Experimental report

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Proposal: **CRG-2669**

Council: 4/2019

Title: Transport of water in soft confinement: influence of surface chemistry

Research area:

This proposal is a new proposal

Main proposer: **Marie PLAZANET**

Experimental team: Judith PETERS
Alexander SCHLAICH
Julie WOLANIN
Marie PLAZANET
Benoit COASNE
Gautier MEYER
Wanda KELLOUAI

Local contacts: Jean Marc ZANOTTI

Samples: PIM-1 (C, N, O, H polymer)

Instrument	Requested days	Allocated days	From	To
IN6-SHARP	4	4	03/10/2019	07/10/2019

Abstract:

Experimental report

Context of the project TWIST (Transport of Water In Soft ConfinemenT):

The aim of this project is to provide a predictive description of water transport in soft porous materials. Soft matter is indeed subjected to several interplaying effects that influence transport at the nanoscale (surface heterogeneities, deformation, swelling effect…). We focused our study on the Polymer with Intrinsic Microporosity (PIM-1, see Figure 1a et b). This rather hydrophobic material presents an interconnected porosity at the nanometre scale (< 2nm). A home-made 7075 aluminium pressure cell (Figure 1c) has been specially designed in order to apply a mechanical compression of the polymer in the direction perpendicular to the applied hydraulic pressure.

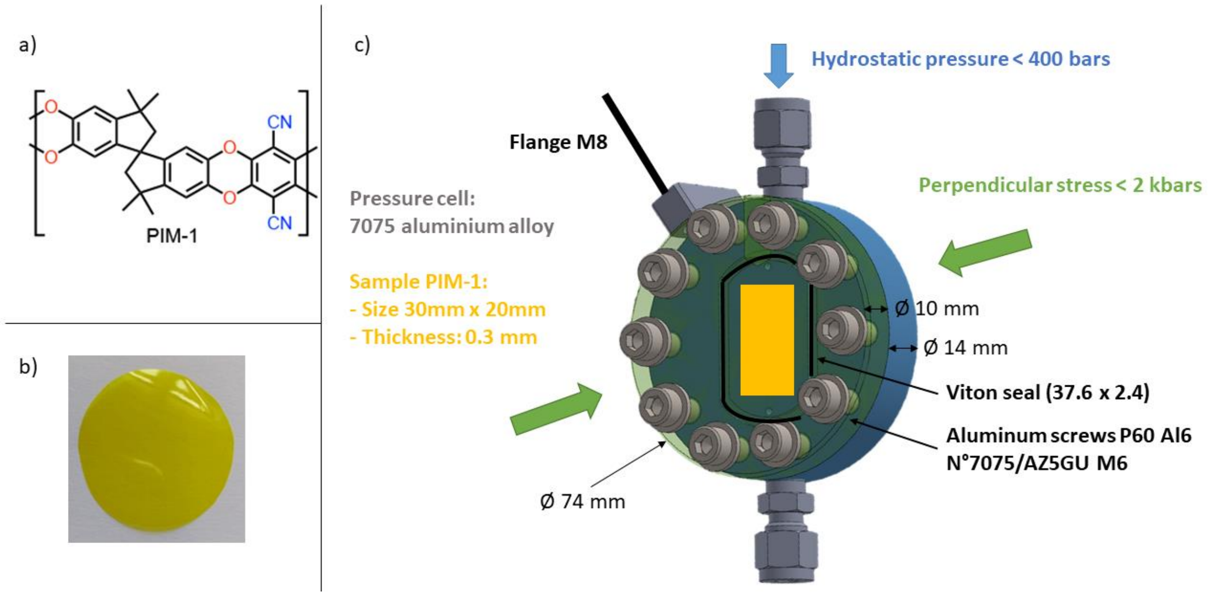

Figure 1: a) PIM-1 structure b) PIM-1 sample c) Homemade aluminium pressure cell. The mechanical stress (< 2 kbars) is applied ex-situ with a torque wrench ensuring a proper tightening level (to a fixed torque value) of the screws.

Results:

Two types of experiments have been carried out. First, measurements under a water pressure gradient. In this case, the cell was connected to a Karcher K7 ensuring the water flow through the cell, between an initial pressure (14-100 bars) and atmospheric pressure. The second type of measurements have been performed under static hydraulic pressure which was applied with a manual pump and increased up to 200 bars.

In both type of measurements, static pressure or gradient, we did not observe any effect of the hydraulic pressure (14, 50 and 80 bars) at constant mechanical stress (tightening of screws at 1, 5 and 8 N/m corresponding to mechanical stresses, σ, of 100, 500 and 800 bars). However, results have shown a clear dependence of the confined water dynamics with the mechanical load (σ between 0 and 800 bars) whatever the hydraulic pressure. Figure 2a shows an example of the results obtained for a hydrostatic pressure fixed at 14 bars. The analysis of the experimental data provides a description of the translational and rotational motion of the water molecules confined in the PIM-1 pores. The results (Figure 2b) show a slowing down of the translational motion when the sample is being compressed. Furthermore, this analysis also shows that the rotational motion was not impacted by either hydraulic pressure or mechanical stress.

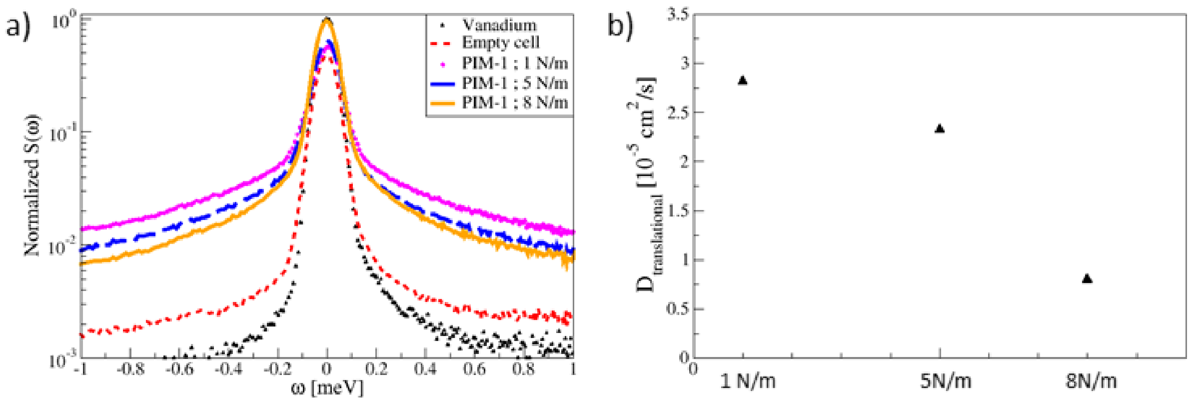

Figure 2: a) Quasi-elastic spectra of the vanadium, the empty cell and of the PIM-1 sample at three different mechanical stresses under constant hydraulic pressure (14 bars) b) Translational motion of the confined water

We also observed that beyond a certain level of mechanical stress (σ>800 bars) and hydrostatic pressure (>200bars), the dynamics of the confined water is dependent on the hydrostatic pressure. Indeed, we observe a 10% increase of the intensity of the elastic peak between 14 and 200 bars (Figure 3). These effects should be enhanced at higher pressures (our experiments were limited to 800 bars mechanical stress and 200 bars hydrostatic pressure). We assumed that this non-negligible difference in intensity could be attributed to the presence of a slower water population, giving a signal out of the IN6 resolution. Indeed, PIM-1 is rather hydrophobic but also contains some hydrophilic sites. The structural heterogeneities of the polymer matrix could be responsible for the different water dynamics observed.

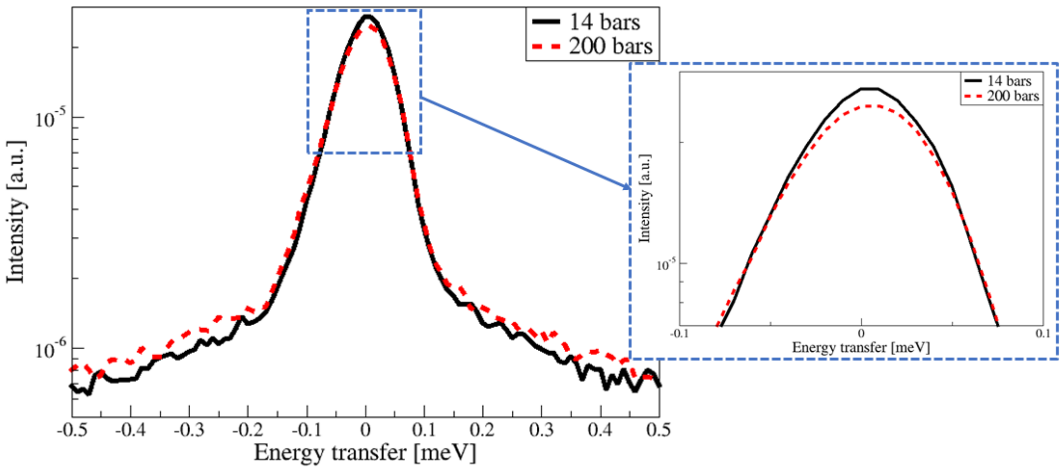

Figure 3: Raw spectra for hydrated PIM-1 at a constant stress of 800 bars and 2 hydrostatics pressures

Finally, the analysis of the generalized density of states in the inelastic region enables us to identify a series of peaks in the -20 meV region corresponding to the contribution of the aluminium cell. We also identify a peak for the confined water in the [-80 meV, -60 meV] region: the amplitude of this peak is clearly dependent of the mechanical stress.

Outlook:

Experiments are planned on D16 in May 2020 in order to characterize the structure of PIM-1 under such confinement conditions. In this way, we will be able to have a better understanding of the swelling of the matrix and its deformation under different hydrostatic pressures and mechanical loads.