

Synthesis and Characterization of SnO₂ Nanoparticles by Co-Precipitation Method

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Abstract

Tin dioxide (SnO₂) nanoparticles were synthesized via a simple co-precipitation method using stannous chloride dihydrate (SnCl₂·2H₂O) as the precursor and ammonia as the precipitating agent, followed by the addition of activated charcoal and calcination at 600 °C. The morphological properties of the obtained nanoparticles were investigated by scanning electron microscopy (SEM). The SEM analysis revealed spherical grain morphology with an average particle size of approximately 21 nm and high crystallinity. The method employs low-cost starting materials and straightforward procedures, making it suitable for the preparation of nanostructured SnO₂ with potential applications in gas sensing and catalysis.

Keywords: SnO₂ nanoparticles; co-precipitation; scanning electron microscopy; nanocrystalline tin oxide

Introduction

Among the technical applications of tin dioxide (SnO₂), the most important uses are as gas sensors, bulk ceramics, glazes, and pigments. The majority of chemical and physical properties of SnO₂ materials depend on structural parameters. In particular, the sensing properties of SnO₂ sensors (sensitivity, selectivity, and reproducibility) critically depend on particle size and specific surface area. Preparation of metal-oxide nanoparticles at low cost on an industrial scale remains a challenge. Therefore, the use of inexpensive materials, simple fabrication processes, and optimized synthesis conditions is essential. The influence of parameters such as initial solution composition, reaction time, and heat-treatment temperature is of great importance for nanopowder production [1-5].

Many methods exist for the preparation of nanoparticles. One important route is the sol-gel method, which offers advantages such as a simple procedure and lower processing temperature. Because of its unique electrical and catalytic properties, SnO₂ has been widely used in electrochemical and catalytic applications, including gas sensors for environmental monitoring. For these applications, small particle size or large specific surface area is essential for high performance. The hydrothermal method is an alternative to calcination for the crystallization of SnO₂ at mild temperatures and is regarded as one of the best methods for producing fine oxide powders. In hydrothermal synthesis, water acts as a catalyst and as a component of solid phases at elevated temperature and pressure; however, the requirement for an

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autoclave is a disadvantage.

Solvothermal synthesis is well suited for the production of nanostructured materials because of its relatively low processing cost, milder conditions, and ability to control grain size. In the present work, SnO₂ nanoparticles were synthesized by a co-precipitation route using cheap starting materials and simple procedures. The obtained particles are in the nanometer size range. This approach represents a practical alternative to sol-gel synthesis from tin alkoxides.

Various Methods for Synthesis of Nanoparticles

Modern materials science is characterized by a close interplay between physics and chemistry. This is especially true for nanomaterials. On the one hand are the top-down methods, which rely on continuous breakup of a piece of bulk matter, while on the other are the bottom-up methods that build up nanomaterials from their constituent atoms. The top-down and bottom-up approaches can also be considered to be physical and chemical methods, respectively [6-10].

Physical methods

Most physical methods involve evaporation of a solid material to form a supersaturated vapor from which homogeneous nucleation of nanoparticles occurs. In these methods, the size of the particles is controlled by temporarily inactivating the source of evaporation or by introducing gas molecules to collide with the particles. Some of the methods used to prepare nanocrystals are arc discharge, ion sputtering, laser ablation, and spray pyrolysis.

Chemical methods

Chemical methods have emerged as indispensable for synthesizing nanocrystals of various types of materials. These methods are generally carried out under mild conditions and are relatively straightforward. Any chemical reaction resulting in a sol consists of three steps: seeding, particle growth, and growth termination by capping. An important process that occurs during the growth of a colloid is Ostwald ripening. Ostwald ripening is a growth mechanism whereby smaller particles dissolve, releasing monomers or ions for consumption by larger particles, the driving force being the lower solubility of large particles. Ostwald ripening limits the ultimate size distribution obtainable to about 15% of the particle diameter when the growth occurs under equilibrium conditions.

(a) Metal nanocrystals by reduction

Metal nanocrystals can be obtained by reducing the corresponding soluble metal salts and terminating the growth with appropriate surfactants or ions. A variety of reducing agents are used to reduce metal salts to obtain nanocrystals of metals. Some of them are:

- (i) Borohydride reduction,
- (ii) Citrate reduction,
- (iii) Alcohol reduction,
- (iv) Reduction using alkylaluminates.

In the 17th century, Faraday prepared gold nanoparticles in the laboratory by reducing a gold salt. He termed them “divided metal,” which gave rise to a brilliant rose color.

(b) Solvothermal synthesis

The solvothermal method provides a means of using solvents at temperatures well above their boiling points by carrying out the reaction in a sealed vessel. The pressure generated in the vessel due to the solvent vapors elevates the boiling point of the solvent. Typically, solvothermal methods make use of solvents such as ethanol, toluene, and water. These are widely used to synthesize zeolites, inorganic open-framework structures, and other solid materials. In the past few years, solvothermal synthesis has emerged as the chosen method to synthesize nanocrystals. Solvothermal methods are ideally suited for the synthesis of nanocrystals of metal chalcogenides and halides rather than metal nanocrystals.

(c) Photochemical synthesis

Photochemical synthesis of nanoparticles can be carried out by the light-induced decomposition of a metal complex or the reduction of metal salts by photogenerated reducing agents such as solvated electrons. The former is called photolysis and the latter radiolysis. The formation of photographic images on AgBr film is a familiar photolysis reaction. Metals such as Au, Cd, and Ti have been obtained by photolysis.

(d) Arrested precipitation

Nanocrystals can be obtained from solutions that precipitate the bulk matter under conditions unfavorable for the growth of particulates in the precipitate. For example, the precipitation of metal salts by chalcogens can be arrested by employing a high pH. To prepare nanocrystals of CdS, CdSe, CdTe, HgSe, HgTe, and CdHgTe, typically a solution containing the metal salt and the capping agent is treated with NaOH to raise the pH, degassed by bubbling inert gas, followed by the introduction of the chalcogen in the form of Na₂S, NaHSe, etc., under inert conditions.

(e) The liquid–liquid interface

Nanocrystals and films of metals, semiconductors, and oxides can be produced by reactions taking place at the interface of two liquids such as toluene and water. In this method, a suitable organic derivative of the metal taken in the organic layer reacts at the interface with the appropriate reagent present in the aqueous layer to yield the desired product. For example, by reacting Au(PPh₃)Cl in toluene with tetrakis(hydroxymethyl)phosphonium chloride (THPC) in water, nanocrystals of Au can be obtained at the interface of the two liquids.

Experimental**a. Materials**

All chemicals used were of analytical reagent (AR) grade and were used as received without further purification. Stannous chloride dihydrate (SnCl₂·2H₂O), ammonia (NH₃), activated charcoal (carbon black powder), and deionized water were employed.

b. Synthesis of SnO₂ Nanoparticles

Nanoparticles of SnO₂ were prepared by the co-precipitation method using SnCl₂ as the precursor, followed by heat treatment at 600 °C. A 0.1 M solution of stannous chloride dihydrate was prepared by dissolving 2.25 g of SnCl₂·2H₂O in 100 mL of distilled water. After complete dissolution, the solution was allowed to stand for 5 min. Then 4 mL of ammonia was added under constant stirring, and stirring was continued for 20 min using a magnetic stirrer. A white gel precipitate formed immediately upon addition of ammonia. The mixture was allowed to settle for 12 h and then filtered using Whatman filter paper. The filtered gel was washed 2–3 times with distilled water to remove ammonium

ions completely. The obtained gel was mixed with

0.27 g of activated charcoal powder, resulting in a gray-colored precipitate. The mixture was dried at 70 °C for 24 h, crushed into powder using a mortar and pestle, and finally calcined at 600 °C for 4 h in an oven. After calcination, white-colored SnO₂ nanoparticles were obtained. The particle size was confirmed by X-ray diffraction (XRD) and scanning electron microscopy (SEM).

Characterization Techniques

Scanning electron microscopy (SEM) is widely used for the characterization of nanomaterials and nanostructures. Its resolution approaches a few nanometers, and magnification can be adjusted from approximately 10× to 300 000×. SEM provides both topographical information and chemical composition near the sample surface. A high-energy electron beam from an electron gun is focused onto the specimen surface, generating signals that yield information on chemical composition, crystalline structure, and external morphology (texture).

SEM images were obtained on a LEICA S440i scanning electron microscope and a field-emission scanning electron microscope (FESEM, NOVA NANOSEM 600). Energy-dispersive X-ray analysis (EDAX) was performed with an Oxford microanalysis group 5526 system attached to the SEM, employing Links (ISIS) software and a Si(Li) detector [11-13].

Results and Discussion

Figure 1. shows the XRD pattern of pristine stannic oxide (SnO₂) nanostructure synthesized by liquid phase via co-precipitation method calcinated at 400°C it is clearly observed that the highest intensity peak is obtained at (110) crystal planes and other peaks lying at (101), (200), (211), (220) and (002) of SnO₂. All the peaks match well with the standard tetragonal structure of SnO₂ with lattice constant $a = 4.723$ nm and $c = 3.238$ nm and its unit cell volume ($V=72.24\text{\AA}^3$) with JCPDS card no. 71-0652. All the peaks are perfectly match with pure SnO₂ nanostructure, which indicates the high purity of obtained SnO₂ nanoparticles. The average crystalline size was found to be 23.19 nm calculated by using Debye-Scherrer formula [12].

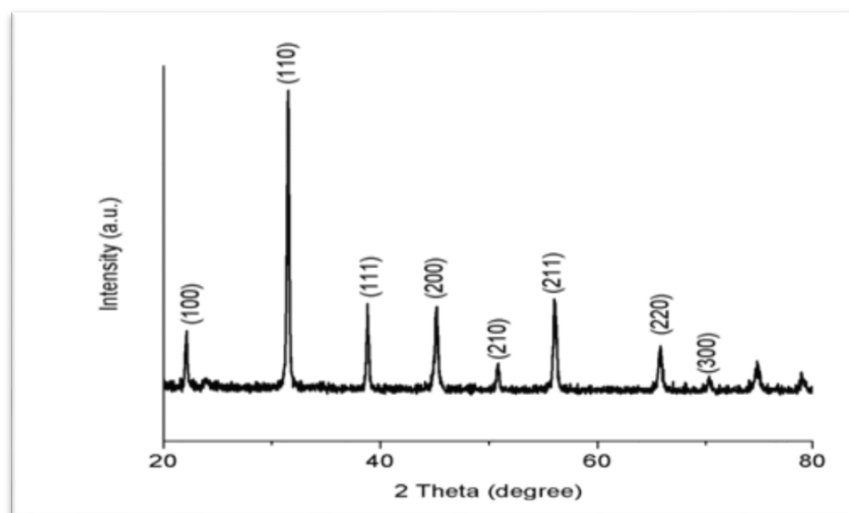


Fig. 1. XRD of SnO₂ nanoparticles

SEM images of the SnO₂ nanoparticles prepared by the co-precipitation method are shown in Fig.2(a,b). The images reveal spherical grain morphology. The average grain size obtained from the SEM micrographs is nearly 21 nm for the sample calcined at 600 °C. Tetragonal patterns of spots observed in selected images indicate the high crystallinity of the particles. Representative micrographs at different magnifications (scales of 100 nm, 1 µm, and various magnification levels from 10 000× to 100 000×) confirm the nanostructured nature and relatively uniform distribution of the particles [13-16].

The particle size determined by SEM (~21 nm) is consistent with the size obtained from XRD analysis (also ~23 nm), confirming the successful formation of nanocrystalline SnO₂.

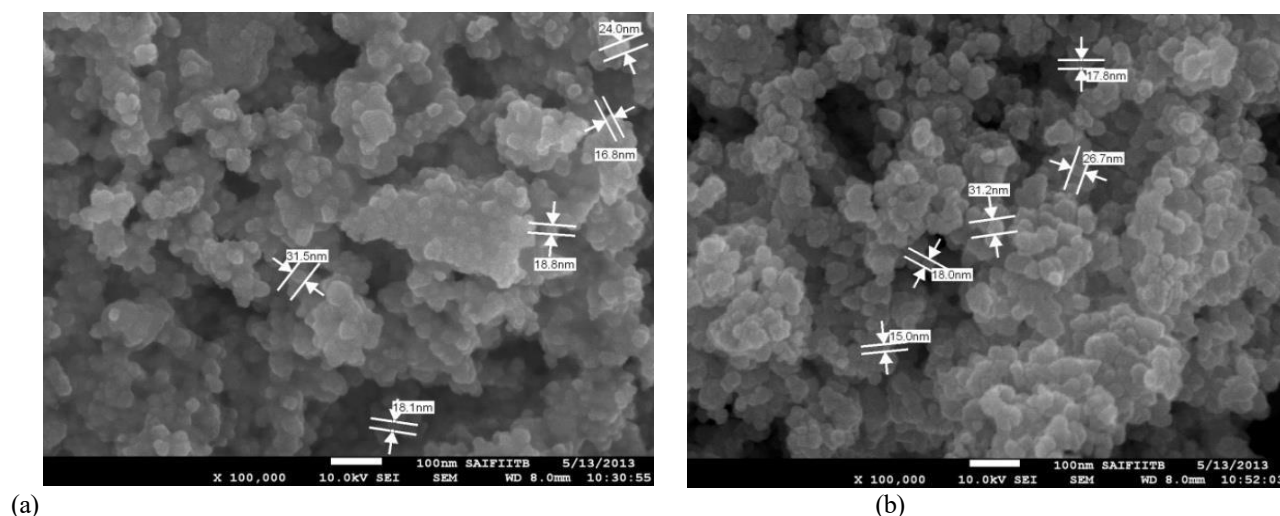


Fig.2(a), (b) SEM Pictures of SnO₂ nanoparticles

Conclusions

SnO₂ nanosized particles were successfully prepared by the co-precipitation method (followed by calcination). The average particle size of stannic oxide nanoparticles was found to be approximately 21 to 23 nm by both scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis. The results demonstrate that nanocrystalline SnO₂ can be obtained via a simple, low-cost co-precipitation route using readily available precursors. The particle sizes determined by the two independent characterization techniques are in good agreement.

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