Experimental report

03/02/2016

Proposal: 9-12-397 **Council:** 10/2014

Title: Methane adsorption in porous MetalOrganic Frameworks

Research area: Materials

This proposal is a new proposal

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Samples: Metal Organic Framework HKUST-1 C18H6Cu3O12
methane CH4
deuterated methane CD4

Instrument	Requested days	Allocated days	From	To
D33	2	0		
D11	2	0		
D22	2	2	24/06/2015	26/06/2015

Abstract:

The proposal is to use SANS to detect on a local scale the pore filling mechanism of methane in a new ultra-high adsorption capacity substrate (metal organic framework) that is mooted for gas storage systems. Unlike the situation with rigid substrates where the local properties of the adsorption process can be observed by SAXS, these systems swell significantly, making it necessary to use contrast variation to distinguish between the substrate and the profile of the condensed gas.

"Methane adsorption in porous Metal Organic Frameworks"

Experimental report on 9-12-397experiment

The aim of the experiment was to use SANS to detect on a local scale the pore filling mechanism of methane in a new ultra-high adsorption capacity substrate (metal organic framework (MOF)) that is mooted for gas storage systems.
The samples studied (Figure 1) were 2 variants of HKUST-1 MOF, which had been synthetized and characterized by standard adsorption methods before the experiment. During the measurements CH_4 and CD_4 adsorption was performed by the HIDEN adsorption apparatus provided by SANE, in the low pressure (< 1 bar) regime. SANS curves were recorded in the 0.002-0.2 $Å^{-1}$ q-range at 273 K. A representative set of curves is shown on Figure 1.

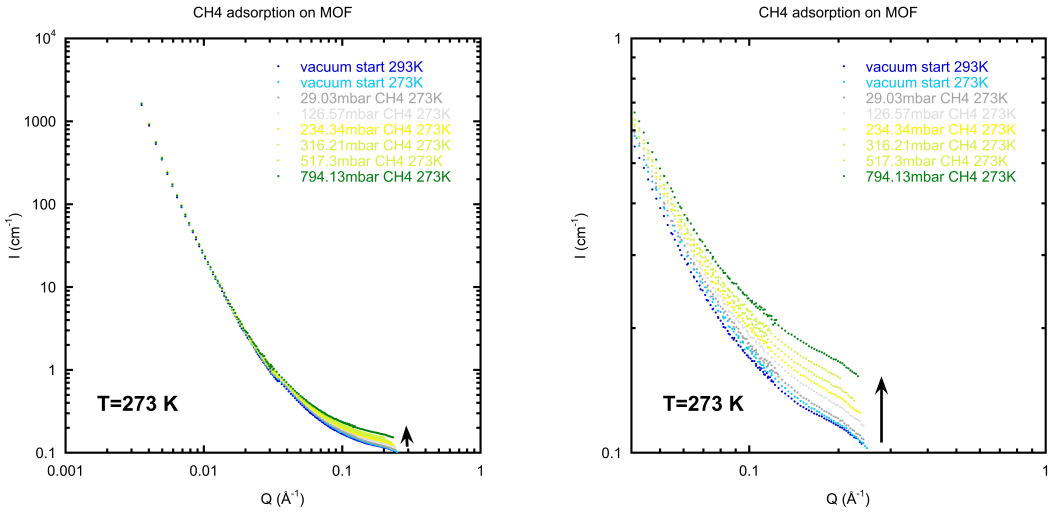

Figure 1. SANS response curves obtained on HKUST-1 upon adsorption of CH4. (a) Full curves (b) Zoom on the 0.05-0.2 $Å^{-1}$ q-range.

As can be seen from Figure 1, significant signal difference was observed in the higher q-regime as a function of the CH_4 loading, thus we could follow the adsorption of the gas. Measurements were performed in the desorption path as well. The two examined variant of HKUST-1 behaved differently during the measurements: while HKUST-1(1) showed compete reversibility in the adsorption/desorption of CH_4, the other examined sample, HKUST-1(2) irreversibly adsorbed a part of the CH_4 loaded. Further experiments, both with CH_4 and CD_4 in order to extract the coherent signal, are needed to explore the reason for this. The detailed data analysis is still in progress, further conclusions will be deduced once it will be finished.