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Synthesis of Nanosized Titania by sol Gel Route

KEYWORDS: Titania; Sol gel; Ethylene glycol; Anatase

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ABSTRACT *The main objective of this work was to prepare anti-pollutant and photocatalyst Titanium dioxide nanoparticles by simple ethylene glycol route to synthesize titanium dioxide nanoparticles at industrial level. In this work TiO_2 was prepared by sol-gel route in presence of titanium n-butoxide (TNBT) as TiO_2 precursor, n-butanol as dilutant and EG as solvent and chelating agent. The X-ray diffraction and scanning electron microscopy studies show that the product has anatase crystal structure with average particle size 20-50 nm. The nanoparticles thus prepared can be used for gas sensing and biological applications, also as photo-electrodes for dye-sensitized solar cells and in removing the organic chemicals which occur as pollutants in wastewater effluents from industrial and domestic sources.*

Introduction

Nanosized titania has been the subject of a great deal of research because of their unique physicochemical properties and applications in the areas of pigments, catalysts and supports, cosmetics, gas sensors, inorganic membranes, environmental purification, and dielectric materials [1-9]. Much interest has been shown in photochemical reactions on nanosized titania particles due to their potential application in the conversion of solar energy into chemical energy [10-13] and electric energy [14, 15]. When titania powder is irradiated with photon energy larger than the band-gap energy, electrons (e^-) and holes (h^+) are generated in the conduction band and the valence band, respectively. These electrons and holes are thought to have the respective abilities to reduce and oxidize chemical species adsorbed on the surfaces of titania particles [16]. The uses and performance for a given application are, however, strongly influenced by the crystalline structure, the morphology, and the size of the particles. It is well known that titania exists in three kinds of crystal structures namely anatase, rutile and brookite. Anatase and brookite phases are thermodynamically metastable and can be transformed exothermally and irreversibly to the rutile phase at higher temperatures. The transition temperatures reported in the literature ranges from 450 to 1200 ^{0}C. The transformation temperature depends on the nature and structure of the precursor and the preparation conditions [17, 18]. Among the three kinds of crystal structures of Titania, anatase TiO^2 has been widely used as a well known catalyst, because of its various merits, such as electronic and optical properties, non-toxicity, high photocatalytic activity, low cost, and chemical stability [19-23]. A number of methods for the synthesis of TiO^2 nanoparticle have been reported, such as chemical precipitation [24], microemulsion [25], hydrothermal crystallization [26] and sol-gel [27]. The sol-gel process is the most successful for preparing nanosized metal oxide semiconductors. For example, sol-gel derived TiO^2 powders have been reported to show high catalytic activity due to their fine structure, wide surface area and high porosity. Thus in this research work we have prepared titania by sol gel route using ethylene glycol as gelling agent and titanium n-butoxide as titania precursor.

Experimental Work

Titanium (IV)-n-butoxide (TNBT) (20 g) was added to n-butanol (16 g) and the mixture was stirred for 5 min using a magnetic stirrer operating at 2000 rpm. After stirring, above mixture was added to Ethylene glycol (100 ml) and mixture was stirred with heating at 85 ^{0}C till sol converted to gel then gel was dried in oven at 50 ^{0}C. Dried sample was calcined at 500 ^{0}C for 3 h.

Results and discussion

The XRD pattern and SEM image of nanosized Titanium Oxide particles, prepared by EG route is shown in figure 1 and 2.

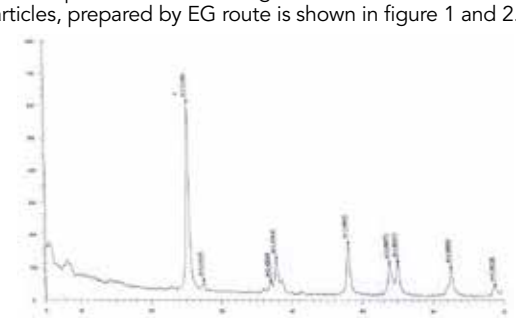

Figure 1. XRD patterns of nanosized titania obtained from sol gel route at 500 ^{0}C

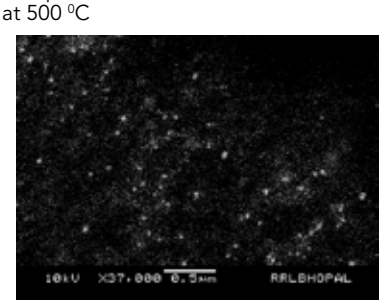

Figure 2. SEM photograph of nanosized titania obtained from sol gel route at 500 ^{0}C

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The XRD patterns of the calcined sample of nanosized titania powder prepared using sol gel route is given in Figure 1. The presence of peaks of anatase titania at'd' values 3.49, 1.88, 2.35 have been observed in XRD pattern. The d-values of the pattern recorded were compared and matched with the standard d-values along with the intensity given in JCPDS manuals. The average crystalline size of nanosized titania has been calculated by applying Scherrer's equation ($d = k \lambda / \beta \cos \theta$) to the 100 % intensity peak and is found 19 nm. The prepared nanosized powder of TiO^2 was fired at 500 ^{0}C, leads to its conversion from amorphous phase to anatase phase.

The crystal size of the powder sample of TiO^2 is of 19 nm size as estimated by Scherrer equation. SEM photograph shows that particles are strongly aggregated which is typical for particles with a size less than 50 nm, so the particle size was estimated to be 20-50 nm.

Conclusion

We synthesized TiO^2 nanoparticles by sol gel route using ethylene glycol as gelling agent. The physical properties, such as crystallite size and crystallinity were investigated by XRD, and SEM. X-ray diffraction pattern shows that TiO^2 particles calcined at 500°C have a stable anatase phase with 19 nm average crystallite size, determined according to the Scherrer equation. The SEM image shows that particles have spherical shape. The yield of the prepared photocatalyst was comparatively higher than other methods used for nanoparticles preparation i.e. microemulsion route.

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