

Influence of Initial Concentration for Bismuth Nitrate on Crystallite Size of Prepared α - Bi_2O_3 Nanoparticles

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Abstract

The bismuth oxide (Bi_2O_3) nanoparticles are easily synthesized from a solution of bismuth nitrate pentahydrate in methanol via chemical reduction method. X-ray diffraction, scanning electron microscopy and UV-visible spectroscopy are used to characterize the products. The results show that the initial solution concentration has significant influence on the crystallite sizes of Bi_2O_3 , and all the synthesized Bi_2O_3 samples have the cubic phase structure.

Key words:

Bismuth oxide, nanocrystalline materials,

1. Introduction

Bi_2O_3 is an important semiconductor with several main crystallographic polymorphs denoted by α - Bi_2O_3 (monoclinic), β - Bi_2O_3 (tetragonal), γ - Bi_2O_3 (cubic), δ - Bi_2O_3 (cubic), ϵ - Bi_2O_3 (triclinic) and ω - Bi_2O_3 (orthorhombic) crystal phases. Under normal atmospheric conditions α - and δ - Bi_2O_3 are the stable phases at room temperature, while β -, γ -, ω and ϵ - Bi_2O_3 phases are high-temperature metastable phases [1-4].

Bismuth oxide (Bi_2O_3) has been investigated extensively due to its optical and electrical properties such as high refractive index, large energy band gap, dielectric permittivity as well as remarkable photoluminescence and photoconductivity. These properties make bismuth oxide an interesting candidate for applications in the fields such as photocatalysis [5], superconductor ceramic glass [6], solar cells [7], piezo-optic materials [8], sensor optical coatings [9], oxide-ion conductors [10], medicine synthesis [11], and gas sensing [12].

Many methods have been reported to prepare Bi_2O_3 such as chemical vapor deposition (CVD) [13], high-temperature oxidation of bismuth metal [14, 15], sol-gel [16-18], pyrolysis of bismuth compound

[19, 20], reactive sputtering deposition [21], hydro-chemical method [22, 23].

The present work, describes synthesis of Bi_2O_3 NPs by chemical reduction method with raw material of bismuth nitrate pentahydrate and CTAB which was used as a capping agent then studying the effect of initial bismuth nitrate concentrations on the crystallite sizes of Bi_2O_3 NPs

2 Experimental Details

2-1 Chemical Materials

All chemicals used in the experiment were analytical reagent grade and were used without further purification.

The chemicals bismuth nitrate pentahydrate [$\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$] was purchased from Fuchen Chemicals Reagent (China), sodium hydroxide (NaOH) were obtained from Sigma-Aldrich chemicals, cetyltrimethylammonium bromide (CTAB) and methanol were supplied from Loba Chemicals.

2-2 Synthesis of Bismuth Oxide Nanoparticles

Bismuth Oxide nanoparticles were synthesized according to the following: 60 ml of a 0.04mol/L solution of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ (or 0.1 mol/L solution of bismuth nitrate) dissolved in methanol and the system was maintained under magnetic stirring until the total dissolution of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$. Then solution of CTAB of 0.03mol/L was added to the bismuth solution slowly with vigorously stirring. After stirring for about 15 min, white precipitate was formed. After stirring for 2 h, the mixture was heated at 90°C for 3 hours until the suspension color changed from white to yellow.

2-3 Measuring Instruments

The samples structure were analyzed with a Shimadzu 6000 X - ray diffractometer using $\text{Cu K}\alpha$ radiation ($\lambda=1.5406 \text{ \AA}$) in reflection geometry. A proportional counter with an operating voltage of 40 kV and

a current of 30 mA was used. . The diffracted X-ray intensity is recorded versus the diffraction angle (2θ) in the range between 10 to 80 degrees.

The topography and particle size of the prepared films were studied by field emission scanning electron microscopes (SEM) type S-1640 HITACHI Company (Japan) with different magnification powers and detectors at the Tehran University.

UV-VIS spectroscopy measurements were carried out by using UV/ 160 Shimadzu spectrophotometer, which operates in the wavelength range of 200 nm to 1100 nm.

3 Results and Discussion

3.1 XRD Studies

Figure (1) shows the XRD plots of the Bi_2O_3 produced by chemical reduction method with the two different initial bismuth nitrate concentrations.

The XRD pattern of the samples exhibits strongest diffraction peak at 27° , 32.6° and 46.8° corresponding to the Miller indices (120), (200) and (010) planes which indicate only pure monoclinic phase of crystalline α - Bi_2O_3 which are in good agreement with the literature value (JCPDF card number 41-1449).

The crystallite size of Bi_2O_3 synthesized with the two different initial bismuth nitrate concentrations can be calculated according to the Scherrer equation. These results show that the mean sizes of Bi_2O_3 are 26.1 and 13.7 nm corresponding to the initial concentrations of 0.04 and 0.1 mol/L, respectively which indicate that the crystallite sizes of the Bismuth oxide decrease with the increasing of the initial bismuth nitrate concentrations. Our results are good agreement with the results of X. Luan et.al [24].

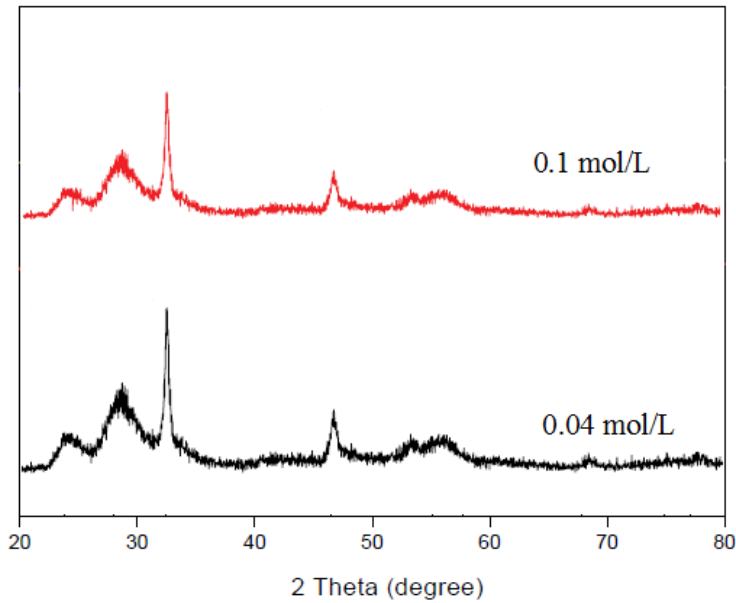


Figure (1): XRD image for Bi₂O₃ with two initial bismuth nitrate concentrations.

3.2 SEM Studies

Scanning electron microscopy (SEM) has been used to study the morphology and size distribution of the samples. The SEM image of the Bi₂O₃ shown in figures (2) and (3) with the two initial bismuth nitrate concentration of 0.04 and 0.1mol/ L. SEM image shows presence of flower- walls particles with the average size of 32.4nm and 24 nm for bismuth nitrate concentration of 0.04 and 0.1mol/L respectively. That's observed, initial bismuth nitrate concentration has significant influence on the crystallite sizes of Bi₂O₃.

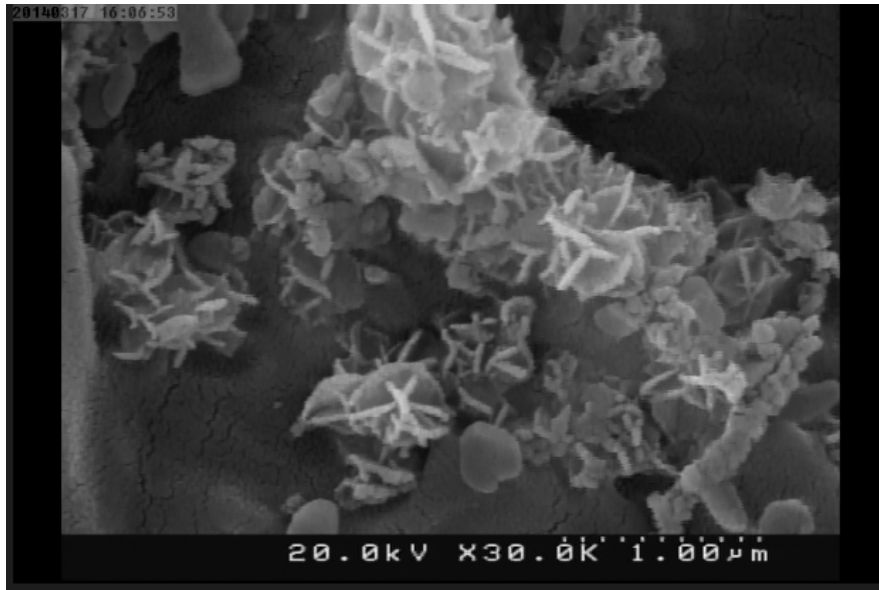


Figure (2): SEM image for Bi_2O_3 when the concentration of bismuth nitrate is 0.04 mol/L.

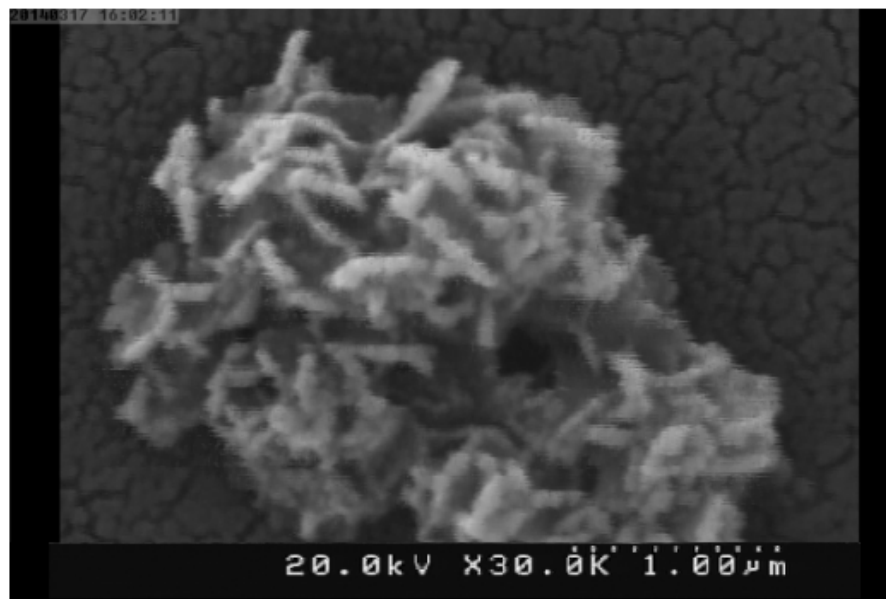


Figure (3): SEM image for Bi_2O_3 when the concentration of bismuth nitrate is 0.1 mol/L.

3.3 UV-Visible Absorption Spectra

The absorption spectra of $\alpha\text{-Bi}_2\text{O}_3$ synthesized via chemical reduction method are shown in Figure (4). It can be seen that both the

samples have a strong absorption at the wavelength 400 to 410 nm with the initial bismuth nitrate concentration of 0.04 mol/L and 0.1 mol/L respectively.

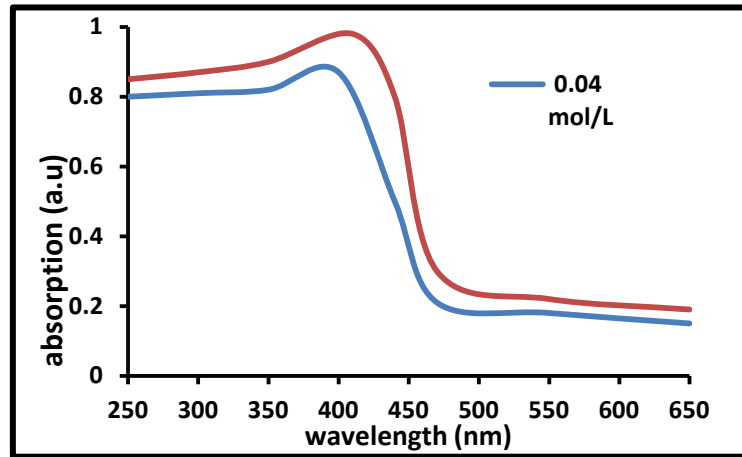


Figure (4): Absorption spectra for α - Bi_2O_3 NPs with two initial bismuth nitrate concentrations.

The energy gap was obtained from the relation between $(\alpha h\nu)^2$ versus $(h\nu)$. The result shows that the energy gap was increased with the increasing of initial bismuth nitrate concentration. As shown in the Table (1).

Table (1): Comparison between E_g calculated from the Absorption of Bi_2O_3 with the two concentration of bismuth nitrate.

bismuth nitrate concentration	Band gap E_g (eV)
0.04	2.79
0.1	2.8

4. Conclusions

α - Bi_2O_3 nanoparticles was prepared directly from chemical reduction method *with changing the* initial bismuth nitrate concentrations and the results proved that the initial bismuth nitrate concentration has significant influence on the crystallite sizes of Bi_2O_3 .

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