Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Supplementary Information

for

The effect of aluminum and platinum additives on hydrogen adsorption on mesoporous silicates

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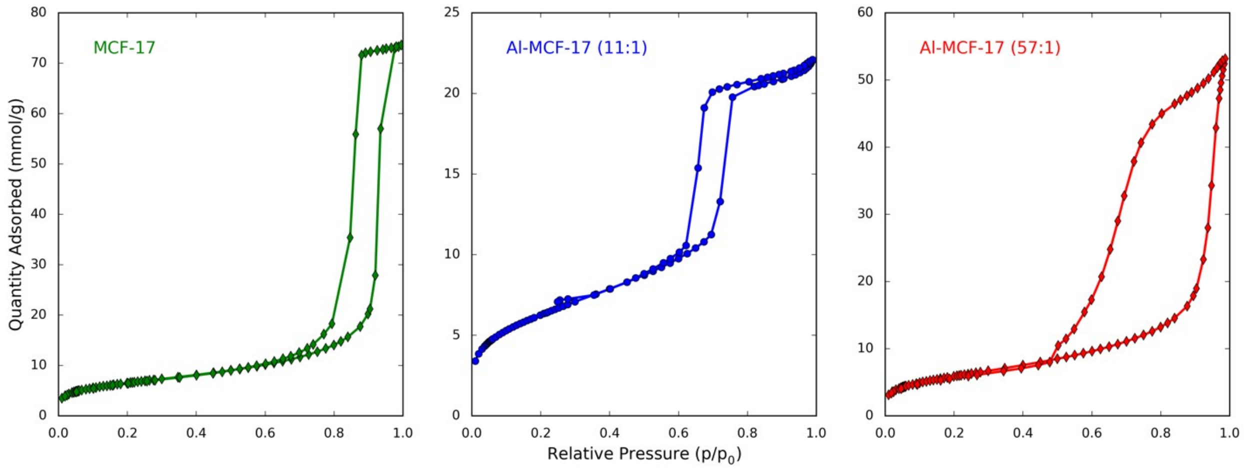

Figure S1: *Nitrogen adsorption isotherms for the different porous supports used in this study.*

Left: pure MCF-17; Middle: 11:1 aluminum-doped MCF-17; Right: 57:1 aluminum-doped MCF-17.

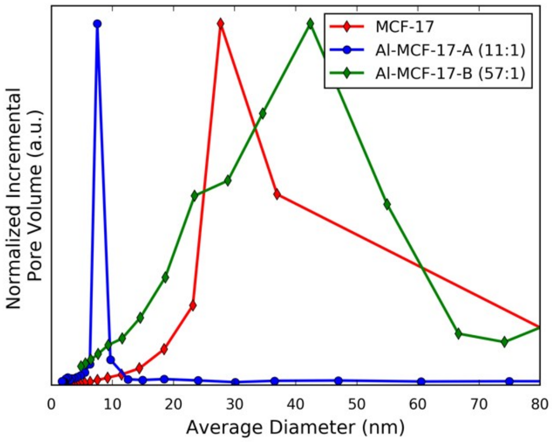

Figure S2: *Pore size distribution for the porous supports used in this study.*

***Table S1**: Porosity data for the supports used in this study.*

	MCF-17	Al-MCF-17 (11:1)	Al-MCF-17 (51:1)	Pt-MCF-17A	Pt-MCF-17B
BET (m^2/g)	622	498	322	571	582
Total Pore Volume (cm^3/g)	2.5	0.7	1.7	2.0	2.1
Micropore Volume (cm^3/g)	0.014	0.006	0.012	0.008	0.009
Average Pore Size (nm)	19	11	15	17	18

Total pore volume was obtained using the Barrett, Joyner, and Halenda (BJH) method,[1] whereas the micropore volume is derived from the t-plot extrapolation technique.

Synthesis of Pt nanoparticles

Platinum nanoparticles were synthesized via the polyol technique using polyvinylpyrrolidone (PVP) as a capping agent. For the synthesis of 1.5 nm Pt nanoparticles, H_2PtCl_6 and NaOH were dissolved in 10 mL ethyleneglycol to form two separate solutions. Then, the solutions were mixed and aged in a preheated oil bath at 160 °C for 3 hours under argon gas atmosphere. The resulting nanoparticles were precipitated using 2.0 M HCl and re-dispersed in a 0.1 mM solution of PVP in ethanol. For the synthesis of 7 nm Pt nanoparticles, PVP and H_2PtCl_6 were dissolved in 10 mL ethyleneglycol separately, and subsequently mixed together in a 50 mL round bottom flask at room temperature under constant stirring. The reaction mixture was then aged in the pre-heated oil bath at 180 °C for 30 minutes under a flow of argon gas. The final suspension was naturally cooled to room temperature, mixed with excess amount of hexane to help nanoparticles separate by centrifugation, and washed with a solution composed of ethanol and acetone (volume ratio is about 1:3) for 3 times. Finally, as-synthesized Pt nanoparticles were re-dispersed and kept in ethanol prior to utilization.

Loading of Pt nanoparticle into silica

For the preparation of MCF-17 supported Pt nanoparticles, colloidal solutions of Pt nanoparticles of given size are diluted in ethanol to the desired final weight loading amounts (final wt. % Pt) desired. That solution is added to the mesoporous oxides, and the suspension sonicated at room temperature for 3 hours (or until discoloration of the supernatant solution) using a commercial ultrasonic cleaner (Branson, 1510R-MT, 70 W, 42 kHz). The brown precipitates were separated by centrifugation (3000 rpm, 20 min), thoroughly washed with acetone (two times) and ethanol (two times), and dried in an oven at 80 °C overnight.

Characterization of surface acidity

The quantities of created Brønsted and Lewis sites was determined using pyridine adsorption detected by infrared spectroscopy, since the FTIR spectrum for pyridine varies with the type of binding site. Briefly, the samples were purged at 450 ˚C under helium for an hour in order to remove any trace of water that could have adsorbed upon storage of the material. The samples were then exposed to a flow of He/pyridine for several hours at room temperature to ensure pyridine saturation of all the surface acid sites contained in the material. Subsequently, the gas-phase pyridine was removed from the flow and the reactor flushed with pure helium. To eliminate interferences from physisorbed and weakly bound pyridine species, the samples were heated under helium at 150 ˚C. The samples were pressed to produce self-supporting wafers without a binding agent and loaded in the in-situ infrared cell. Finally, FTIR spectra were acquired by transmission at room temperature using an FTIR spectrometer (Nicolet Nexus-670) equipped with a Mercury-Cadmium-Telluride (MCT) detector (1500 scans at 4 cm^{-1} resolution). The extension coefficients used to estimate the acid density were taken from references 2 and 3.

References:

1 K.S.W. Sing, R.T. Williams, "Physisorption hysteresis loops and the characterization of nanoporous materials", Adsorption Science and Technology, 2004, **22**, 773–782.

2 M. Guisnet, P. Ayrault, C. Coutanceau, F. Alvarez, and J. Datka, "Acid properties of dealuminated beta zeolites studied by IR spectroscopy," *J. Chem. Soc. Faraday Trans.*, 1997, **93**, 1661–1665.

3 P. Ayrault, J. Datka, S. Laforge, D. Martin, and M. Guisnet, "Characterization of the Internal and External Acidity of H-MCM-22 Zeolites," *J. Phys. Chem. B*, 2004, **108**, 13755–13763.