Metal Oxide Aerogel

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Aerogels are very attractive and promising materials owing to their high total pore volume, specific surface area and variable and adjustable porosity as well in many research areas. Nowadays, aerogel's main applications are based on their adsorption efficiency and low weight relative to their high porosity. Therefore, aerogels are mainly used as heavy metals adsorbents in waste water treatment technologies or additives into building materials. However, their application potential in catalysis/photocatalysis is markedly limited by structural properties of synthesized aerogels, namely amorphousness. Aerogels commonly prepared by sol-gel technique in combination with supercritical drying using supercritical carbon dioxide as extraction solvent show amorphous character. In this case the required crystallinity is reached only by subsequent thermal treatment.

Therefore, in this study the extraction technique opening up the possibility to prepare crystalline aerogels was developed and thoroughly investigated. Moreover, the first catalytic activity tests in AO7 azo-dye photooxidation over synthesized aerogels were performed. Pressurized fluid extraction using combination of subcritical water and super/subcritical methanol was applied to prepare a group of metal oxides - TiO_2, ZrO_2 and mixtures TiO_2-ZrO_2 and TiO_2-CeO_2. The effect of extraction temperature and pressure, as well volume of extraction solvent on textural properties, structural properties and purity of the final porous aerogels was studied. Rigid organogels used for extractions were prepared by the sol-gel process controlled in the reverse micellar environment of the nonionic surfactant Triton X-114 in cyclohexane at ambient temperature. The molar ratio of cyclohexane: Triton X-114: water: alkoxide was kept constant at 11: 1: 1: 1 for single oxide aerogels. In the case of mixed oxide aerogels the molar ratios of individual metal precursors were changed in the range from 0 to 1. Titanium (IV) isopropoxide, 70 wt-% zirconium (IV) propoxide in propan-1-ol and cerium (III) nitrate hexahydrate were used like metal precursors.

Nitrogen physisorption at -196°C, helium pycnometry, high-pressure mercury porosimetry, X-ray diffraction, Raman spectroscopy, organic elementary analysis and UV-Vis were used as characterization tools. Moreover, textural and structural properties of synthesized aerogels were compared with those of chemically identical but conventionally treated – calcined xerogels.

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