Use of Water-Modified Supercritical Carbon Dioxide for Direct Preparation of CrystallineMonolithic TiO2 Aerogels.

Cerhová, Marie
2017

Use of Water-Modified Supercritical Carbon Dioxide for Direct Preparation of Crystalline Monolithic TiO_2 Aerogels

Student: Ing. Marie Cerhová
Supervisor: Ing. Marie Sajfrtová, Ph.D.
Supervising Expert: Ing. Lenka Matějová, Ph. D.

TiO_2 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 have been extensively applied for the various applications such as solar energy conversion[2] and photocatalysis.[3] The use of TiO_2 for a given application depends not only on its 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 TiO_2 aerogels are commonly prepared by calcination accompanied by an uncontrollable porous structure destruction, decrease of surface area, as well as porosity.[4] Also the photocatalytic performance cannot be easily managed. Using supercritical carbon dioxide ($scCO_2$) seems to be a promising way to obtain crystalline and pure TiO_2 monoliths without any subsequent thermal treatment.[5,6] With this gentle method the structural, textural and physicochemical properties can be better controlled.

We studied a combination of supercritical CO_2 and water for the preparation of crystalline and pure nanostructured TiO_2 without any thermal processing. The effect of temperature (40–100 °C) and pressure (10–30 MPa) on the purity, structural and textural properties was investigated. The prepared aerogels were characterized with respect to the (micro)structural properties by Raman spectroscopy and X-ray diffraction. 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 pressure of 10 MPa did not cause any crystallization. When the pressure increased, crystallization to anatase and brookite occurred. Increasing the temperature resulted into a decrease of specific surface

areas, change of the monoliths' colour from white to yellow, and into the more fragile blocks.

References

1. Sökmen, M.; Özkan, A. *J. Photochem. and Photo. A: Chemistry* **2002**, *147*, 77–81.
2. Li, X.; Wang, H.; Chen, J.; Zhou, Q.; Xiano, Z. *Electrochim. Acta.***2014**, *145*, 281–285.
3. Chong, M. N.; Jin, B.; Chow, Ch. W. K.; Saint, Ch. *Water Res.* **2010**, *44*, 2997–3027.
4. Pourmand, M.; Mohammadizadeh, M. R. *Current Nanoscience.* **2008**, *4*, 151–156.
5. Shimoyama, Y.; Ogata, Y.; Ishibashi, R.; Iwai,Y. *Chem. Eng. Res. Des.***2010**, *88*, 1427–1431.
6. Matejova, L.; Matej, Z.; Fajgar, R.; Cajthaml, T.; Solcova, O. *Mater. Res. Bull.***2012**, *47*, 3573–3579.