Preparation and phase transformation of titanium dioxide in supercritical carbon dioxide

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Nano-sized titania has received much attention because of its unique physico-chemical properties for the applications of pigments, cosmetics, fine ceramics, and photocatalysts. In this work new method has been tried to synthesize TiO2 nano-particles which possess desired crystalline structure, e.g. anatase phase, without any heat treatment utilizing supercritical carbon dioxide.

The experimental results show that the structure of TiO2 has been changed from amorphous to anatase phase through the treatment in supercritical CO2 by applying small amount of acid. Amorphous TiO2 obtained from hydrolysis of titanium isopropoxide has been acidified with HCl or HNO3 and transferred into supercritical carbon dioxide media to carry out the crystallization at 600C and 208bar for 12 hours. The polymorphs greatly depend upon the amount of water as well as the concentration of acid in supercritical media. The SEM pictures of the products show that the sizes of particles are about $5\sim10$ nm, and the XRD patterns of TiO2 powders reveal that the produced particles are anatase phase when 5wt% water and $0.06\sim0.2wt\%$ acid have been included in supercritical CO2.