AUSTRALIAN JOURNAL OF BASIC AND APPLIED SCIENCES

ISSN:1991-8178 EISSN: 2309-8414
Journal home page: www.ajbasweb.com

Synthesis of Bismuth oxide Nano powders viaelectrolysis method and study the effect of change voltage on the size for it.

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ARTICLE INFO

Article history:
Received 18 February 2017
Accepted 5 May 2017
Available online 10 May 2017

Keywords:
α-Bi_2O_3, different voltage, ECD, electrochemical cell, electrochemical precipitation, Nano powder, rod electrode.

ABSTRACT

In recent year, a rapid increase in the use of nanomaterial due to high activity result of low particles size. Now days, the researchers were focused to prepare this materials and used it in different applications. By electrochemical method (ECD), Bi_2O_3 nanoparticles with different average sizes were prepared. Rods of bismuth and graphite were used as anode and cathode electrode respectively in cell depending on the reduction efforts. Sodium hydroxide was used as electrolyte solution to prepare basic mediumwhere combine the hydroxide ion with bismuth ions to form bismuth oxide. Many applications for bismuth oxide nanoparticle were used such as gas sensors, capacitor and catalyst. Bismuth oxide contained many phases and according electrolysis method, alpha phase of it was prepared. From the result, the energy gap of oxide nanoparticles was appeared blue shift and this means when increased the size of particles the energy gap for oxide increased. As result, the average of nanoparticles was increased with increasing voltage due to the speed in building nanoparticles. The identity and the structure of nano oxide were appeared from XRD technique while the average size for nanoparticles was characterized by AFM respectively.

INTRODUCTION

In the universe there is number of metal oxides are available in nature but some of the metal oxides are most useful in accordance with their applications of day to day life in science and technology. In the periodic table transition metals are large in number and have number of applications in different field applications.In the same way Bi_2O_3 is also one of the useful metal oxides and which has so many applications in different fields (Poizot, *et al*., 2000). Wide applications for Nano Bi_2O_3 were used as a catalyst (Sheng-ming, 2001 and Han-pei, *et al*., 2002) capacitor(Yu-bao, 2001 and Makarova, *et al*., 2006), additive in paints and gas sensors (Lazarevic, 2005, Nikolina, 2009, Laurent, 2008, Zong, 2001 and Sunarso, *et al*., 2009). Because of the compounds of Bi_2O_3 has structure face-centered cubic, it has high conductivity compare with all conductors oxide and it is the best solid electrolytes (Suk, 1996, Matsuoka, 1971 andDevi,*et al*., 1999). Four crystallographic polymorphs for Bi_2O_3were existed: α, β, γ and ε. Many methods were used to prepare it such as sol gel, chemical precipitation and hydrothermal methods. Sol gel method was contained brown gel of bismuth oxide that prepared by adding base to homogenous solution of bismuth salt and citric acid (Crişan, *et al*., 2008) while chemical precipitation contained formation of bismuth oxide by adding base to bismuth oxide and burn the precipitate (Tartaj, *et al*., 2000). Electrochemical deposition process is explained mainly by passing electric current between two electrodes. In this technique, the anode pole suffering from oxidation and cathode suffered of shorthand and converts to the metal ions with the help of stabilizers.In this research, ECD method was applied to prepare Nano powder of Bi_2O_3 by usingelectrochemical cell, different voltage value were used to

ToCite ThisArticle: Zaid Hamid., Synthesis of Bismuth oxide Nano powders via electrolysis method and study the effect of change voltage on the size for it. ***Aust. J. Basic & Appl. Sci.,*** *11(7): 97-101, 2017*

prepare different size of it.The electrochemical cell was contained from two electrode anode and cathode with electrolyte solution. According these method, bismuth rod was suffered oxidation process to prepare bismuth ions and in presence of basic medium, oxide was prepared.

2- Experimental:

2.1 Characterization:

Many device were used to character the nanoparticles that prepared by electrochemical deposition. Energy gap and functional group for nanoparticles were determined by using UV and FTIR techniques. AFM and XRD were used to character the image surface, average andcrystal system for it.

2.2 Synthesis of Bi_2O_3 nanoparticles:

100ml of (0.4) M NaOH was used as an electrolyte solution in electrochemical cell as fig 1.Two rod of bismuth and graphite were cleaned sonically before usedby using cleaning organic solution(ethanol, acetone, chloroform, de-ionized water). A rod of bismuth with dimension (3 cm x 1.5 cm x 0.1 cm) was used as an anode while rod of graphite with dimension (5 x 50 mm) use as cathode. Different voltage (10, 20 and 30V) was applied between two electrodes with current (0.07 to 0.1mA) for 30min.Yellow precipitate was appeared and washed with de-ionized water and dried over night at 60C.

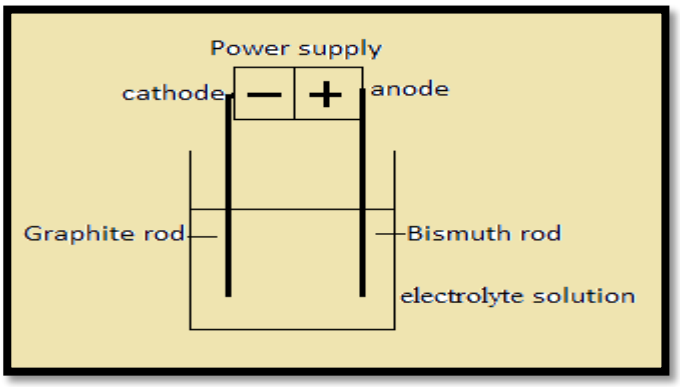

Fig. 1: Electrochemical cell

RESULT AND DISCUSSION

3.1- Energy gapstudies:

The energy required to transfer for the electron from the top to the bottom of valence shell known as the optical energy gap. The value of the optical energy gap for electronic transitions was calculated by using edge of absorption of Bi_2O_3 from the following equation:

$$Eg = 1240/\lambda max$$

Table 1 shows the value of energy gap. According to the results obtained, blue shift was occurred during increase the particle size of it due to a quantum confinement effect (Geetha*et al*., 2014).

Table 1: Band gap and absorption edges of α-Bi_2O_3 NPs.

sample	Voltages used in the preparation (V)	Average particle size (nm)	Absorption edge (nm)	gap energy (eV)
Bi_2O_3	10	39.08	371	3.34
Bi_2O_3	20	68.52	376	3.29
Bi_2O_3	30	89.05	397	3.12

3.2 - Study the effect of change voltage on the size:

According to practical part of the selection method of electrochemical deposition method for preparation bismuth oxides nanoparticles by using a variable voltage differential (10, 20, 30V), the standard of differentiation in this way is the quality of the product, and the intended quality here is a trend towards an increase in the amount of nanoparticles small diameter relative to the total amount of the prepared tube, and what is meant of quality is the trend towards increasing the amount of particles small diameter relative to the total amount of the particles that prepared (Kwon *et al*., 2007). The statistical and the morphology of surface study for nanoparticles were obtained by using an atomic force microscope and the results of the tests are shown in Fig 2, 3 and 4.

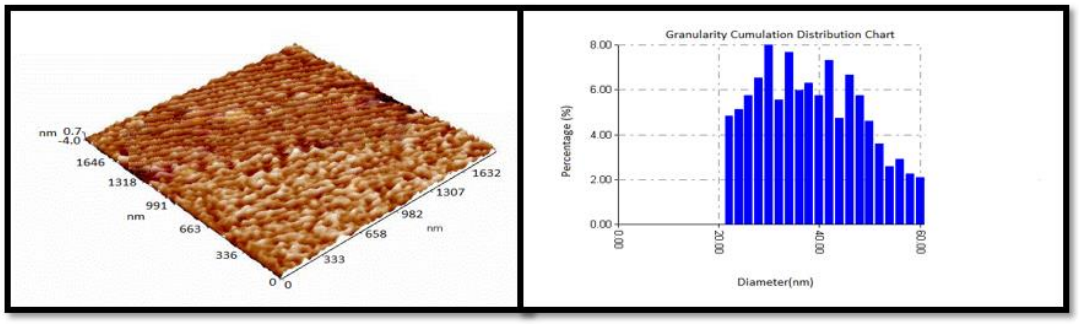

Fig. 2:3D image of AFM and granularity cumulative distribution chart for α-Bi_2O_3 that prepared by using 10Volt

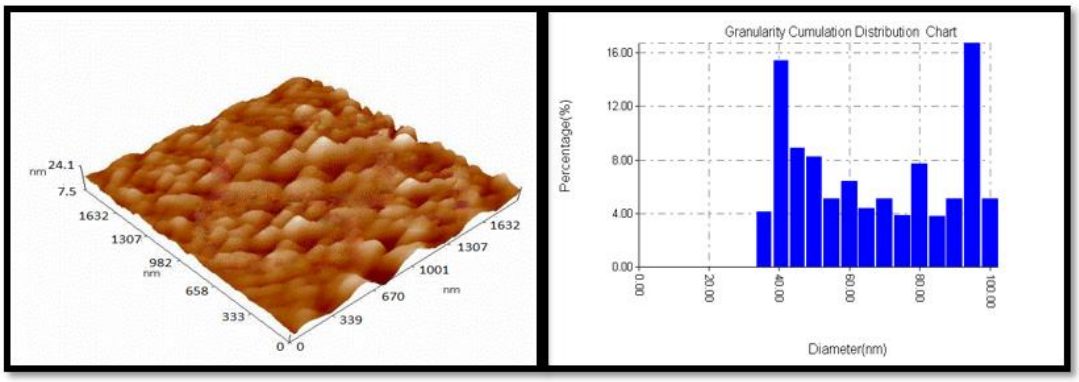

Fig. 3:3D image of AFM and granularity cumulative distribution chart for α -Bi_2O_3 that prepared by using 20Volt

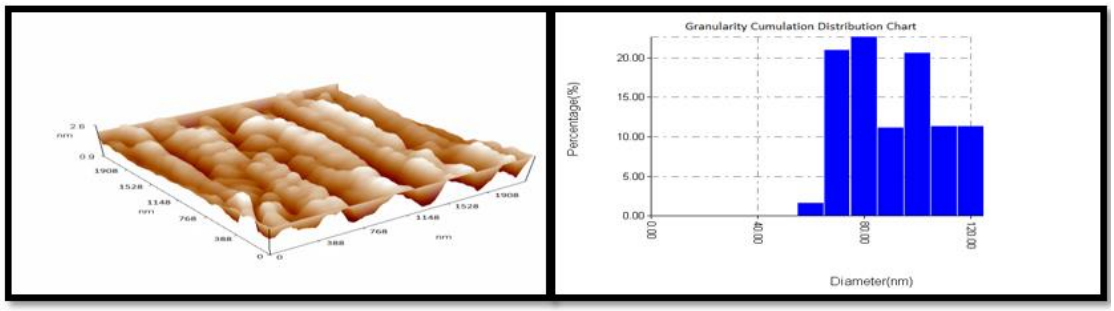

Fig. 4: 3D image of AFM and granularity cumulative distribution chart for α-Bi_2O_3 that prepared by using 30Volt

The electrochemical precipitation pathway to form **α**-Bi_2O_3(Lokhande*et al* 1998):-

$$Bi \rightarrow Bi^{+3} + 3e$$
$$Bi^{+3} + 3OH \rightarrow Bi(OH)_3$$
$$2Bi(OH)_3 \rightarrow Bi_2O_3 + 3H_2O$$

Through examination by device atomic force microscope for particles that prepared by using a variable voltage differential, the rate of the diameter of particles directly proportional to the user potentiometers which used during the preparationthat means we could get a few diameters when use the less voltage and the reason for this is that the high-voltage teams working on the speed of movement the electrons in the solution, which leads to rapid oxidation of bismuth atoms and turn it into a bismuth oxide, which yields the speed in building nanoparticles.

3.4- XRD analysis:

The structure of alpha phase for bismuth oxide nanoparticles were characterized by using XRD technique that prepared by electrochemical methods. Fig 5 was showed the pattern of alpha phase for it and it showed that the nanoparticles have monoclinic structure according standard card (JCPDS 00-041-1449).

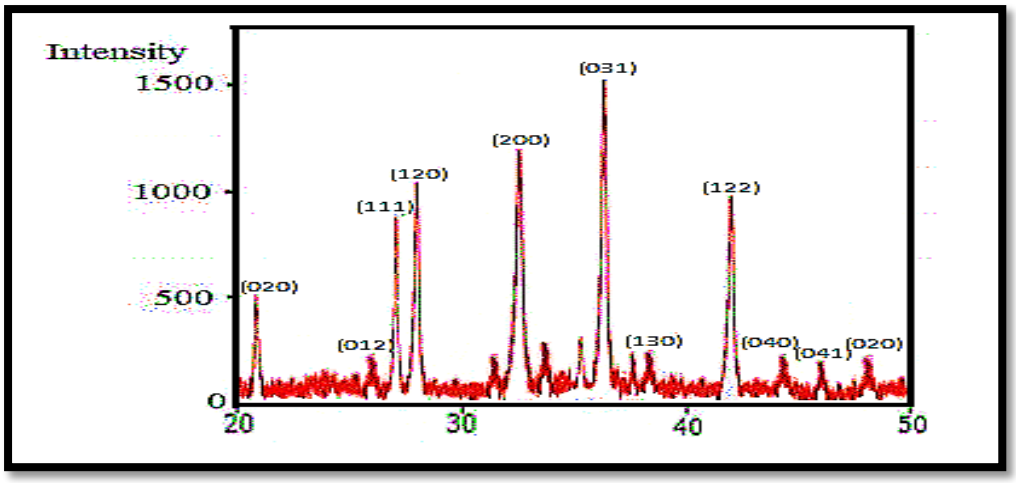

Fig.5:XRD of α-Bi_2O_3 NPs

The crystal size of nanoparticles was determined by using Debye-Scherrerequation: (Ahmad *et al*., 2016)

$D = k \lambda / \beta Cos\theta$

Where λ is the wavelength of the Cu-Kα radiations that used, k = 0.9scherrer constant, β is the full width at half maximum and θ is the angle obtained from 2θ values corresponding to maximum intensity peak in XRD pattern. The diffraction peaks corresponding to (020), (012), (111), (120), (200), (031), (130), (122) are quite identical to characteristic peaks of the alpha-Bi_2O_3crystal with the monoclinic structure.

Table 3-2:crystal size of α-Bi_2O_3Npsas calculated from Scherrer formula

sample	Voltages used in the preparation (V)	Crystal size (nm)
Bi_2O_3	10	22.3
Bi_2O_3	20	35.8
Bi_2O_3	30	40.1

4- Conclusion:

By simple electrolysis method, bismuth oxide nanoparticles have been successfully prepared. The checking of the AFM and XRD were showed us the composition of α-Bi_2O_3 nanoparticles in a way sedimentation electrochemical by using a variable voltage differential, as we found at least potentiometers we can get the small diameters rate due to speed in build for nanoparticles. Red shift with increasing the size of particles was appeared. AFM and XRD technique were proved the average size of particles size less than40nm.

5- Future work:

Using simple and low cost method such as electrolysis to prepare another nano oxide and use it in many applications such as remove heavy materials.

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