Synthesis and Characterization of Iron Molybdate Aerogels Synthesized by the Sol-Gel Chemistry and High Temperature Supercritical Drying

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In this work, iron molybdate ($\beta$-FeMoO$_4$) aerogels were prepared by the sol-gel synthesis of iron(III) and molybdenum(VI) organic precursors in methanol solution followed by a drying in an autoclave. The dried aerogels were characterized by means of scanning calorimetry/thermogravimetric analysis, X-ray diffraction, Mössbauer spectroscopy, electron scanning microscopy, mid-infrared adsorption, and nitrogen physisorption. The conventional drying to remove the solvent in a gel often collapses the porous network. One possible way to overcome this phenomena is to remove the solvent methanol by supercritical drying using temperatures of about 260 °C and pressures of about 100 bars. The X-ray results reveal that the iron molybdate powder obtained by the sol-gel and drying is converted completely into the ferric molybdate, Fe$_2$(MoO$_4$)$_3$, after heat-treatment at 500 °C in an air atmosphere. The aerogel network was determined to be composed of spherical primary particles with features in the 0.75-1 µm range as shown in Fig. 1.

Mössbauer results indicate high-spin Fe$^{2+}$ species to be present in the produced aerogels. The synthesized iron molybdate aerogels have mostly crystallized structure, whereas most of those aerogels produced by the sol-gel and supercritical drying are X-ray amorphous. A low temperature heat-treatment is used in the range 350-400 °C to remove the organic matrix. The potential of this iron molybdate aerogel as a catalyst/solid-state gas sensor is currently being investigated.