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Influence of water-ethanol-modified supercritical carbon dioxide at the preparation of crystalline monolithic TiO₂ aerogels

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Praha 2

TiO₂ forms three crystalline phases (anatase, brookite, rutile), of which anatase is the most investigated because of its high photocatalytic activity. Due to its chemical stability, strong oxidation activity and non-toxicity, anatase is used especially for air- and water-purification [1]. Titania aerogels in the monolithic form has been extensively applied for the various applications such as solar energy conversion [2] and photocatalysis [3]. The use of TiO₂ for a given application depends not only on the phase composition, but also on specific surface area, crystallinity and crystallite-size. These properties can be significantly influenced by the preparation method used.

Crystalline and pure TiO₂ aerogels are commonly prepared by calcination accompanied by the uncontrollable porous structure destruction, decrease of surface area as well as porosity [4] and also the photocatalytic performance cannot be easily managed. As a perspective way to obtain crystalline and pure TiO₂ monoliths without any subsequent thermal treatment seems to be the using of supercritical carbon dioxide (scCO₂) [5, 6]. With this gentle method can be the structural, textural and physicochemical properties better controlled. In our previous studies [7-9], the efficiency of this method was demonstrated for TiO₂ in the forms of thin films and aerogels in which water and ethanol were used as modifiers. The critical effect of water on the TiO₂ crystallization and the beneficial influence of ethanol on the purity and phase composition of TiO₂ were found.

This work is focused on a study of TiO₂ aerogel SFC with scCO₂ modified by mixture of ethanol and water in a ratio 1:1. The effect of temperature (40-100 °C), pressure (10-30 MPa) and modifier concentration in scCO₂ (5-15 wt.%) and the additional drying with pure CO₂ on microstructure, purity and textural properties of TiO₂ aerogel was investigated. Processed monoliths were characterized by XRD analysis, Raman spectroscopy and N₂ physisorption.

Increasing temperature, pressure or modifier concentration in scCO₂ showed the positive effect on the aerogel purity. However, the temperature of 100 °C or the presence of too large amount of mixture (i.e. 15 wt.%) resulted into the aerogel destruction. The specific surface areas of mesoporous titania changed between 222-498 m²/g. Increasing the pressure and temperature resulted into the decrease of monolith specific surface areas, change of the monoliths colour from white to yellow and into the more fragile blocks.

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