Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics

Construction of quantitative molecular isotherms from FTIR analysis of dinitrogen (N_2) adsorption on microporous NaY zeolite

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Supporting Information

Experimental procedures and materials:

'One pot' manometric and IR measurements:

Progressive N2 adsorption at 77K was carried out for a self-supporting thin pellet of fully sodium-exchanged $Na_{56}Y$ zeolite (Union Carbide, Si/Al ~ 2.5, anhydrous weight m = 4.06 mg). Both manometric and molecular descriptions were simultaneously obtained in the IR cell.[1] IR spectra of NaY zeolite were recorded with a resolution of 2 cm^{-1}. The pellet of the NaY zeolites was first activated with a heating rate of 2 K min^{-1} up to 650 K and was held for 2 hours in a home-made designed cell connected to a vacuum adsorption system ($P < 10^{-3}$ Pa). This activation totally dehydrated the $Na_{56}Y$ zeolite (no residual water was IR detected). After activation, the wafer was cooled down progressively to liquid nitrogen temperature (77 K) and the background spectrum of the NaY zeolite was measured. The N_2 molecules were introduced stepwise from a control volume (2 cm^3) at 77 K by doses ranging from 0 to 24 μmol of N2. For each added dose, both the IR spectrum and the corresponding equilibrium pressure (PN2) were recorded thanks to a capacitive gauge (10^{-3} mbar or 0.1 Pa of exactness). Molecular investigations consisted of analyzing IR spectra through IR signatures of adsorbed dinitrogen.

Molecular N_2 isotherms constructions in the IR cell were derived from macroscopic PN_2 measurements. Calculations were made using adsorbed N_2 quantities that we deduced by correlating each equilibrium pressure (PN_2) to the N_2 doses added. For this task, the cell pressure was previously calibrated under the same conditions, but without a pellet. The number of adsorbed N_2 molecules were calculated and expressed per supercage based on the following data: molecular weight of anhydrous $Na_{56}Y$ = 12752 g/mol, 8 super cages per unit cell, a pellet having a surface area of S= 1.327 cm^2 and anhydrous weight of m = 4.06 mg. As a result, both molecular and macroscopic investigations were made in one pot in the IR cell and joined for a quantitative assessment with molecular accuracy.

1. O. Cairon, J. P. Bellat, *J. Phys. Chem. C,* **2012***, 116,* 11195.