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The effect of experimental design on crystal size of TiO₂ thin films prepared by using modified supercritical carbon dioxide

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TiO₂ is known as an efficient photocatalyst for its good ability to produce the pairs electron-hole under UV light [1]. Moreover, titania exhibits high chemical stability, strong oxidative activity and non-toxicity. It has been investigated in a form of thin films in many application areas; as sensing films of gas sensors [2], coatings for self-cleaning surfaces [3], photocatalyst promising in water- and air-purification [4] or degradation of organic pollutants [5].

Properties as phase composition, crystallinity or crystallite-size influencing the photocatalytic activity and are size dependent. That means the controlled preparation of nanocrystallites is important for their applications. Calcination is a commonly used method for the purification and to crystallize TiO₂ thin films [6]. Unfortunately, this thermal treatment is accompanied by excessive sinteration, crystallite growth or recrystallization. To reduce the process temperature and to increase the thermal stability and the photocatalytic activity the TiO₂ thin films can be also extracted by scCO₂, subcritical water or pressurized organic solvents [7].

In this work, the method using pure and modified supercritical carbon dioxide was tested to prepare a set of crystalline TiO₂ thin films deposited on soda-lime glass. Precursor solution for TiO₂ films was prepared by a templated sol-gel method in the reverse micelle environment. Simple approach or combination of several steps performed at different temperature (40-150 °C) and solvent composition was applied at pressure 30 MPa. Water, ethanol and water : ethanol (1:1) mixture were added into supercritical CO₂ as modifiers at concentration 10-30 wt.%. The effect of extraction design on microstructure and purity of TiO₂ thin films were examined.

The thin films were characterized with respect to the (micro)structural properties by Raman spectroscopy. The most promising thin films samples were analyzed by means of X-ray diffraction to determine the crystallite size and the phase composition.

TiO₂ thin films treated by water modified scCO₂ exhibited the crystalline structure anatase. Subsequent modification of scCO₂ by 10 wt. % ethanol led to the removal of the organic precursor used in the sol-gel method and thus to increase the purity of TiO₂ thin films. The additional drying with 200 g of pure scCO₂ had no positive effect on crystal growth but the mixture of anatase and brookite crystals was formed. At the optimal conditions of SFC by water : ethanol (1:1) mixture modified scCO₂ (30 MPa, 150 °C, 100 g of scCO₂) the anatase crystals with the size of 13 nm were obtained.

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[1] Prieto O. et al. *Sol. Energy*. **2005**, *79*, 376-383. [2] Savage N. O. et al. *Sens. Act. B*. **2001**, *72*, 239-248. [3] Fujishima A. et al. *Electrochim. Acta*. **2000**, *45*, 4683–4690. [4] Hsien Y.-H. et al. *Appl. Catal. B: Environ.* **2001**, *31*, 241–249. [5] Nakata K. et al. *Electrochim. Acta*. **2012**, *84*, 103. [6] Zhou M. et al. *J. Hazard. Mat.* **2008**, *154*, 1141–1148. [7] Matejova L. et al., EMSF 2014, Marseille, France, p.161 (Full text on Flash disc).