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SYNTHESIS OF Bi_2S_3 NANOPARTICLES BY FORCED CONDENSATION METHOD

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Abstract

Bismuth sulfide (Bi_2S_3) Successfully synthesized by Forced condensation method. The structural, morphological, and optical characteristics of Bi_2S_3 nanoparticles produced by the reflux technique at 160°C for 3 and 6 hours have been examined in this study. Powder X-ray diffraction (XRD) analysis was used to examine the structures of the Bi_2S_3 nanoparticles and discovered that they are orthorhombic. Scanning electron microscopy (SEM) has been used for the morphological research. Using UV-Vis spectroscopy, the optical band gap and functional group analysis have been described.

Keywords: Bi_2S_3 nanoparticles, XRD, SEM, UV-Vis, reflux method.

1. Introduction

A nanoparticle, also known as an ultrafine particle, is a matter particle with a diameter of 1 to 100 nanometres. The term is also applied to larger particles with diameters of up to 500 nm, as well as fibres and tubes with diameters of less than 100 nm in only two directions [1]. Tissue-specific nanoparticles have demonstrated significant promise as contrast agents for in vivo medical imaging of various cancer types. So far, much of the research has concentrated on nanoparticle contrast agents for magnetic resonance and optical imaging studies. The physical and chemical qualities of the materials used must be under control for technologies like solar cells, supercapacitors, photocatalytic coatings, and electrochromic windows to function optimally [2]. Nano materials are used to enhance the engineering materials and variety of applications. Reliable, sensitive, selective, and inexpensive electrochemical sensors and methods for detecting (heavy) metal ions and other relevant substances continue to pique the interest of researchers due to the toxic nature of these analytes, their widespread production and application, and, as a result, their widespread and increasing occurrence in the environment and, ultimately, in the food chain [3]. Despite the well-known toxicity of mercury and its associated occupational health hazards, the inconvenience of application in flow systems, and difficulties in its handling, storage, and disposal, various types of mercury electrodes have been most used for metal ion and other analyte detection over the last six decades [4]. Attempts to replace mercury with less toxic electrode materials have been done in the past, but none have come close to the exceptional electroanalytical performance of mercury.

Designing new functional nanomaterials is made possible by the synthesis of inorganic nanomaterials with adjustable shape, orientation, and dimensionality [5]. Due to its potential applications in the disciplines of electronics, magnetism, optoelectronics, catalysis, and biomedicine, the self-assembly of low dimensional (0D, 1D, and 2D) nanostructures into three-dimensional (3D) superstructures is particularly hot right now [6]. A variety of inorganic materials, such as metal, metal oxide, hydrate, sulphide, etc., have been created having hierarchical superstructures. However, [7] directed construction of nanorods, nanotubes, nanoplates, and other structures is typically challenging. To control their directed growth, it typically takes hard or soft templates, which not only introduces heterogeneous contaminants but also raises production costs [8]. Bismuth sulfide (Bi_2S_3) is a naturally occurring mineral. The creation of Bi_2S_3 has been extensively researched and examined during the last few decades. Controlling the crystal size and nanocrystalline structure poses a new challenge for scientists studying synthetic chemistry and materials [9].

Chemical synthesis methods prove to be a boon for young researchers as they are cost effective and easy to handle and its band gap energy makes it a good photocatalyst that is active in the visible range of the solar spectrum. One of the most widely studied structural form of bismuth sulfide is the 1D nanostructures. [10,11,12]. In this study, the successful synthesis of Bi_2S_3 nanoparticles utilising the basic reflux process is described, along with the impact of reactions duration and the initial components of bismuth nitrate, thioura, and NaOH. With help of XRD, SEM, and UV-Vis spectrophotometers, the synthesized sample's properties, including its structure, optical, morphological, and vibrational behaviour, have been investigated. The results are discussed below [13].

2. Experimental procedure

All the starting materials were analytical grade and used

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without any further purification. Bismuth nitrate ($Bi(NO_3)_3.5H_2O$), and thiourea ($CS(NH_2)_2$, were used as the starting materials. Sodium Hydroxide (NaOH) was employed as a surfactant. In typical synthesis of, 0.025 mole of Bismuth nitrate and 0.05 mole of thiourea were dissolved in 200 ml of double distilled water. 0.025 mole of Sodium Hydroxide was added in the above solution and the solution was stirred and sonicated for the chemicals to dissolve. The solution was transformed to round bottom flask and refluxed at 160°C with continues stirring for two different hours (3 hours and 6 hours) respectively. The black solid particle was collected and washed several times with double distilled water and acetone. Then the product was dried using hot air oven at 60°C for 6 hours. The final products were characterized.

3. Result and Discussion

3.1 Structural analysis

By using an X-ray diffraction (XRD) pattern, the crystal structure of the produced Bi_2S_3 nanoparticles was examined. The 3 hour and 6-hour reaction periods used to prepare the Bi_2S_3 nanoparticles were shown in Fig.1 XRD pattern. Both the interval to the source and the size of the crystallite can be used to correlate the peak XRD intensity. Broad weak peaks, on the other hand, correspond to small particles of high crystalline material and a sharp intensity peak, huge particles off of high crystalline material with a significant number of flaws [10]. The XRD pattern of as-prepared samples 160 C was indexed as a pure orthorhombic phase which is very close to the in JCPDS # 17-0320 card values with the lattice Constance a=1.11300nm, b=1.11260nm and c=0.3960nm. The strong and sharp reflection peaks in the XRD patterns indicated that Bi_2S_3 products were well-crystallized.

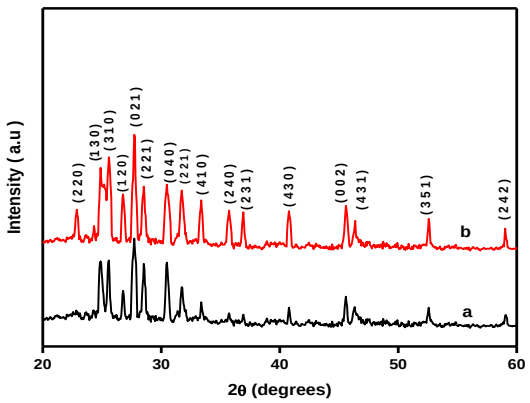

Fig.1 XRD pattern of the Bi_2S_3 nanoparticles prepared with (a) 3 hours and (b) 6 hours.

The crystallite size was calculated by using Scherer's formula.

$$D=\frac{k\lambda}{\beta\cos\theta}$$

Where the constant K is a shape factor usually 0.94, λ is the wavelength of the X-ray (0.1541 nm), β is the full-length half maximum (FWHM) in radians and Θ is Bragg's angle. The crystallite size of 3 hours and 6 hours as-prepared Bi_2S_3 nanoparticles were found to be 17.89 nm and 27.3 nm respectively. Comparing the XRD patterns obtained at different reaction time, both the integrity and the intensity of the crystalline were found to be improved with an increase of the reaction time.

3.1.1 W-H Plot

This plot is used to estimate the crystallite size and micro strain. It plays the vital role in separating particle size and micro strain in broadening analysis of XRD pattern. The effective relation between size and strain is given by $\beta_{hkl} = \beta_d + \beta_s$

$$\beta_{hkl}\cos\theta = \frac{K\lambda}{D} + 4\varepsilon\sin\theta$$

The straight line in W-H plot indicates the homogeneous distribution of particle size and micro strain. The strain and distortion were calculated using the formula given below,

$$\varepsilon = \frac{\beta_{hkl}}{4\tan\theta}$$

The W-H plot is drawn between 4sinθ along the x-axis and y-axis has $\beta_{hkl}\cos\theta$.

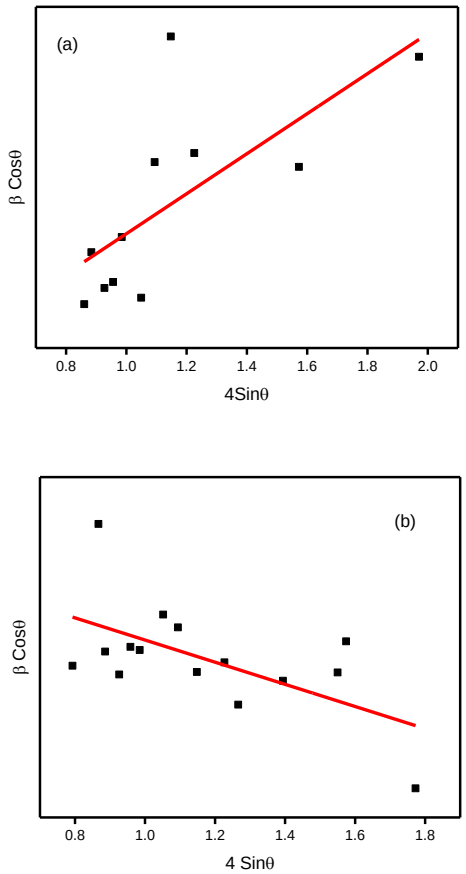

Fig. 2 a) W-H plot for 3 hours; b) W-H plot for 6 hours.

From this XRD pattern, while compared to the time in Scherrer equation, in 6 hour the crystallite size gets decreased and the time of 3 its increased. At the same time in

W-H plot the crystallite size both are same. From the W-H plot, the micro strain is decreased for 3 hours.

Table 1: Scherrer equation and W-H plot crystallite size.

Time	Crystallite Size (nm)		Micro strain
	Scherrer equation	W-H Plot	
3	17.89 nm	24.1 nm	2.27235E-06
6	27.3 nm	24 nm	1E-06

3.2 Morphological analysis

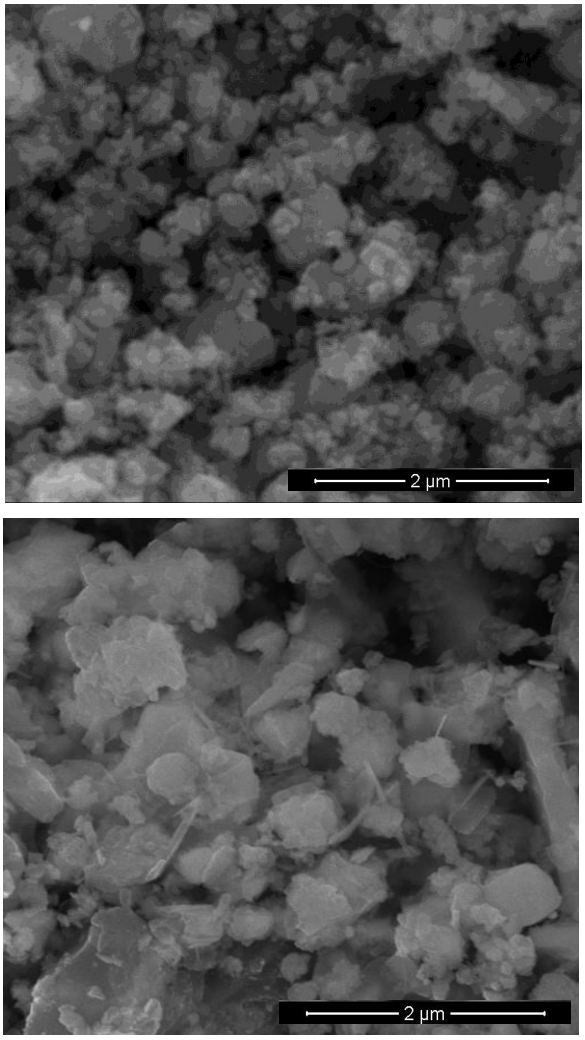

Fig. 3 SEM image of the Bi_2S_3 nanoparticles prepared (a) 3 hours and (b) 6 hours.

The morphology of the as-prepared Bi_2S_3 nanoparticles have prepared with different reaction times (3 hours and 6 hours) using reflux method 160°C as shown in fig.3. The micrograph representing the Bi_2S_3 nanoparticles revels homogenous nanocrystalline structure composed of spherically shaped. It is seen that spherical particles were distributed over the sample and the particles are highly agglomerated for the Bi_2S_3 nanoparticles samples prepared with 3 hours. Whereas the samples prepared for 6 hours show formation of plate like structures here and there. An average particle size of around 66 nm and 73nm were witnessed for the samples prepared for 3 and 6 hours respectively.

3.3 Optical analysis

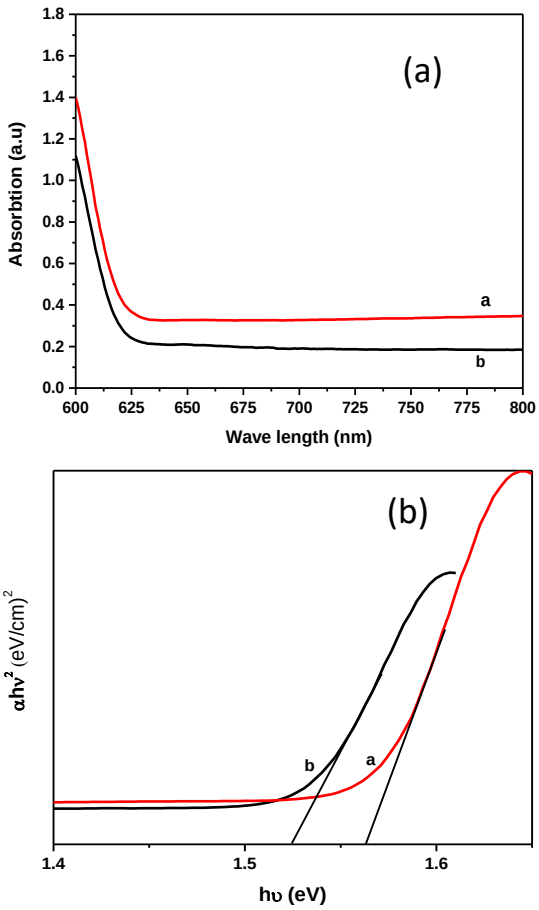

Fig: 4 a) UV-Vis absorption spectra and band gap of different reaction times of 3 hours and 6 hours as shown in fig.4 (a) & (b).

The UV-Vis spectroscopy has become an effective tool in determining the size and the optical properties of the nanoparticles. The optical absorbance spectra of the prepared samples are recorded in the wavelength range from 200 nm to 800 nm. The UV-Vis absorption spectra of the as-prepared orthorhombic Bi_2S_3 nanoparticles prepared with different reaction times of 3 hours and 6 hours as shown in fig.4 (a) & (b). From the spectra it is observed that the Bi_2S_3 nanoparticles are highly absorbed in the UV-Vis region. This could be because of the very small variation observed in the particle size of the samples with different reaction time. The UV lower cut off wavelength is found to be 252.8 nm and 313.26 nm respectively. The band gap energies as estimated from the spectra of Bi_2S_3 nanoparticles prepared with different reaction time namely 6 and 3 hours is found to be 1.52 eV and 1.56 eV respectively.

4. Conclusion

In summary, one dimensional Bi_2S_3 nanoparticles have been successfully prepared by reflux method. From the investigation on the effects of reaction time, it was conclud-

ed that the increase in the reaction time resulted in higher degree of crystallinity. However, the particle size increased with increasing crystallinity. The structure of the prepared sample was obtained from XRD pattern as orthorhombic crystal system and the prepared samples exhibited a polycrystalline nature. The morphology of the prepared samples was studied using SEM. The UV-Vis spectroscopic studies reveal that the optical band gap is 1.52 eV and 1.56 eV.

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