Supporting Information

Porous MoS₂ Synthesized by Ultrasonic Spray Pyrolysis

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USP Experimental Apparatus

S.I. Figure 1: The typical USP experimental apparatus.

A humidifier ultrasonically nebulizes the precursor solution. The aerosol droplets are then carried by a gas into a furnace where solvent evaporation and precursor decomposition occurs. Bubblers collect the desired product, and byproducts remain dissolved in the collection solvent or are flushed out of the system by the carrier gas.

Powder XRD

Powder patterns were obtained with a Rigaku D-Max diffractometer using Cu K α radiation; crystallite sizes were determined using the Jade x-ray analysis software package. Included in S.I. Figure 2 are powder diffraction patterns for conventional MoS₂ and three USP synthesized products.

S.I. Figure 2: Selected XRD patterns for conventional and USP MoS₂

XPS

XPS spectra were obtained with a Physical Electronics PHI 5400 X-ray Photoelectron Spectrometer using Mg K α radiation. Spectra were referenced to the graphitic carbon 1s peak at 284.5 eV. For 20 nm silica templated USP MoS₂, after leaching of the colloidal silica, the binding energies for Mo(3d_{5/2}) and Mo(3d_{3/2}) were 229.6 and 233.0 eV respectively, characteristic of Mo(IV); the binding energies for S(2p_{3/2}) and S(2p_{1/2}) were 162.7 and 163.9 eV respectively, characteristic of a S²⁻ environment. The spectra are shown in S.I. Figure 3 along with the corresponding spectra for conventional MoS₂.

S.I. Figure 3: XPS spectra of conventional and USP prepared MoS₂. Top figure, spin coupled S(2p_{3/2}, 2p_{1/2}) doublets. Bottom figure, spin coupled Mo(3d_{5/2}, 3d_{3/2}) doublet.

A shift to lower binding energy is observed for USP prepared MoS₂ compared to conventional MoS₂. A lower binding energy is characteristic of an overall lower molybdenum oxidation state. Electron-rich molybdenum centers have been found to be more intrinsically active for hydrodesulfurization.¹

Electron Micrographs

Electron micrographs at lower magnification are shown below in S. I. Figure 5. A high resolution electron micrograph, S. I. Figure 6, resolves the interlayer spacings characteristic of MoS₂, a layered material.

S.I. Figure 5: (A) TEM image of 20 nm silica templated USP MoS₂, after leaching of the colloidal silica. (B) SEM image of 80 nm silica templated USP MoS₂, after leaching of the colloidal silica.

S. I. Figure 6: High resolution TEM image of 20 nm silica templated USP MoS₂, after leaching of colloidal silica. Lattice spacings measured to be 0.42 ± 0.01 nm.

Additional References

- (1) (a) Ekman, M. E.; Anderegg, J. W.; Schrader, G. L. *J. Catal.* **1989**, *117*, 246-257. (b) Sullivan, D. L.; Ekerdt, J. G. *J. Catal.* **1997**, *172*, 64-75.