SbO_2 Nanoparticles: Structural, Morphological and Optical analysis for Photocatalytic applications

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Abstract

A simple co-precipitation method is employed to synthesize pure SbO_2 nanoparticles in this present work. The synthesized nanoparticles are characterized using powder X-ray diffraction (PXRD), field effect scanning microscopy (FESEM), (Energy dispersive X-ray analysis (EDAX), Raman and photoluminescence spectroscopy (PL). PXRD studies revealed the formation of SbO_2 in orthorhombic from JCPDS File No. 65-2446. The surface of FESEM images of antimony oxide nanoparticles showed both spherical and granular like structure. Energy dispersive X-ray of SbO_2 displayed the presence antimony (Sb) and oxygen (O). Raman spectra also revealed the formation of pure SbO2 nanoparticles. PL emission spectrum of the synthesized sample indicated a blue shift and emission in the visible region. Thus the synthesized SbO_2 nanoparticles can be finely tuned for photocatalytic applications.

Keywords - *Co-precipitation, SbO_2 nanoparticles, PXRD, FESEM, EDAX, Raman, PL spectroscopy*

I. INTRODUCTION

Oxide nanoparticles have increasingly gained interest over the past decade due to the possibilities of fabricating the nanostructured materials showing novel properties compared with bulk materials [1-3]. Antimony oxide is one of the important sensor materials for detecting leakage of inflammable gases because of their high sensitivity to low gas concentration and used in industries as a conductive material, functional filters, adhesives and textile coating [4,5]. Antimony oxide nanoparticles are semiconductor V-VI binary compounds used as catalyst, retardant, fining agent and optical materials [6,7]. Nanostructured antimony oxide nanoparticles have been prepared through various techniques such as vapour transport and condensation [8-10] of metallic antimony in oxidizing environment, pulsed laser ablation method [11], chemical method [12], hydrothermal process [13-15].

In the present work, pure SbO_2 nanoparticles are prepared via cost effective, co-precipitation method and the structural, morphological and optical properties are investigated.

II. EXPERIMENTAL DETAILS

All chemicals used were of analytical grade and purchased from Merck.

0.25M of antimony chloride was dissolved in 50 ml of double distilled water. Also 0.75 M of oxalic acid wa sdissolved in 50 ml of double distilled water. Both the solutions were stirred using magnetic stirrer for 20 minutes at room temperature. Then the antimony chloride solution was added dropwise into the oxalic acid solution. 32M of NH_4OH was mixed in 50 ml of double distilled water and this solution was added dropwise into the prepared solution until the pH of the solution becomes 12. During this process, precipitation had taken place. Then the solution was washed using double distilled water for three times and ethanol for two times. Now the solution was dried in a hot plate at 100°C for 3 hours. Then it was finely ground and calcined in the muffle furnace at 500°C for 2 hours. Finally the white coloured pure SbO_2 nanopowder is formed.

The structural analysis of SbO_2 nanoparticles was performed by recording the powder X-ray diffractometer (PANalytical X'Pert Pro) with Cu-K_α as the radiation source (1.54056 Å) over the 2θ range of 10° to 80°. The morphology of SbO_2 nanoparticles is investigated by FEI QUANTA-200 microscope and EDAX analysis is done. The Raman spectroscopy of the nanocomposites is carried out using Laser Raman spectrometer.. Photoluminescence (PL) studies were carried out using a photoluminescence spectrophotometer (Varian Cary Eclipse) and the spectra were recorded at a scan rate of 600 nm/min in the range of 375 nm - 600 nm using an excitation wavelength of 325 nm.

III. RESULTS AND DISCUSSION

A. PXRD analysis

The d-spacing values obtained from PXRD data of the as-prepared pure SbO_2 nanoparticles matched well with that of the JCPDS File No.65-2446

[a=4.814Å:b=5.435Å:c=11.783Å]. It is confirmed that the synthesized pure SbO_2 nanoparticles exhibit an orthorhombic structure. The sharp PXRD peaks (Fig.1) clearly indicate the polycrystalline nature of the synthesized pure SbO_2 nanopowder sample [16].

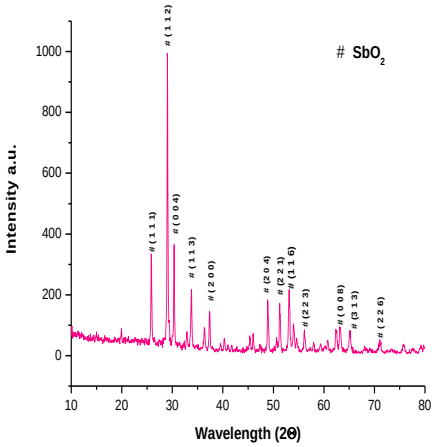

Fig.1 PXRD pattern of pure SbO_2 nanoparticles

The average grain size is found to be 57.9975 nm as calculated using Debye Scherrer formula

$$D = \frac{0.94\lambda}{\beta_{1/2}\cos\theta}$$

where λ is the wavelength of the CuK_α radiation [λ=1.5406Å], $\beta_{1/2}$ is the full width at half maximum of the diffraction line, θ is the angle of diffraction.

B. FESEM and EDAX

The FESEM image and EDAX are shown in Fig.2 and Fig,3 respectively. The surface of FESEM images of antimony oxide SbO_2 nanoparticles shows both spherical and granular like structure. Energy dispersive X-ray of SbO_2 shows the presence antimony (Sb) and oxygen (O). No additional peaks corresponding to any other elements except Sb and O, which confirms the purity of the nanopowder sample.

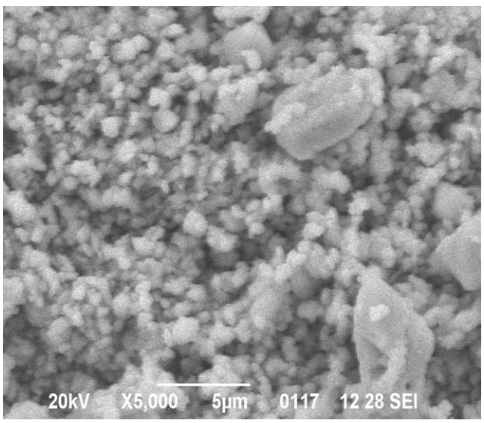

Fig.2 FESEM pattern of pure SbO_2 nanoparticles

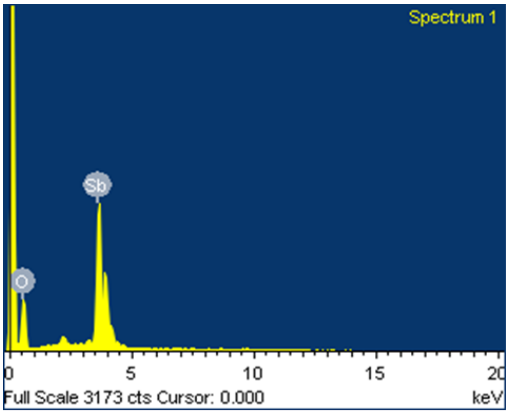

Fig.3 EDAX of pure SbO_2 nanoparticles

C. Raman Spectroscopy

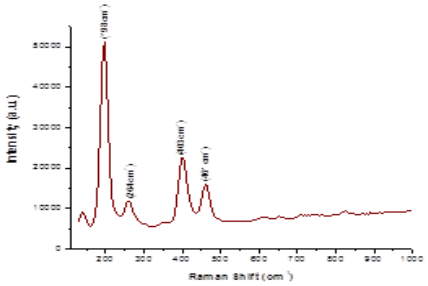

Fig.4 Raman spectra of pure SbO_2 nanoparticles

Fig.4 shows the Raman spectra of SbO_2 nanoparticles with its characteristic peaks at 198 cm^{-1}, 264 cm^{-1}, 403 cm^{-1},461 cm^{-1}. , which are responsible for the Sb-O andSb-O-Sb stretching vibration [17] .

D. PL analysis

Fig.5 depicts the photoluminescence emission spectrum showing a maximum peak at 361 nm and other peaks are seen at 437 nm and 489 nm. Some small peaks are observed at 376 nm and 410 nm. The blue shift confirms the nanosize of the synthesized SbO_2. These peaks are in good agreement with the findings of Cebriano et.al. [17].

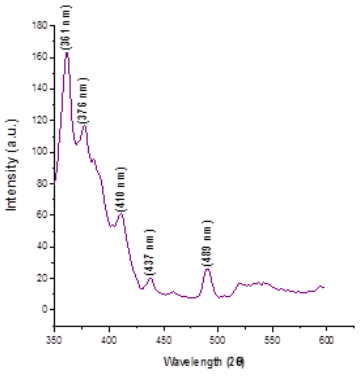

Fig.5 PL emission spectrum of pure SbO_2 nanoparticles

IV. CONCLUSION

In the present work, pure SbO_2 nanoparticles are synthesized by co-precipitation method. The preparation process is simple, homogeneous and cost effective. PXRD confirms the formation of the pure SbO_2 nanoparticles. The surface of FESEM images of antimony oxide SbO_2 nanoparticles shows both spherical and granular like structure. Energy dispersive X-ray of SbO_2 shows the presence antimony (Sb) and oxygen (O). Raman spectra also reveals the formation of pure SbO_2 nanoparticles. PL emission peaks of the synthesized nanoparticles show a blue shift. Thus the synthesized SbO_2 nanoparticles of varied morphology and structures can be used for high optical and photocatalytic applications.

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