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Gel–sol synthesis and aging effect on highly crystalline anatase nanopowder

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Abstract. Highly crystalline TiO_2 anatase nanoparticles were synthesized via gel–sol method by using titanium isopropoxide and triethanolamine. The products were characterized by X-ray diffraction, transmission electron microscopy, thermogravimetric/differential thermal analysis and nitrogen gas absorption methods. The particle size ranged from 7 to 24 nm having specific surface area of 64 to 220 m^2/g. Selective $Ti(OH)_4$ gel specifications and hydrothermal test conditions resulted in thermodynamically-stable phase-formation. Aging at 130°C for 4 h resulted in particle size of 7 nm; while at 130 and 160°C for 12 h resulted in 12 and 21 nm, respectively.

Keywords. Gel–sol; anatase; titania; nanoparticles; aging.

1. Introduction

Recently, utilization of TiO_2 nanocrystals in optical, electrical, photocatalytic and pigment has received considerable attention. Superior physicochemical behaviour, relatively low-cost and easy handling are some of their advantages. TiO_2 properties are strongly related to phase structure, morphology and particle size distribution (Hoffmann *et al* 1995; Fujishima *et al* 2000; Diebold 2003; Chen and Mao 2007). TiO_2 photocatalytic performance is especially critically affected by its particle size. The monodisperse TiO_2 nanoparticles of TiO_2 also exhibit photocatalytic effect (Almquist and Biswas 2002).

Titanium dioxide has three principal crystallographic structures called anatase (tetragonal), rutile (tetragonal) and brookite (orthorhombic). The most stable phase is rutile; whereas anatase is a metastable phase at ambient temperature. The photo-activity of the latter is at the same time generally superior to that of the former. High temperature photocatalysis applications of TiO_2 nanoparticles demand, therefore, anatase thermal stability not to convert into the rutile phase (Hoffmann *et al* 1995; Cappelletti *et al* 2008).

Sol–gel is a widely used technique for production of titanium dioxide nanopowders of amorphous structure. For improving crystallinity, post-calcination is generally required. High-temperature treatment results in size-increment and agglomeration (Kim *et al* 1999; Song and Pratsinis 2001; Sivakumar *et al* 2002; Tang *et al* 2002; Phonthammachai *et al* 2003; Chen and Mao 2007). Hydrothermal processing is another method practised by many researchers as a common simple procedure capable of producing inexpensive photocatalytic pure nanopowder (Yanqing *et al* 2000; Byrappa and Adschiri 2007).

Gel–sol method is a newly developed technique used for production of large quantities of nanoparticles (Sugimoto *et al* 1998; Itoh and Sugimoto 2003). Sugimoto *et al* (1998) spent 4 days conducting experiments to produce TiO_2 nanoparticles of suitable specifications. A highly viscous gel (like metal hydroxide gel) donates the network required for nuclei containment, product protection from coagulation and agglomeration, prevention of particle growth and protection of metal and/or hydroxide ions from precursor monomers release even at highly concentrated conditions. The nanoparticles obtained have high monodispersity, geometric consistency and diversity of shape (Sugimoto and Sakata 1992; Hosokawa *et al* 2007). On completion of nanoparticles formation, the gel-phase gradually disappears while leaving a smooth homogeneous sol-phase consisting of uniform nanoparticles.

In this paper, highly-crystalline pure anatase nanoparticles synthesized by the newly developed gel–sol method is illustrated. Substantial reduction in the synthesis time together with a narrow size distribution is obtained. The whole process takes only one day. Effect of first and second aging treatments on nanoparticles size and crystal structure is investigated. Products are characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD), thermogravimetric/differential thermal analysis (TG–DTA) and nitrogen gas absorption (BET).

2. Experimental

2.1 *Preparation of titanium dioxide nanoparticles*

Triethanolamine (TEA) and titanium (IV) isopropoxide (TTIP) (both of Merck Co., Germany) were mixed together

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