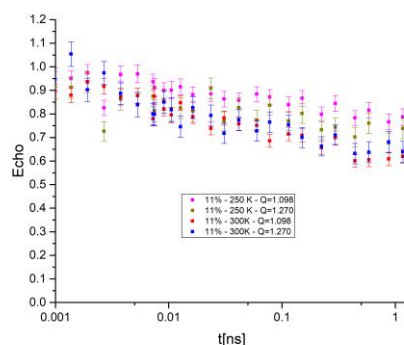


Figure 2. Echo for the hydrated 11wt% sample, at 250 K and 300K, for  $Q=1.098$  and  $Q=1.270$