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Titanium dioxide in the form of aerogels belongs to the most desirable materials due to its crystalline anatase structure exhibiting high photocatalytic activity. TiO_2 is used in the environmental applications such as air or water purification.[1]

Crystalline and pure TiO_2 aerogels are prepared by the calcination, which is accompanied by uncontrollable destruction of the porous structure or a decrease in specific surface and porosity.[2] However, the thermal treatment has recently been successfully replaced by gentle low-thermal supercritical fluid crystallization (SFC) with modified supercritical carbon dioxide ($scCO_2$).[3] In our previous studies,[3–5] the efficiency of this method was demonstrated for TiO_2 in the forms of thin films and aerogels in which water and ethanol were used as modifiers. The critical effect of water on the TiO_2 crystallization and the beneficial influence of ethanol on the purity and phase composition of TiO_2 were found.

Therefore, this work is focused on a deeper study of TiO_2 aerogel SFC with $scCO_2$ 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 $scCO_2$ (5–15 wt.%) on microstructure, purity and textural properties of TiO_2 aerogel was investigated. Processed monoliths were characterized by XRD analysis, Raman spectroscopy and N_2 physisorption.

Increasing temperature, pressure or modifier concentration in $scCO_2$ 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. Therefore, an optimal temperature and amount of mixture in $scCO_2$ in terms of the aerogel crystalline structure presence and sufficient purity was determined to be 80 °C and 10 wt.%. The increasing pressure led to the undesirable decrease of monolith specific surface area.

References

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