Synthesis and Comprehensive Characterization of Nanocrystalline SnSe: Structural, Compositional, and Morphological Analysis

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Abstract:

SnSe attracts considerable attention due to its potential applications in thermoelectric, optoelectronic, and energy storage devices. This study reports a simple, cost-effective chemical precipitation method for synthesizing nanocrystalline SnSe. Detailed characterization of the synthesized SnSe was performed using XRD, EDAX, and SEM to gain insights into its crystallographic structure, elemental makeup, and morphological features. An orthorhombic phase was identified through XRD and the average crystallite size was found to be 24.34 nm. EDAX analysis verified the presence of Sn and Se as the major constituent elements with slight deviation from ideal stoichiometry. SEM images revealed agglomerated layered microstructures formed by clustered nanocrystalline domains. These findings confirm that the adopted synthesis method is suitable for preparing nanocrystalline SnSe with controlled characteristics.

1. Introduction:

As a prominent IV–VI semiconductor, SnSe possesses unique physicochemical characteristics that make it a promising material for gas sensors, thermoelectric devices, solar cells, and energy storage applications [1-3]. Various synthesis routes including hydrothermal, solvothermal, hot-injection and solid-state methods have been employed to produce SnSe nanostructures [4-7]. Nevertheless, these methodologies typically demand elevated processing temperatures, extended reaction durations and the use of sophisticated or specialized instrumentation [5, 6]. In recent years, there has been growing interest in low-temperature and solution-based methods to synthesize nanocrystalline SnSe with controlled particle size and morphology [6, 8-10]. Among these techniques, chemical precipitation is particularly notable for its operational simplicity, ease of scalability and good control over crystallite size and morphology [8-10]. A detailed understanding of the relationship between structure and properties at the nanoscale is important for enhancing the functional performance of SnSe-based devices [2, 11]. The combination of low thermal conductivity and a relatively high Seebeck coefficient has established SnSe as a promising thermoelectric material [2, 11, 12]. Additionally, its earth abundance, non-toxicity and environmental stability make it an attractive alternative to conventional heavy metal-based semiconductors [1, 2]. By tailoring synthesis conditions, it is possible to engineer the morphology and crystalline quality of SnSe to suit specific applications [4, 6, 8]. Therefore, developing a facile and efficient synthesis route along with detailed characterization is essential for advancing SnSe-based materials research [1-3]. This study focuses on a chemical precipitation method offering simplicity, cost-effectiveness and effective control over crystallite size and morphology [8-10]. Nanocrystalline SnSe was

synthesized through the reaction of Sn^{2+} ions complexed with L-ascorbic acid and Se^{2-} ions obtained by reducing selenium powder with $NaBH_4$. Structural, Compositional, and Morphological characteristics were analyzed using XRD, EDAX, and SEM techniques.

2. Materials and Methods:

2.1 Chemicals and Precursors:

Commercially available analytical-grade reagents were used throughout the study without any pre-treatment. Tin (II) chloride dihydrate, elemental selenium powder, L-ascorbic acid and sodium borohydride were used as the metal source, chalcogen source, stabilizing agent and reducing agent respectively. The common solvent used in all synthesis procedures was double-distilled (DI) water.

2.2 Green Synthesis of Nanocrystalline SnSe:

The nanocrystalline SnSe was obtained using a precipitation-based method in solution under rigorously optimized conditions [8-10]. To initiate the process, 0.05 mol of $SnCl_2 \cdot 2H_2O$ was dissolved in 25 mL of deionized water under magnetic stirring until complete dissolution was achieved. Subsequently, 0.5 mol of L-ascorbic acid ($C_6H_8O_6$) was introduced to act as a chelating and stabilizing agent ensuring controlled complexation of Sn^{2+} ions. This mixture was maintained at 70 °C with continuous agitation for 10 minutes producing a clear, homogeneous solution designated as Solution A. In a separate beaker, 0.04 mol of fine selenium powder was dispersed in 25 mL of deionized water. A measured quantity of $NaBH_4$ (0.06 mol) was then added as a strong reducing agent facilitating the conversion of elemental Se to selenide ions (Se^{2-}). This precursor solution was also stirred at 70 °C for 10 minutes yielding a transparent solution referred to as Solution B. Following preparation, Solution B was introduced dropwise into Solution A under vigorous stirring promoting uniform ion interaction and initiating the nucleation of SnSe. The combined reaction mixture was heated to 90 °C and held for 10 minutes enabling crystal growth. Upon completion, the system was cooled naturally to room temperature resulting in the appearance of a fine black precipitate. The solid product was recovered by vacuum filtration, rinsed repeatedly with deionized water to eliminate residual by-products and dried for 5 hours to obtain nanocrystalline powder.

2.3 Characterization Techniques:

A combination of structural, compositional and morphological analyses was employed to evaluate the synthesized nanocrystalline SnSe. Crystallographic information and phase identification were obtained from X-ray Diffraction (XRD) measurements. The elemental constituents were evaluated using Energy Dispersive X-ray Analysis while Scanning Electron

Microscope was employed to examine the surface features and internal structure of the material.

3. Result and Discussion:

3.1 X-ray Diffraction (XRD):

X-ray diffraction (XRD) analysis was carried out to examine the structural properties of the synthesized nanocrystalline SnSe and the resulting diffraction pattern of the sample is shown in Figure 1. The diffraction pattern exhibits sharp reflections at 2θ values of 15.32°, 21.04°, 29.76°, 30.69°, 37.35°, 41.36°, 43.71°, 46.89°, 48.53°, 52.83°, 61.52°, 63.67°, 67.18° and 78.46° which can be indexed to the (200), (101), (011), (111), (311), (102), (411), (112), (121), (402), (122), (521), (113) and (901) crystallographic planes of orthorhombic SnSe [4, 9, 10, 13]. This indexing is in excellent agreement with the standard reference data provided by JCPDS card no. 00-032-1382 confirming the successful formation of orthorhombic SnSe without detectable secondary phases [4, 13]. The refined lattice constants were calculated to be a = 11.56 Å, b = 3.39 Å, and c = 4.53 Å closely matching reported literature values for orthorhombic SnSe, thereby validating the structural characteristics of the as-synthesized SnSe [4, 9, 13]. The widening of the diffraction peaks suggests that the synthesized SnSe possesses a nanocrystalline structure. The Debye–Scherrer equation [14] was employed to determine the crystallite size from XRD peak broadening:

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

In the given expression, K represents the dimensionless shape factor (0.94), λ is the X-ray wavelength (1.5406 Å), β signifies the full width at half maximum in radians and θ denotes the Bragg diffraction angle. By employing this technique, the sample exhibited an average crystallite size of 24.34 nm [4, 6, 13].

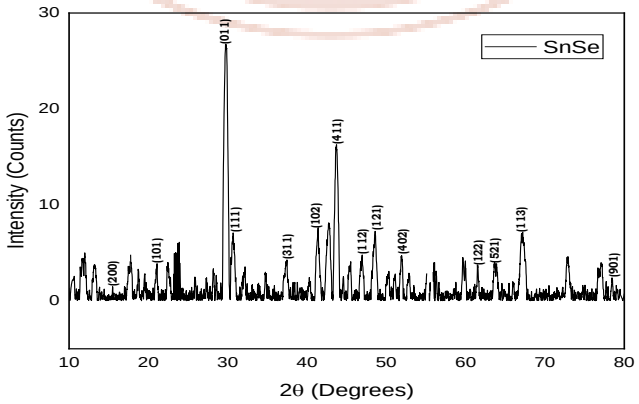

Figure 1: XRD pattern of as-grown nanocrystalline SnSe powder

3.2 Energy Dispersive X-ray Analysis (EDAX):

Figure 2 was used to depict the EDAX spectrum corresponding to the prepared nanocrystalline SnSe. The spectrum is dominated by well-defined peaks corresponding to tin (Sn) and selenium (Se) confirming the intended elemental composition [13, 14]. In addition, low-intensity peaks for carbon (C) and oxygen (O) are visible which can be attributed to surface adsorption from ambient air, residual carbon-based supports or minor environmental contamination. Such extraneous signals were either excluded from quantitative evaluation or corrected to ensure accurate compositional determination of the primary elements. The EDAX results indicate the presence of Sn and Se as the major constituents although a slight Sn-rich deviation from ideal stoichiometry is observed, as summarized in Table 1.

Table 1: The Sn and Se elemental composition of the as-synthesized SnSe was determined via EDAX

Elements	Experimental Weight (%)	Theoretical Weight (%)
Sn	65.26	60.06
Se	34.74	39.94

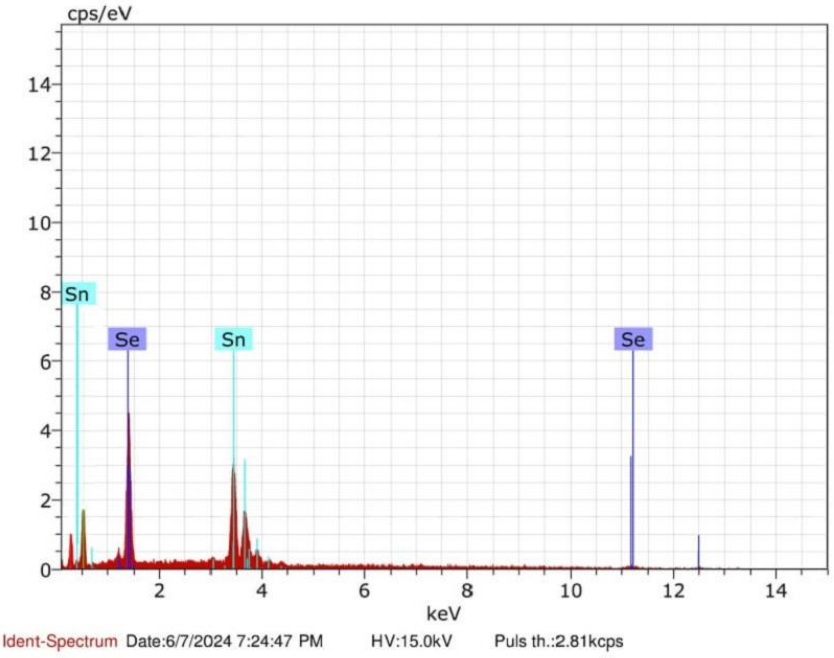

Figure 2: EDAX spectrum of as-grown nanocrystalline SnSe powder

3.3 Scanning Electron Microscopy (SEM):

Scanning Electron Microscopy (SEM) analysis of the synthesized nanocrystalline SnSe as illustrated in Figure 3 reveals agglomerated layered microstructures with irregular morphology [4, 6, 8, 9]. Although the observed structures are in the micrometre range, the XRD results confirm the nanocrystalline nature of the synthesised material suggesting that these larger structures are formed through the aggregation of nanoscale crystallites [14]. The layered morphology and surface roughness observed in the SEM images may be attributed to particle agglomeration during the synthesis, filtration and drying processes; these structural features are commonly observed in chemically synthesised SnSe materials and are thought to be advantageous for thermoelectric and optoelectronic applications.

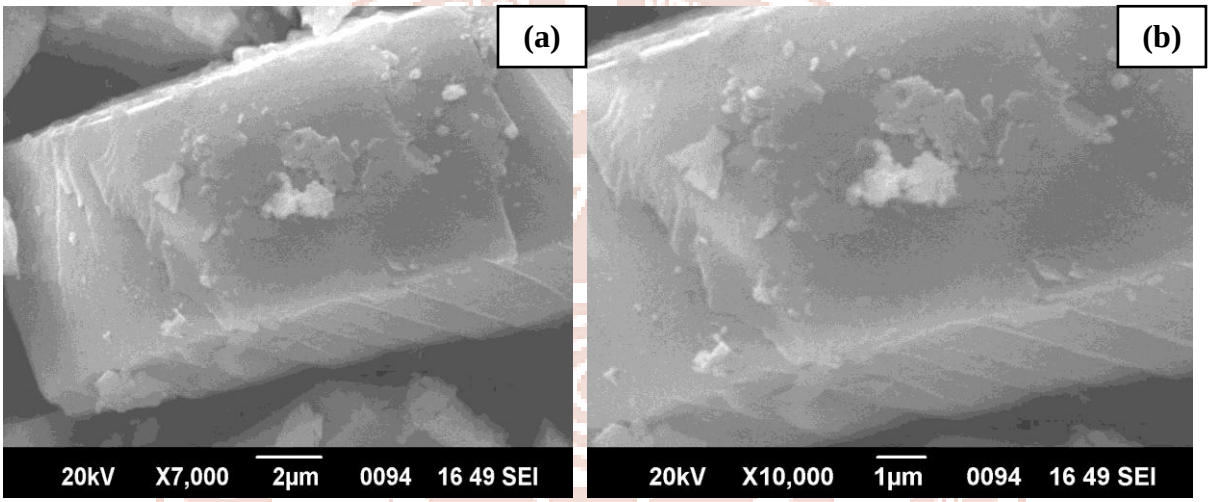

Figure 3: SEM image of as-grown nanocrystalline SnSe powder

4. Conclusions:

This work successfully demonstrates a facile and efficient chemical precipitation route for the synthesis of nanocrystalline SnSe. X-ray diffraction analysis confirmed the formation of an orthorhombic SnSe phase with a mean crystallite size of 24.34 nm while EDAX verified the presence of Sn and Se as the major constituent elements. SEM findings revealed agglomerated layered microstructures formed by clustered nanocrystalline domains. Overall, these findings suggest that the chosen synthesis method is suitable for producing nanocrystalline SnSe with favourable structural, compositional and morphological properties making it a promising material for use in thermoelectric, optoelectronic and energy-related devices.

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