Supplementary data: Adsorption isotherms

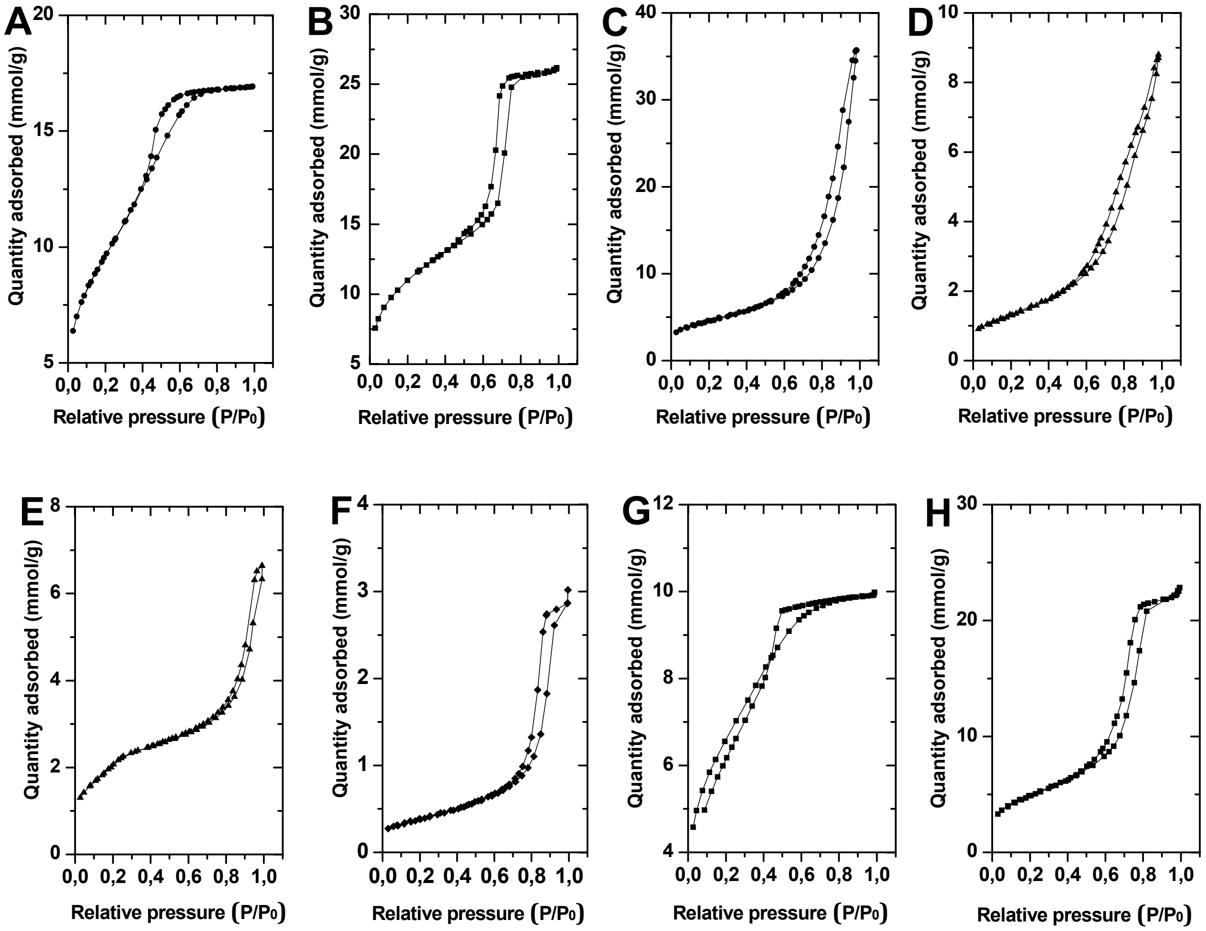

Figure 1. Nitrogen adsorption isotherms and pore size distributions derived from the adsorption branch. (A): SiO_2-A; (B) SiO_2-B; (C) Al_2O_3; (D) TiO_2; (E) Al_2O_3/SiO_2; (F) WO_3; (G) WO_3/SiO_2-3 and (H) WO_4SiO_2.

ICP-AES Analysis

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Method

0.1 g was melted with 0.375 g $LiBO_2$ and was dissolved in HNO_3. LOI (loss of ignition) was performed at 1000°C. Analysis has been performed according to EPA-method (modified) 200.7(ICP-AES) and 200.8 (ICP-QMS).

Result

ICP-AES analysis for W in the WO_4/SiO_2 material: 9880 mg/kg.

Characterization and analysis techniques

^{1}H-NMR was used for quantitative analysis of the products obtained after each run. The spectra were recorded at 400 MHz using a JEOL, model Eclipse FT-NMR Oxford instrument. The olefin bond in cyclohexene at 5.60 ppm was monitored and quantified using benzene as internal standard. The α and β methylene groups in adipic acid, which appear at 2.20 ppm and 1.50 ppm, respectively were used for quantification of the yield. The two α methine groups in cyclohexanediol at 3.25 ppm were used to determine the amount of this compound in the reaction mixture. The ^{1}H-NMR runs were made at 25°C using either $CDCl_3$ or DMSO-d_6 as solvent. The concentration of added standard (benzene) was controlled by weight with an accuracy of ± 1mg.

Determination of the specific surface area was performed on an ASAP 2010 instrument, using nitrogen adsorption and the Brunauer-Emmett-Teller (BET) method.[22] The pore size distribution was calculated from the isotherms using the Barret-Joyner-Halenda (BJH) procedure.[23] All samples were dried at 225°C in a vacuum oven for approximately 3 h before measurement.

Samples for the transmission electron microscopy (TEM), run on a JEOL 1200 EX II instrument at 120 kV, were prepared by placing a drop of an ethanol dispersion of the mesoporous material onto a copper Holey grid.

Scanning electron microscopy (SEM) was performed with a LEO Ultra 55 FEG equipped with an Oxford Inca x-sight EDX system, operated at 6.0 kV with WD = 3 mm. Specimen were prepared by placing the material onto a carbon tape with a silver glue.

Small angle X-ray scattering (SAXS) was performed with a Kratky compact small angle system on a HECUS Mbraun, Graz instrument. All runs were performed under vacuum at 50 kV and 40 mA. The runs were performed with monochromatic CuKα1 radiation using a Ni-filter. The samples were prepared by moulding the mesoporous oxide and then placing the particles in a paste holder with thin mica windows.

Low angle X-ray powder diffraction (XRD) was performed on a LynxEye AXS D8 ADVANCE θ/2θ diffractometer, linear detector. The runs were performed at 40 kV and 40 mA, in monochromatic mode with G(111) CuKα1 radiation (λ=1.5406 Å, step size 0.050, step time 366 s and primary slit width 0.2 mm).

X-ray photoelectron spectroscopy (XPS or ESCA) was performed on a Perkin-Elmer PHI 5000C spectrometer equipped with a pre-treatment reactor cell. The runs were performed under a base pressure in the analysis chamber of 1×10^{-8} Pa, in a monochromatic mode with MgKα radiation at 187.85 eV. A small amount of mesoporous material was placed onto a tape with adhesives on both sides. The information depth was approximately 4-5 nm. The surface composition is given as atomic percentage of the elements.