Experimental report

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Proposal: **CRG-2764** **Council:** 4/2020

Title: Transport of water in soft confinement

Research area:

This proposal is a new proposal

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Samples: PIM (polymer)

Instrument	Requested days	Allocated days	From	To
IN6-SHARP	2	2	01/09/2020	04/09/2020

Abstract:

Experimental report

Context

Transport of water in soft porous materials is relevant to a broad range of applications but remains unclear in many aspects because of several effects that have to be considered: surface chemistry, diffuse boundaries and deformations or mechanical effects. The aim of this project is to experimentally investigate the interplay between mechanical deformation, structure and transport. QENS experiments at ambient temperature combining hydrostatic pressure and mechanical stress on the time-of-flight spectrometer (IN6) have been performed on the selected porous material (PIM, Intrinsic Microporosity Polymer). Complementary experiments will be performed in November 2020 on D2am (ESRF) to characterize the evolution of the PIM-1 matrix structure under such confinement conditions. PIM-1 presents an interconnected porosity at the nanometre scale (< 2nm).

Results

Experiments were performed using a homemade high-pressure cell made of 7075 aluminium alloy. The following measurements have been carried out: empty cell, dry and hydrated PIM-1. Different conditions of mechanical stresses and hydrostatic pressures have been tested. S(ω) spectra for the dry and hydrated sample are shown in Figure 1. The dry sample gives an elastic peak confirming that the QENS signal that appears for the hydrated sample is purely linked to the water dynamics.

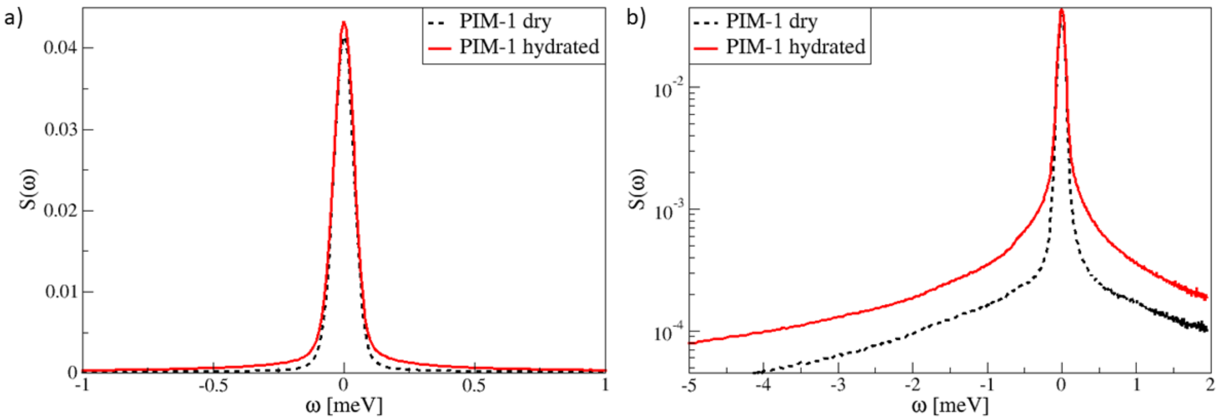

Figure 1: a) Sum over Q, from 0.4 to 2.0 Å^{-1}, of the quasi-elastic spectra of the dry and hydrated sample b) Log-plots to highlight background and quasi-elastic contributions

Some S(ω) spectra obtained at different experimental conditions are presented below. In Figure 2a, the mechanical stress is fixed at 8 Nm and we can see a slight influence of the water pressure above 100 bars, highlighted by the small increase of the intensity between 100 bars and 300 bars. Similar results have been obtained with a mechanical stress fixed at 5 Nm and 12 Nm. Moreover, in Figure 2b, the hydrostatic pressure is fixed at 300 bars, and the influence of the compression level is slightly visible. Similar results have been obtained at 14 and 100 bars of hydrostatic pressure.

QENS spectra were fitted by two Lorentzian functions supposing that the first one corresponds to translational motion and the second to rotational one, and assuming that both motions are decoupled. The translational motion was described with the jump diffusion model. We note that the analysis of the water dynamics has been performed including the dry PIM-1 spectra as a background contribution considering a sample transmission of 0.95. Numerical values of the extracted fitting parameters are given in Table 1. The influence of the water pressure and mechanical stress cannot be estimated with the extracted fitting parameters because of the poor differences measured on the QENS spectra.

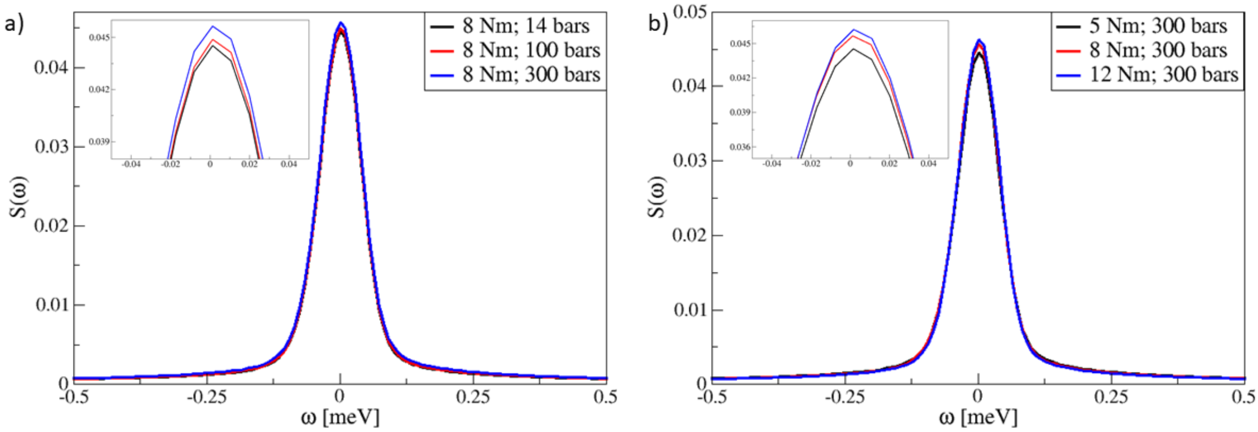

Figure 2: Sum over Q, from 0.4 to 2.0 Å^{-1}, of the quasi-elastic spectra of hydrated PIM-1 at different conditions a) at three different hydraulic pressures under constant mechanical stress (8 Nm) b) at three different mechanical stresses under constant hydraulic pressure (300 bars)

Table 1: Extracted fitting parameters

Water pressure [bar]	Mechanical stress [Nm]	D_T x10^{-5} [cm^2/s] - self diffusion coefficient	τ - residence time [ps]	D_R [ps] - rotational diffusion constant
14	3	3.23	0.61	2.88
14	5	3.14	0.64	3.07
100	5	3.25	0.66	2.96
300	5	3.32	0.73	2.78
14	8	3.21	0.76	2.73
100	8	2.96	0.67	2.66
300	8	3.13	0.77	2.83
14	12	3.13	0.55	3.12
100	12	3.12	0.56	3.1
300	12	3.09	0.6	3.04

Conclusion

The very small differences observed between the different tested conditions are probably linked to the small pores' diameters of the selected sample, limiting the measurable effect of the different applied mechanical stresses and hydrostatic pressures.