

Synthesis and characterization of nanoparticle of TiO_2 co-doped with Sc^{3+} and V^{5+} ions

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Abstract

Nanoparticle of TiO_2 co-doped with Sc^{3+} and V^{5+} ions was synthesized by the sol–gel method and calcinated at 500 °C. Particle size of $\text{TiO}_2 + 2.0$ at.% (Sc + V) calculated from XRD spectrum is 16.2 nm. The size and shape of nanoparticles synthesized were also obtained by transmission electron microscopy (TEM) and mean diameter of particle was determined as 17.4 ± 5.4 nm, which is in agreement with that of calculated from XRD spectrum. Image from high-resolution transmission electron microscopy (HRTEM) study on the sample exhibited well-defined lattice fringe. Elemental analysis was conducted by energy-dispersive X-ray microanalysis (EDX) and confirmed the presence of Sc^{3+} and V^{5+} ions in the TiO_2 matrix.

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1. Introduction

TiO_2 has been intensively investigated for many years because of its applications in many fields, such as photocatalysis [1,2], sensors [3,4], solar cells [5], and memory devices [6]. TiO_2 has three different polymorphs: rutile, anatase, and brookite. The bulk and surface properties of rutile have received the most investigation due largely to the availability of large bulk rutile single crystals. Although anatase TiO_2 exhibits superior photocatalytic properties as well as a number of interesting behaviors [7–9], experimental investigations on anatase single-crystal surfaces have been very limited and existing results remain controversial.

The presence of metal ion dopants in the TiO_2 crystalline matrix significantly influence its properties. Choi et al. [1] performed a systematic study of metal ion dop-

ing in quantum (Q)-sized (2–4 nm) TiO_2 colloids by measuring their photoreactivities and the transient charge carrier recombination dynamics, and found that doping with Fe^{3+} , Mo^{5+} , Ru^{3+} , Os^{3+} , Re^{5+} , V^{4+} , and Rh^{3+} at 0.1–0.5 at.% significantly increases the photoreactivity for both oxidation and reduction while Co^{3+} and Al^{3+} doping decreases the photoreactivity. Martin et al. [10] studied photochemical mechanism of size-quantized vanadium-doped TiO_2 particles. They found that sintering at higher temperatures (200–400 °C) results in the formation of islands of V_2O_5 on TiO_2 while sintering at 600 and 800 °C produces non-stoichiometric solid solutions of $\text{V}_x\text{Ti}_{1-x}\text{O}_2$ and doping (1 at.%) of the TiO_2 crystals with vanadium reduces the photooxidation rates of 4-chlorophenol compared to the undoped aggregates. Zhang et al. [11] reported role of particle size in nanocrystalline TiO_2 -based photocatalysts and showed that particle size is a crucial factor in the dynamics of the electron/hole recombination process and optimal Fe^{3+} dopant concentrations for TiO_2 of different average

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particle sizes was presented. Yang et al. [12] prepared nanoparticles of titanium dioxide co-doped with Fe^{3+} and Eu^{3+} by using the sol–gel method and demonstrated that optimal concentration for nanocrystalline TiO_2 co-doped with Eu^{3+} and Fe^{3+} ions are 1 at.% Fe^{3+} and 0.5 at.% Eu^{3+} , respectively, which significantly increases the photodegradation activity of nanosized TiO_2 .

In the present study, we synthesized nanoparticle of TiO_2 co-doped with Sc^{3+} and V^{5+} ions by the sol–gel method and calcinated at 500 °C. For the first step, the aim of present study is to observe effect of Sc^{3+} and V^{5+} ions in the formation of nanocrystalline of TiO_2 . The product synthesized was characterized by XRD, HRTEM, SAED, and EDX spectroscopic techniques. To our best knowledge, it is the first time doping with scandium element.

2. Experimental

All chemicals were obtained from Aldrich Chemical Co. and used as received. In a typical synthesis of TiO_2 nanoparticle, we use titanium tetrachloride (TiCl_4 , 99.9%), scandium oxide (Sc_2O_3 , 99.9%), and vanadium oxide (V_2O_5 , 99.6%) as starting materials. 38 mg (0.28 mmol) of scandium oxide and 50 mg (0.28 mmol) of vanadium oxide were simultaneously added to 200 mL of deionized water under heavy stirring at room temperature, then 3 mL (27 mmol) of titanium tetrachloride was dropwise added to the solution. Hydrolysis of TiCl_4 occurs immediately, as indicated by the appearance of turbidity. After stirring for another 2 h, 7.3 mL of ammonia solution (28%) was dropwise added to the solution to complete formation of TiO_2 . During the period of stirring, Sc_2O_3 and V_2O_5 were completely dissolved by the hydrochloride produced by hydrolysis of TiCl_4 , as a result, the color of the solution changed from initially yellowish, due to vanadium oxide, to white. Afterwards, the doped TiO_2 nanoparticle was gathered and washed 3 times with deionized water by centrifugation to remove the hydrochloride and ammonia residual in the product. The final product was dried in a oven at 60 °C for further characterization. The product synthesized was almost amorphous phase, and calcined at 500 °C for 6 h for the better crystallinity.

The crystal structure of synthesized nanoparticles was identified by X-ray powder diffraction (XRD) with a Philips X'Pert-MPD System with a $\text{CuK}\alpha$ radiation source ($\lambda = 0.154056$ nm). The size and shape of nanoparticles were obtained by transmission electron microscopy (TEM). TEM measurements were carried out on a HITACHI H-7500 low resolution TEM. Sample for TEM was prepared on 300 mesh copper grid coated with carbon. A drop of the nanoparticle solution was carefully placed on the copper grid surface and dried. The size distribution of the particles was measured from en-

larged photograph of the TEM image. High resolution transmission electron microscopy (HRTEM), selected-area electron diffraction (SAED), and energy-dispersive X-ray (EDX) spectroscopy for the elemental analysis of doped TiO_2 were conducted on a JEOL JEM2010 TEM operated under an acceleration voltage of 200 kV.

3. Results and discussion

Generally, XRD can be used to characterize the crystallinity of nanoparticle. Fig. 1 shows the XRD pattern of $\text{TiO}_2 + 2.0$ at.% (Sc + V) calcined at 500 °C. The discernible peaks in Fig. 1 can be indexed to (101), (103), (004), (200), (105), (211), (204), (116), (220) and (215) planes of a tetragonal unit cell, which correspond to those of titanium dioxide structure (JCPDS card no. 83-2343). The product, which was crystallized by calcination at 500 °C, was determined to be a pure anatase phase. Average particle size calculated from the broadening of the (101) XRD peak of anatase phase was about 16.2 nm. Zhang et al. [11] reported 21 nm of TiO_2 synthesized by hydrothermal treatment route (450 °C for 2 h). Reddy et al. [13] synthesized CeO_2 – TiO_2 , V_2O_5 /CeO₂– TiO_2 mixture oxides by hydrolysis of $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$ and TiCl_4 with aqueous ammonia solution and their crystallite sizes after calcined at 500 °C for 5 h were 10.0 and 12.9 nm, respectively. For the sake of low dopant concentration (2.0 at.%) for both Sc^{3+} and V^{5+} ions, we did not observe any secondary phase.

It has been well known that anatase phase of TiO_2 , which is less stable than rutile phase thermally and transfer into rutile phase at higher temperature, has an important role in photocatalysis and sensor. It was reported [14] that anatase phase of TiO_2 shows higher efficiency than rutile phase in decomposition of organic compounds under illumination. TiO_2 as a gas sensing

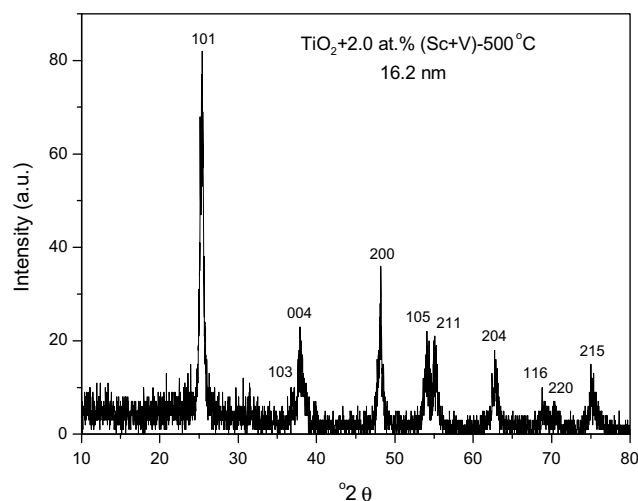


Fig. 1. XRD pattern of $\text{TiO}_2 + 2.0$ at.% (Sc + V) calcined at 500 °C.

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