Ethylenediaminetetraacetic Acid-Assisted Synthesis of Nano Antimony Oxide by Microwave Method

Azadeh Tadjarodi*, Mohammad karimpour

Department of Chemistry, Iran University of Science and Technology, Narmak, Tehran 16846-13114, Iran

E-mail: tajarodi@iust.ac.ir

Abstract

Organic ligands are widely used in the synthesis of metallic complexes, which can be burned and eliminated from the coordination sphere at high temperatures and remaining only oxide group. The reaction of Ethylenediaminetetraacetic acid (EDTA) as an organic ligand and antimony trichloride in a domestic microwave leads to formation of antimony-EDTA complex. Then, the resulting compound was calcined and got nano antimony oxide. The product was characterized by powder x-ray diffraction (XRD), fourier transform infrared spectroscopy (FT-IR), field emission scanning electron microscopy (FE-SEM) and UV–vis diffuse reflectance spectrum (DRS). This technique is a simple, fast, environmental friendly and can draw a usable viewpoint for the synthesis of nanomaterials.

Keywords: Microwave, Green, EDTA, Antimony oxide

1. Introduction

Oxide nanoparticles have attracted great attention over past decade due to novel properties compared with bulk materials [1,2,3]. There are many reports on the synthesis, structure, and applications of nanocrystalline materials [4,5,6,7]. Antimony oxides are well known to exist in several different phases including Sb_2O_3, Sb_2O_4, Sb_6O_{13}, Sb_2O_5 and the polymorphism, which have attracted great attention in the field of catalysts, medicines, optics and semiconductors. Among the different antimony

oxides, Sb_2O_4 is the most common form of polymorphism, which has two types, namely the orthorhombic α phase and monoclinic β phase, respectively [8]. α-Sb_2O_4 has been widely used together with other oxides (e.g. Fe_2O_3, MoO_3 etc.) as a catalyst for the ammoxidation of propane to acrylonitrile, the oxidation of propane or propene to acrylic acid and for hydrocarbon cracking and hydrogen reduction, and in the production of ceramic enamels [9,10]. In this paper, for the first time, we report the synthesis of leaf-like α-Sb_2O_4 nanostructures by the microwave assisted method, which is a simple, fast, environmental friendly and green technique.

2. Experimental

2.1. Materials of synthesis

All of the chemical reagents used in this experiment were analytical grade and were used without further purification. Deionized water was used throughout.

2.2. Preparation

912 mg of antimony trichloride, $SbCl_3$, dissolved in deionized water and stirred for 10 min. Then 744 mg of EDTA under stirring was slowly added into this solutions, and stirred for 15 min. The resultant emulsion transferred into a microwave system in 180W for 20 min. The resulting product was collected, filtered and washed with deionized water for several times to remove residual ions in the product, and drying at 80℃ in air atmosphere for 3 h to produce the white products. Then the resulting powder was calcinated at 600 ℃ under the ambient atmosphere for 2 h and obtained white powder.

2.3. Characterization

The morphology of the product was observed directly by field emission scanning electron microscope (FE-SEM,VEGA/TESKAN microscope with an accelerating voltage of 30.00 KV). The phase of the product was determined by powder X-ray diffraction (XRD) analysis on a JEOL X-ray diffractometer with monochromatized Cu Kα radiation (λ=1.5418 Å). The UV−vis diffusive reflectance spectrum was performed in a Shimadzu, Mini 1240. The groups on the samples were studied by infrared spectroscopy using a FT-IR Shimadzu- 8400S Spectrometer.

3. Result and discussion

FTIR spectrum of the product has been shown in Fig. 1. The product reveals absorption peaks at 470, 532, 603, 651, 665, 759 cm^{-1}, which are characteristic peaks for the Sb-O-Sb bands.

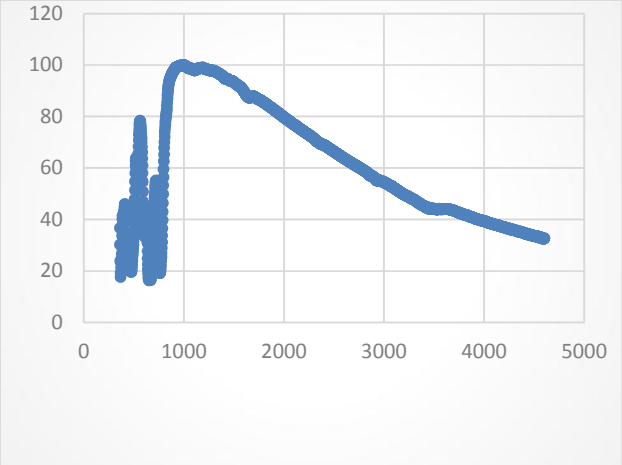

Fig 1. FT-IR spectrum of the product (α-Sb_2O_4)

The XRD pattern as given in Fig. 2 confirms that the product is pure antimony oxide, α-Sb_2O_4, with orthorhombic structure (JCPDS Card No. 01-071-0143) [7].

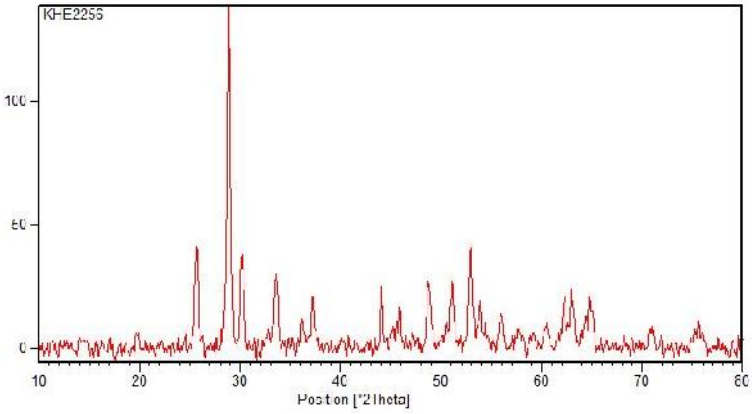

Fig 2. XRD pattern of the product (α-Sb_2O_4)

The band gap of α-Sb_2O_4 nanostructure is 3.6 eV, which was determined by the UV-Vis spectrum. Fig. 3 indicates the FE-SEM image of antimony oxide, α-Sb_2O_4, with leaf-like morphology. The diameter and width of leaf-like nanostructures are 150-200 nm and 80-100 nm, respectively.

Fig 3. SEM image of leaf like α-Sb_2O_4 nanostructure

4. Conclusion

We synthesized leaf-like α-Sb_2O_4 nanostructure by microwave method for the first time. FT-IR spectrum confirms the formation of the α-Sb_2O_4 nanostructures. The product was characterized by using XRD pattern. The FE-SEM image reveals morphology of α-Sb_2O_4 nanostructures as leaf-like. The band gap of the leaf-like α-Sb_2O_4 nanostructure is determined by UV-Vis spectrum, which is 3.6 eV.

References

1. D.W. Zeng, C.S. Xie, B.L. Zhu, W.L. Song, *Materials Letters* 58 (2004) 312-315.

2. D.W. Zeng, B.L. Zhu, C.S. Xie, W.L. Song, A.H. Wang, *Materials Science and Engineering A* 366 (2004) 332-337.

3. D.W. Zeng, X. Chen, R. Jiang, C.S. Xie, B.L. Zhu, W.L. Song, *Materials Chemistry and Physics* 96 (2006) 454-458.

4. Q. Sun, Q. Ren, H. Li, *Electrochemistry Communications*13 (2011) 1462-1464.

5. B. Ouni, M. Lakhdar, R. Boughalmi, T. Larbi, A. Boukhachem, A. Madani, K. Boubaker, M. Amlouk, *Journal of Non-Crystalline Solids* 367 (2013) 1-7.

6. Z. Zhang, L. Guo, W. Wang, *Journal of Materials Research* 16 (2001) 803-805.

7. A. Jamal, M. Rahman, S. Bahadarkhan, M. Abdullah, M. Faisal, A. Asiri, A. Parwazkhan, K. Akhtar, *Journal of Chemical Society of Pakistan* 35 (2013) 570.

8. Sh. Liu, B. Wen, W. Jiang, Ch. Liu, W. Ding ,N. Wang, W. Chai, *Ceramics International* 40 (2014) 15991–15995.

9. D. Orosel, P. Balog, H. Liu, J. Qian, M. Jansen, *Journal of Solid State Chemistry* 178 (2005) 2602-2607.

10. G. Ren, C. Wang, J. Xia, J. Liu, H. Zhong, *Materials Letters* 63 (2009) 605-607.