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Use of supercritical carbon dioxide for direct preparation of crystalline monolithic TiO₂ aerogels

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Titania aerogels are special highly-porous materials often used in the form of monoliths [1]. Their very high specific surface area makes them attractive candidates in many applications: solar energy conversion [2], photocatalysis [3], electronic devices [4], or as supporting material in the catalytic reactions [5]. The (photo)catalytic activity of monoliths critically depends on the structural properties such as crystal phase composition and crystallite size. Crystalline and pure TiO₂ aerogels are commonly prepared by calcination. However, this thermal treatment results in uncontrollable destruction of porous structure of titania aerogels, commonly accompanied by the decrease of the surface area as well as the porosity [6]. Therefore, gentler methods for the preparation of TiO₂ aerogels are desirable to optimize their morphology and functionality. Use of supercritical carbon dioxide (scCO₂) seems to be perspective way to obtain crystalline and pure TiO₂ monoliths without any subsequent thermal treatment [7, 8].

The present work deals with the crystallization of monolithic TiO₂ aerogels in environment of pure and water modified scCO₂. To remove organic precursors used during sol-gel synthesis and to crystallize TiO₂ the combination of several steps with pure and water-modified scCO₂ was tested with respect the high porosity and the specific surface area of titania blocks.

The effect of temperature (40-100 °C) and water concentration in scCO₂ (5-30 wt. %) on structural and textural properties was investigated at constant pressure of 30 MPa. The structural properties of the prepared monolithic TiO₂ were characterized by Raman spectroscopy. The textural properties such as specific surface area, content of mesoporous and volume of microporous were determined from nitrogen physisorption and skeletal density by helium pycnometry measurements.

The pure scCO₂ caused any crystallization. When 5 or 15 wt. % of water was added as a scCO₂ modifier a crystalline structure was obtained. The specific surface areas of mesoporous titania changed between 200-634 m²/g. Increasing the temperature and the concentration of water in scCO₂ resulted into the decrease of specific surface areas and change of the monoliths into the more fragile blocks.

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